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Tema: Lavra e tratamento de minérios

PASTE PRODUCTION FROM PHOSPHATE ROCK TAILINGS TROUGH FREE SETTLING AND VACUUM FILTRATION*

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Abstract

Dewatering stage is among the most important industrial unit operations, and is wide adopted in many different industries, due the fact that a large portion of industrial products is produced using suspensions of solids in liquids. Nowadays the mineral processing tailings disposal is a big problem due the environmental degradation produced. The phosphate rock processing in Anglo American Phosphate Brazil, situated in Catalão/Go/Brazil, generates around 180 t/h of tailings for a plant feed of 480 t/h (approximately 37,5% of the processing plant feed), with 5 to 10% of solids and approximately 14% of P_2O_5. Nowadays the tailings are sent direct to the tailings dam. The present work proposes the paste production using with the tailings from phosphate rock processing plant divide in two different stages. The first one composed by the free settling of the material in presence of industrial flocculants in a graduated cylinder with 2.0 L of internal volume for one hour, in the same way it must occur in conventional thickeners. After that time, the clarified liquid was drained and the remaining pulp was sent to the second stage consisting in the tailings vacuum filtration. Ten different flocculants and nine different filter media were tested. The found results were satisfactory for paste tailings production with solids percentage around 65%.

Keywords: Mineral paste; Phosphate rock; Dewatering; Tailings.

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1 INTRODUCTION

Tailings produced in mineral processing stages offer a large range of environmental issues regard its disposal, usually in dams, which demands very wide areas, contaminating hydric resources, has a high construction cost and require a very long monitoring according to environmental regulations. All considering, studies about tailings disposal as paste are ongoing in several countries, since this disposal option usual offers high water recover and recirculation, lower investment and operation costs and lower environmental impact.

Usually ores require water in its process, being mandatory recover most of the water added in its industrial processing. The solid/liquid separation process is, therefore, a very important process in the mineral processing chain. These processes had been studied due not only for the water recovery, resource scarcer and costlier every day, but also due its large energy consumption [1].

Sedimentation is a solid/liquid separation process based in the density difference of the suspension compounds. Therefore, the solid particles removal in a liquid media can be perform by the gravitational field action, which grant the process low cost and great operational simplicity [2]. The wide adoption of industrial thickeners and clarifiers promote a rising interest in the settlers dimensioning and operation, objecting improve its utilization and efficiency without lose its primary operational objectives.

In the specifically case of mineral particles smaller than 10 µm the solid/liquid separation process is extremely affected by the particle dispersion effect, caused by the Brownian motion, characteristic in colloids systems, which difficult the settling. In this case, the agglomeration of the solids particles before the solid/liquid separation process must be consider, either by flocculation or by coagulation. The presence of ultra-fine particles in the suspension affect the thickener efficiency, low the filtration rates and may produce cakes with moisture levels higher than desired, therefore is very important the pre-processing of the mineral pulps.

Flocculation is a fine particles agglomeration process in which the particles are aggregated into flocks. The process involves three major steps [3].

1. **Fine particles destabilization** thorough the elimination of any particle/particle repulsion force, due superficial electrical charges or any force which may oppose to agglomeration of the particles;
2. **Flock formation and grow**, when the particle-particle agglomerated develops either by collisions or by adherence;
3. **Flock degradation**, which involves broken mechanisms due shear stress, turbulence etc. in the flow stream.

Thickening can be defined as process to obtain a more dense mineral pulp (higher solidi percentage) and to produce a clarified liquid phase. The classical mechanism in this operation is the free settling (influenced only by the gravity force) in cylindrical tanks (often with diameter higher than its height) made of different materials such as wood, steel or concrete. In conventional thickeners, the pulp is fed in the upper center part of the tank and can leave it by one of two exists. One in the lower center part, where most of the solids leave the thank, and another in the higher part all around the tank, where a circular groove collects the clarified liquid. A rake motioned by a vertical axis takes the settled material into the discharge opening in the lower center part. Small industrial thickeners can present a flat bottom, but is most common for thickeners have a slightly inclined bottom in the center direction easing the solid discharge [4].

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The design of continuous thickenings consists in the evaluation of the settling area and the equipment height, being the necessary data for the design as well as other operational informations of this equipment obtained by practical tests using graduated cylinder of 2.0 L [5].

Filtration can be defined as a solid-liquid separation process wherein the mineral pulp is forced to pass through a porous media (called filter media) which retain must of the solids and allow the liquid phase passage [6]. In the mineral industry, this process is used very often after the thickening. The filter media correct chosen is one of the most important aspects to achieve a high efficient filtration, since its function is also act as a support base for the produced cake. A very important feature required of the filter media is its capacity of retain solids without obstruct its porous, otherwise the liquid passage became very difficult, besides be mechanically and corrosion resistant. Usually a blow of air in the cake discharge performs the filter media clearing.

Filter media are manufactured from cotton, wool, linen, jute, nylon, silk, glass fiber, porous carbon, metals, rayon and other synthetics, and miscellaneous materials such as porous rubber. Cotton fabrics are by far the most common type of medium, primarily because of their low initial cost and availability in a wide variety of weaves. They can be used to filter solids as fine as 10 µm. However new materials have emerged as substitute to the materials usually used. It is possible to highlight the geotextile fabrics [7].

Geotextile fabrics are permeable, flexible and thin fabrics made of synthetic or natural fibers. The geotextile fabrics present advantages in relation to the conventional filter media such as thinner for the same permeability, easy installation and maintenance, low cost, defined and controlled both physical and chemical characteristics being the geotextile fabrics an industrial product [8]. The geotextile fabrics can have porous obstructed by fine particles in suspension (diameters from 7 to 50 µm) during the mechanical filtration. Colloidal particles can be captured by its porous thorough physical, chemical or electrostatic interactions. In unfavorable conditions to chemical or electrostatic interactions, the colloidal behavior can be different from the bigger particles, not being filtered and following the liquid flow.

Paste tailings can be defined as a colloidal system which present itself as an homogeneous fluid, in which does not occur granulometric segregation of the particles and do not show significantly liquid bleed when gently disposed in a stable surface [9]. A simple and practical definition of paste tailings is tailings that have been sufficiently dewatered that they do not have a critical flow velocity when pumped, do not segregate when deposited and produce minimal water bleed when discharged from the end of a pipe [10]. The increased viscosity associated with this dewatering also means that paste tailings require positive displacement pumps for pipeline transport, thereby limiting the distance over which paste tailings can be economically transported. Typically, paste tailings will flow to a slope about three to ten degrees, provided the underlying material has stabilized. The construction of stack disposal with paste tailings is a natural follow on from depositing tailings as a dense, non-segregating slurry.

The present paper propose a solid-liquid combination of tests to optimize water from mineral process recovery contained in the tailings generated in the phosphate rock mineral processing by Anglo American Phosphate Brazil. Ten different flocculants and nine different filter media were tested. The solids percentage and pH used in the samples were identical to the ones used in the industry. Although nowadays the phosphate rock tailing is send direct to the tailings dam, the obtained results are

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consistent and satisfactory for paste tailings production, which can be a future alternative for this tailing disposal.

2 MATERIALS AND METHODS

The phosphate rock tailing used were donated by Anglo American Phosphate Brazil, located in Catalão, Goiás, Brazil. The tested tailings was the slime produced in the comminution closed circuit and it is removed by de-sliming cyclones. Nowadays the slime is pumped direct to the tailings dam.
Free settling tests in graduated cylinders of 2.0 L were carried out to determine the better flocculant to be used with the phosphate rock tailing. The samples collected direct in the mineral processing plant and delivered to the laboratory in 200 L barrels. The barrels were sealed to avoid any water loss or contamination. The tailing in the barrel were homogenized and samples of 10 L were collect to feed the free settling tests. Ten flocculants (see Table 1) from three different companies (Basf, Clariant and Kemira) were tested regard efficiency, settling duration, flock formation and stability in the vacuum filtration.

Table 1. Tested flocculants and their description

Fabricant	Trade name	Description
Basf	Magnafloc 10	Very high molecular weight, slightly anionic polyacrylamide
	Magnafloc 155	High molecular weight anionic polyacrylamide
	Magnafloc 351	High molecular weight non-ionic polyacrylamide
	Magnafloc 1011	Very high molecular weight anionic polyacrylamide
Clariant	Bozefloc A61 BT	Copolymer of sodium acrylate and acrylamide
	Bozefloc C630	Copolymer of sodium acrylate and acrylamide
Kemira	Superfloc A-100	High molecular weight and very low relative charge anionic polyacrylamide
	Superfloc A-130	High molecular weight and medium degree of anionic charge anionic polyacrylamide
	Superfloc C-492	High molecular weight and very low relative charge cationic polyacrylamide
	Superfloc C-494	High molecular weight and medium relative charge cationic polyacrylamide

The pH control was performed by a pH meter Gehaka T-1000 and all tests were conducted in the pH 7. After the flocculant addiction, the samples were left free settling for one hour. The clarified liquid produced was drained and its turbidity was measured using a Hanna Instruments HI 93703C portable turbidimeter. The settled solids were vacuum filtered using a Prismatec 132 vacuum pump for six minutes with an initial vacuum of 700 mmHg. To hold the filter media a device made of polyurethane was develop, with internal diameter of 13 cm. The filtered liquid were stored in a Kitasato flask directed connected with the vacuum pump. Figure 1 shows the experimental apparatus used in the vacuum filtration tests.

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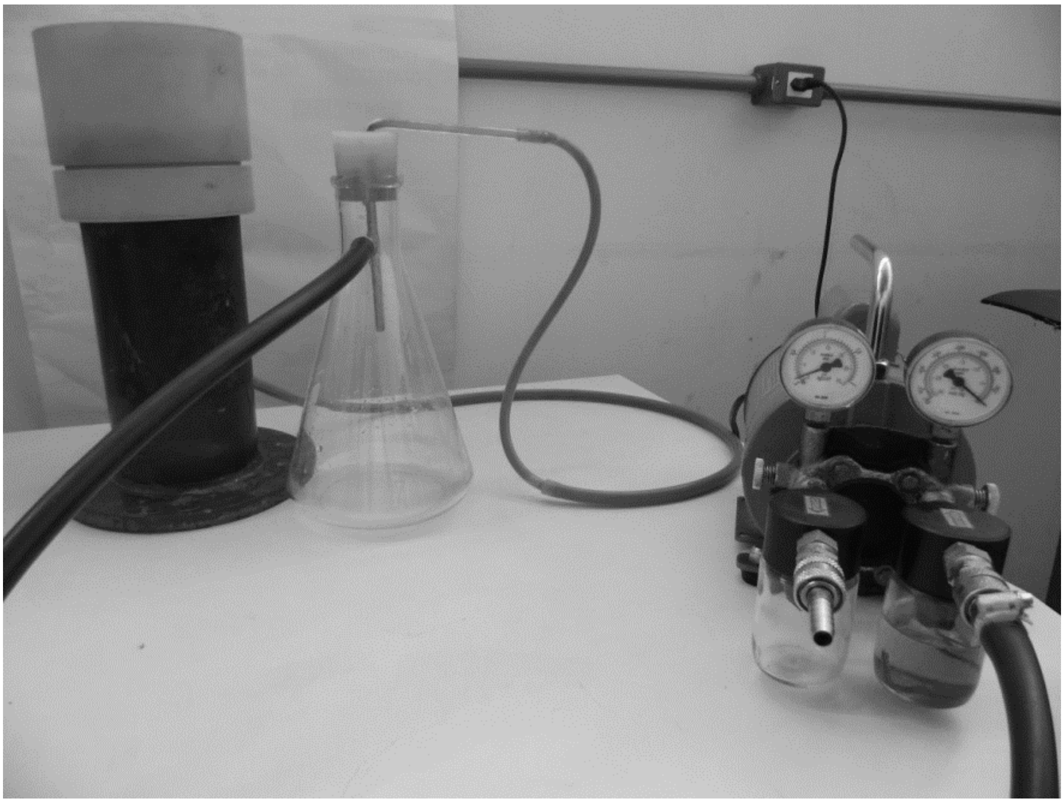
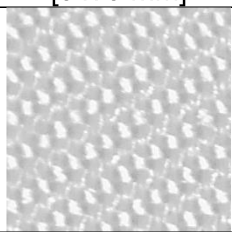
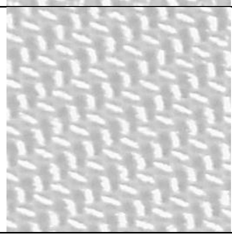


Figure 1. Vacuum filtration assemble used in the tests

Nine different filter media (regarding composition, finishing and permeability) were tested (three tests for each filer media and flocculant, 270 tests in the total), three made of Nylon, four from Polypropylene (all made by Remae) and a geotextile fabric (trade name Bidim CC-10 made by Bidim). Table 2 shows the major characteristics of the filter media tested, as well as a picture (9 x 9 mm) of each one.

Table 2. Filter media used in the tests

Fabricant	Trade name	Fiber	Finishing	Permeability [$m^3/min/m^2$]	Picture [9 x 9 mm]
Remae	1097-EL	Multiple wire Nylon	None	7.0 – 14.0	
Remae	1097-TE	Multiple wire Nylon	None	1.5 – 6.0	

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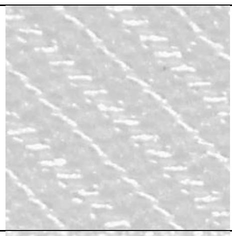
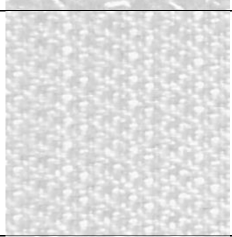
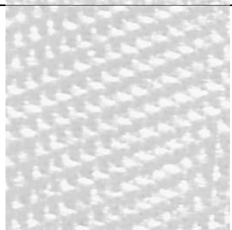
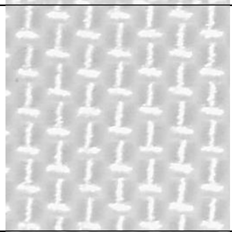
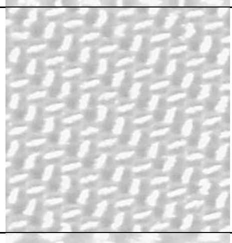
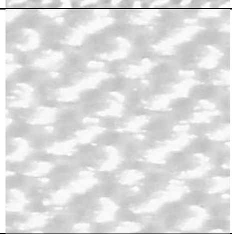
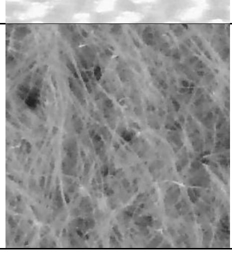
Remae	1142-T	Multiple wire Nylon	Thermoset	10.0 – 16.0	
Remae	4230-T	Single wire Polypropylene	Thermoset	60.0 – 80.0	
Remae	4233-TC	Single wire Polypropylene	Calendered	15.0 – 20.0	
Remae	4400-T	Multiple wire Polypropylene	Thermoset	0.6 – 1.2	
Remae	4520-T	Multiple wire Polypropylene	Thermoset	1.5 – 5.0	
Remae	4710-T	Multiple sliced wire Polypropylene	Thermoset	0.3 – 0.5	
Bidim geotextile	CC-10	Geotextile non-tissue 100% polyester	None	4.2 – 7.2	

After the vacuum filtration the paste where weighed and sent to dry in drying oven at 120o C for five hours. The dried paste and the filter media were both weighed after drying to allow the evaluation of the solids percentage in paste tailing produced.

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3 RESULTS AND DISCUSSION

The chemical analysis of the phosphate rock tailings sample used in this work, obtained by X-Ray diffraction, is presented in Table 3 and the granulometric analysis is given in Table 4. For the granulometric analysis was performed the wet screening, which allows finer sizes to be processed efficiently down to 250 µm and finer[7]. Both analysis were performed in Anglo American Phosphate Brazil mineral characterization lab.

Table 3. Major oxides present in the phosphate rock tailings

Oxide	P_2O_5	Fe_2O_3	SiO_2	BaO	Al_2O_3	CaO	MgO	Nb_2O_5
%	11.10	31.37	21.45	1.22	3.72	12.91	1.62	0.70

Table 4. Phosphate rock tailings granulometric analysis

Screen aperture	Mesh [#]	65	100	150	200	270	325	400
	µm	209	148	105	74	52	45	37
% accumulated passing		99.32	98.81	98.45	97.59	97.05	96.61	96.16

Figures 2 to 4 shows the average results for the sedimentation tests. Each test were conducted three times to confirm the results and to allow a statistical analysis. In all tests, the pH was set to seven, to avoid additional stages of water treatment in a possible industrial use. It is possible to notice that flocculants behavior changes with its concentration.

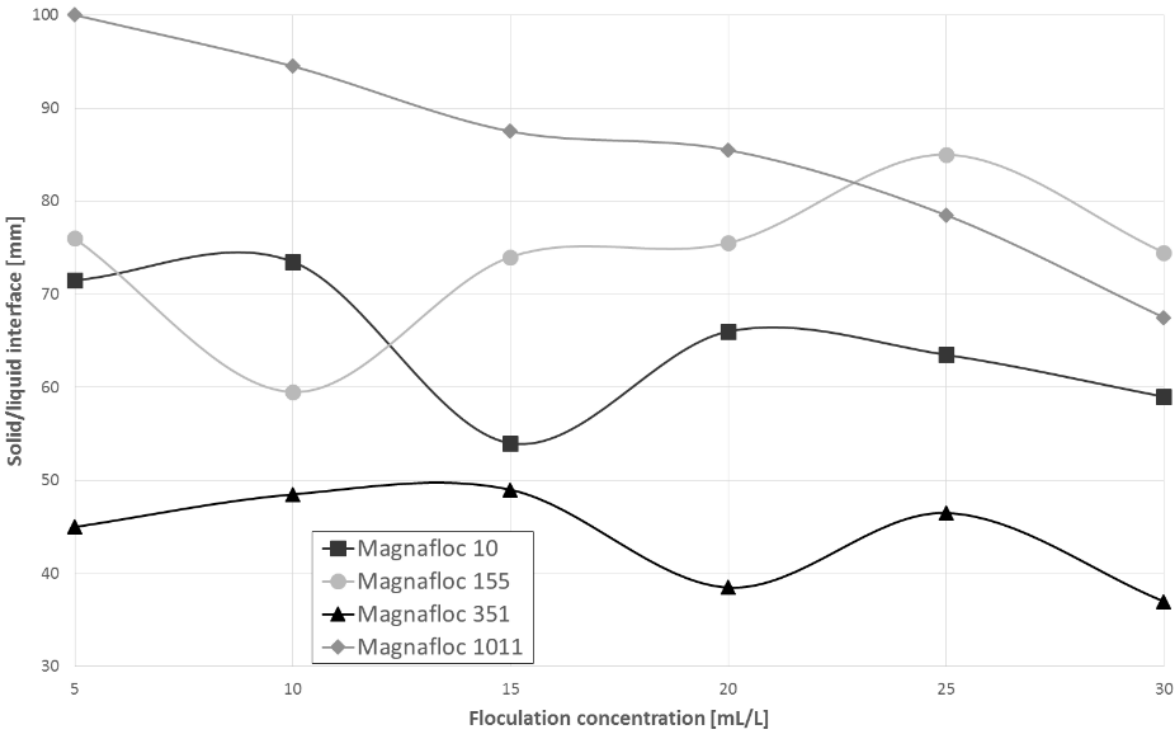

Figure 2. Tests with flocculants produced by Basf

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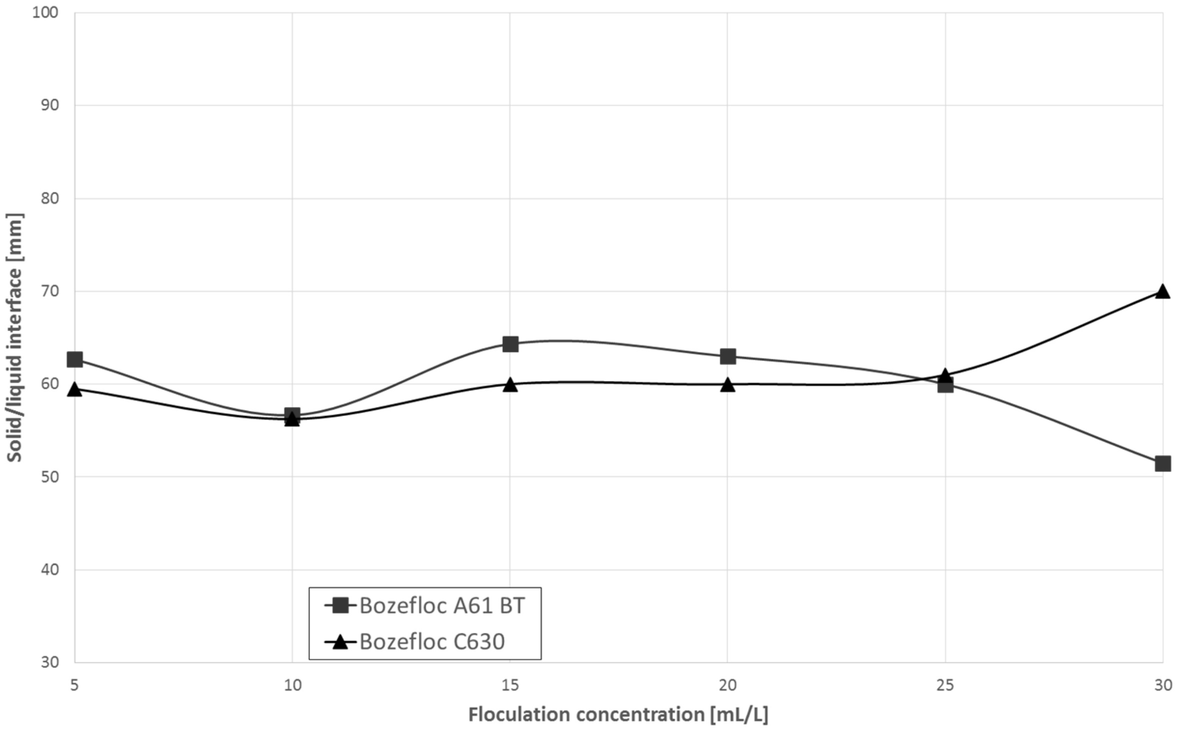

Figure 3. Tests with flocculants product by Clariant

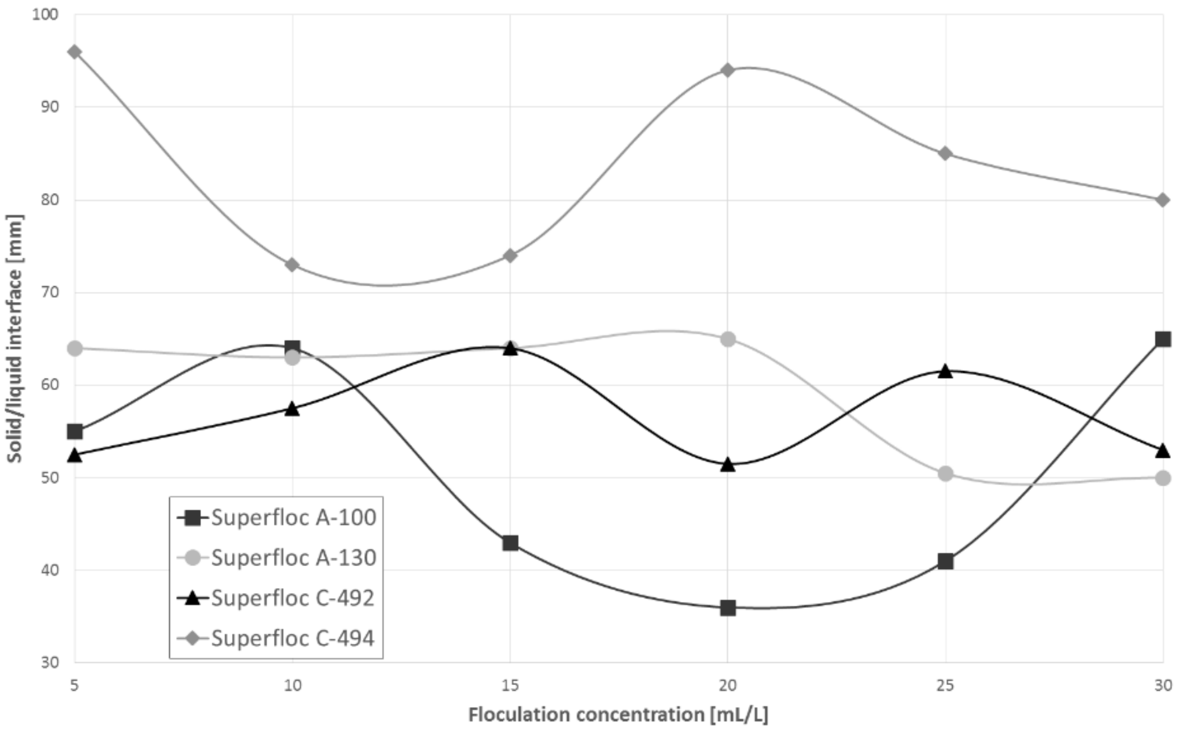

Figure 4. Tests with flocculants product by Kemira

The flocculant chosen for the vacuum filtration tests were Kemira Superfloc A-100 (20 mL/L), because it has an excellent performance in the settling tests and was able to produce stable flock for the filtration. Free settling tests with long duration were performed (see Figure 5) and no significant changes were observed in the solid/liquid interface height was observed after one hour. The other tested flocculants produced

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breakable flocks in the vacuum filtration and did not generate even a filtered cake, most of the solid material passing through the filter media. The Figure 6 shows a sample of tailings after liquid draining and before the vacuum filtration.

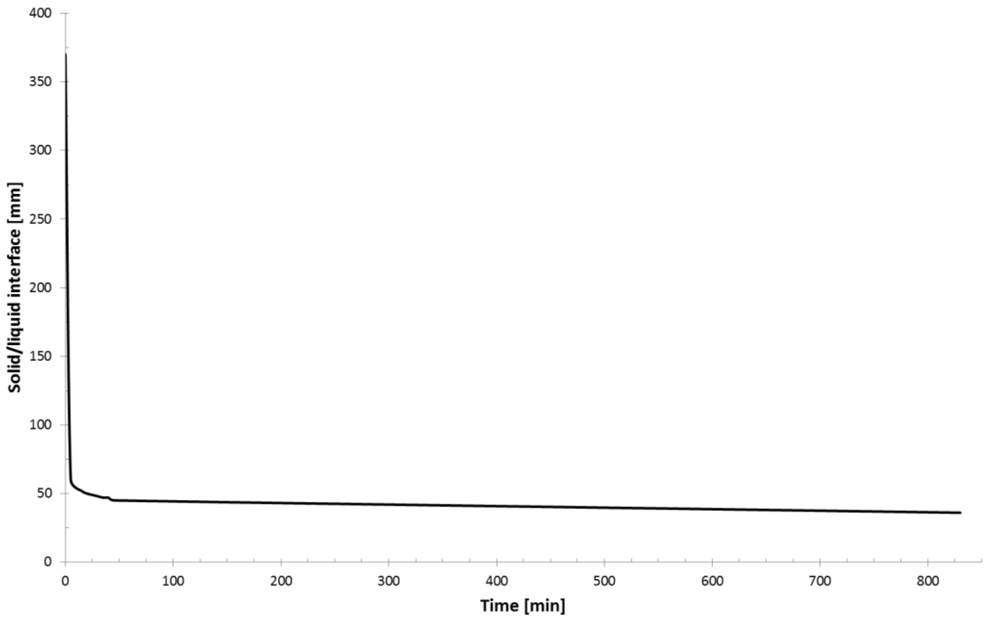

Figure 5. Free settling versus solid/liquid interface height for Kemira Superfloc A-100 20 mL/L

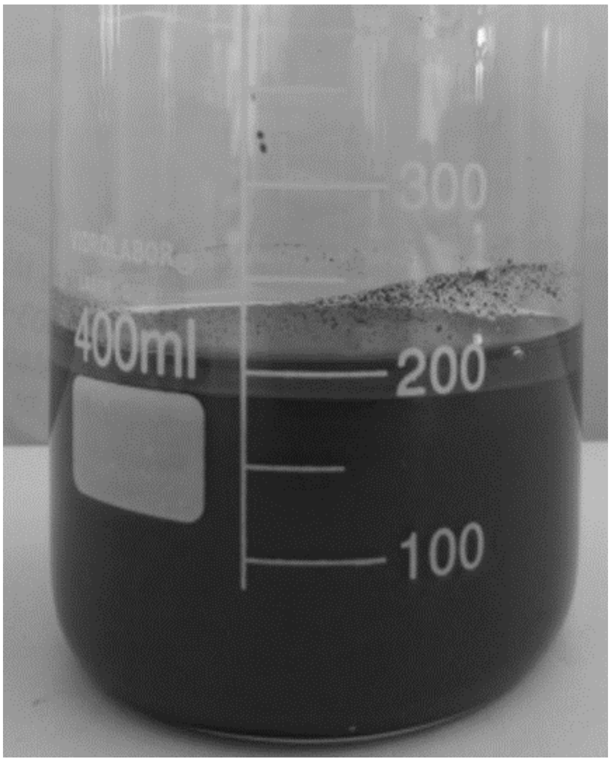

Figure 6. Sample of flocculated material before vacuum filtration

Figure 7 shows six different paste tailings produced from phosphate rock tailings using the proposal methodology. It is possible to see the reflection of the remaining water present in the paste, although it partially resists to the shear stress and retain a

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cylindrical form from the recipient used in the vacuum filtration, indicating a rheology consistent with a paste tailings.

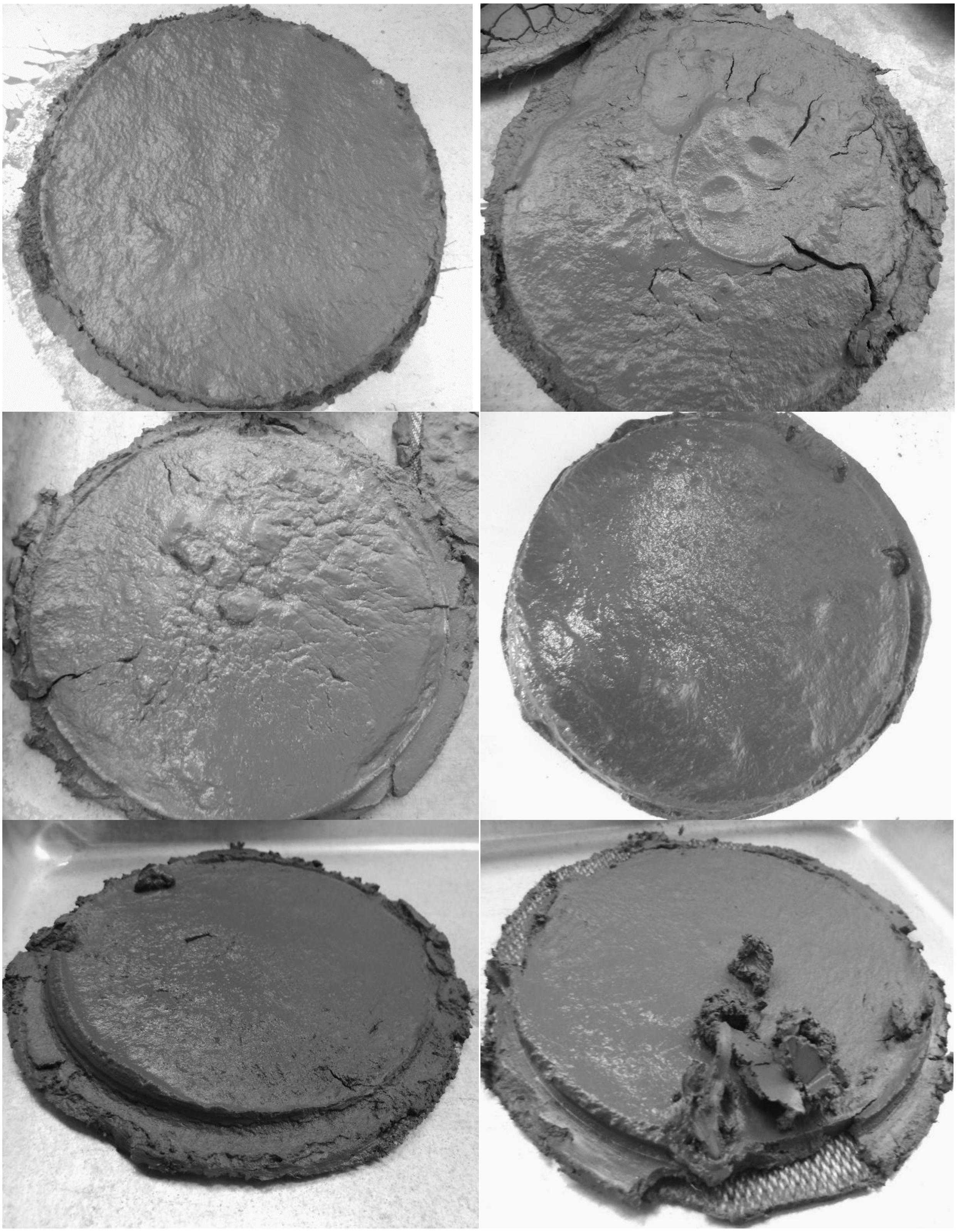

Figure 7. Six different paste tailings produced from phosphate rock tailings

Table 5 summarizes the results of the vacuum filtration tests showing the volume of liquid filtrated, mass and solids percentage in the paste and the pressure drop from

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the beginning and the end of the test. A high vacuum pressure indicate a high permeability from both filter media and pulp and a low vacuum pressure indicate a low permeability. It is possible to notice that for best results (filter media Remae 4230-T and 4233-TC) according to the solids percentage in the paste the pressure drop was zero, although both initial and final vacuum pressure were low. This is consistent with the presence of capillary ducts in the paste, which allow the water follow but not spontaneously. For the other filter media, the porous obstruction were noticed after the test and the filter media needed to be clean to remove the remaining particles, which explains the pressure drop. For these cases, the paste height was higher and the solids percentage lower.

Table 5. Average vacuum filtration results for phosphate rock tailings paste tailings production

Filter media	Flocculated (mL)	Filtrated (mL)	Paste mass (g)	Paste solids percentage (%)	Vacuum (mm Hg)	
					Initial	Final
1097-EL	615.00	440.00	112.49	59.53	500.00	350.00
1097-TE	190.71	128.57	81.90	54.59	400.00	302.86
1142-T	161.67	96.67	83.17	52.32	390.00	316.67
4230-T	175.00	91.67	81.56	62.42	100.00	100.00
4233-TC	175.00	93.33	79.95	64.55	103.33	103.33
4400-T	175.00	115.00	71.75	47.56	433.33	323.33
4520-T	166.67	95.00	66.16	50.50	256.67	173.33
4710-T	189.29	117.86	94.32	56.10	140.00	120.00
Bidim geotextile	190.00	112.14	96.68	47.81	211.43	147.14

Figure 8 shows the average solids percentage for the filter media tested with Kemira Superfloc A-100 (20 mL/L). The best result according the solids percentage was obtained with Remae 4233-TC (64.55%). The geotextile fabric showed a poor result (47.81%) and often showed tailing remaining in it after the test and its cleaning was very difficult, due its own nature and fabric.

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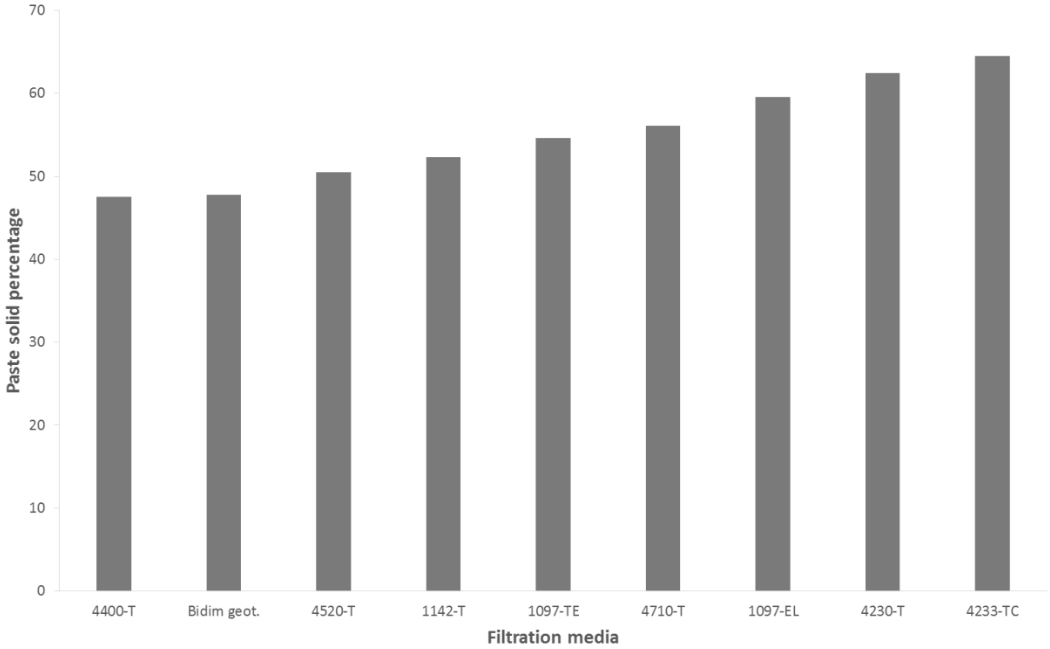

Figure 8. Average solids percentage in the produced paste tailings

It was not possible establish a statistical correlation between the pressure drop and the solids percentage (see Figure 10) because, in great part, the differences in the filter media permeability. There were no pressure drop for the two better filter media (Remae 4233-TC and Remae 4230-T) both with high permeability values (60.0 – 80.0 and 15.0 – 20.0 $m^3/min/m^2$, respectively). However, the Remae 4710-T (with low permeability, 0.3 – 0.5 $m^3/min/m^2$) and the geotextile fabric (medium permeability, 4.2 – 7.2 $m^3/min/m^2$) had both small pressure drop but with different values of solids percentage in the paste produced (56.10 and 47.81, respectively).

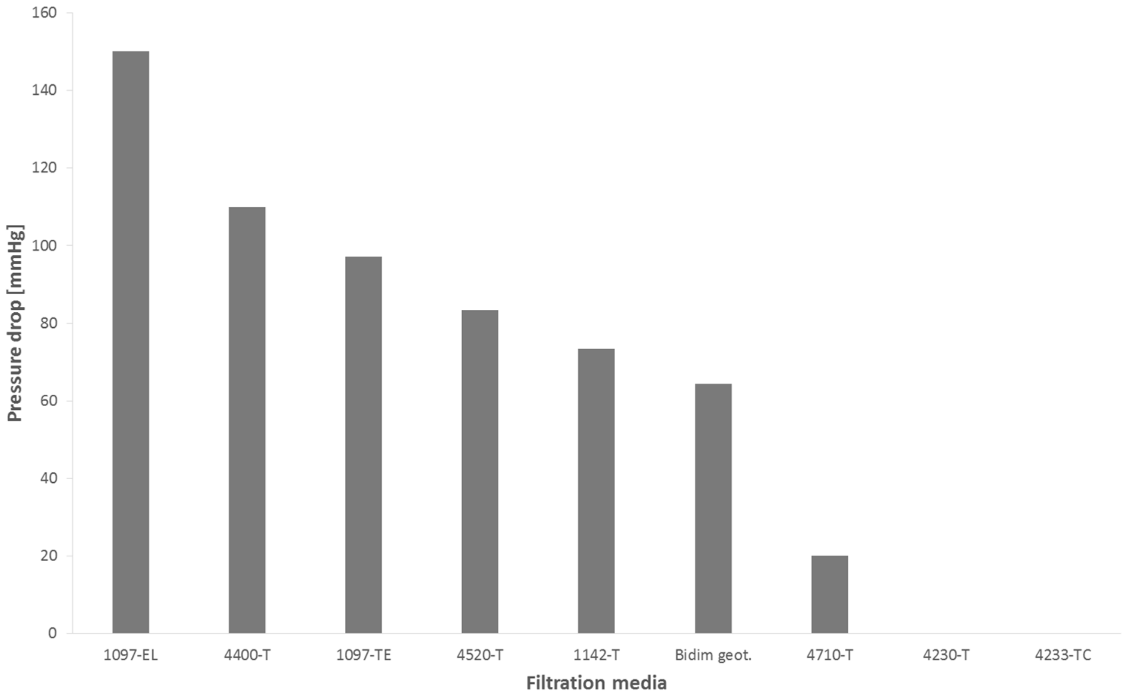

Figure 9. Average pressure drop in the vacuum filtration tests.

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4 CONCLUSIONS

For the free settling tests, the good results according the settling speed and solid/liquid interface height were obtained with Basf Magnafloc 351 (38.5 mm at 20 mL/L), Kemira A-100 (36.0 at 20 mL/L) in the pH 7. However, for the vacuum filtration tests only the results with Kemira A-100 where shown since this flocculant was able to form very resistant flocks and capable of resist the filtration. The flocks from the other tested flocculants broke in the filtration, reducing the liquid percolation and obstructing the filter media. In some cases, the filter media was not able to filter the solid and great part of it pass through it with the liquid.
Remae 4233-TC and 4230-T showed higher efficiency for solids retain in vacuum filtration tests. For this filter media no pressure drop has been noticed, which indicate a lower porous obstruction.
Visual and tact analysis of the produced cake indicated a behavior compatible with paste tailings and the adoption of this strategy can lead to a considerable economy with tailings disposal, replacing the actual system using a tailings dam by a pile disposal system. More tests are still ongoing aiming in determine the optimal pH in the free settling and to characterize the rheology of the paste tailings produced.

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