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Development and Testing of a Laboratory Scale Paste Thickener

R.H. Olcay Barreda *Mining Engineering Department, Universidade Federal de Minas Gerais, Brazil*

A.C. de Araujo *Mining Engineering Department, Universidade Federal de Minas Gerais, Brazil*

G.E.S. Valadão *Mining Engineering Department, Universidade Federal de Minas Gerais, Brazil*

C.A. Hernández *Engineering Department, Universidad Arturo Prat, Chile*

Abstract

The major objective of this study was the design, construction and testing of a laboratory scale paste thickener. This unit has a modular construction and can be operated on a totally batch mode or as a fully continuous thickener. The thickener was constructed in Plexiglas in order to allow the direct observation of the slurry behaviour inside the equipment. It has two modules reaching a maximum height of 1.2 m with 10 cm of internal diameter. It still makes use of a system for taking samples with orifices of 1cm of internal diameter and equally spaced in the vertical axes by 9 cm. The mineral pastes produced by the equipment were characterised for their rheological properties by means the determination of: viscosity (viscometry), consistency (slump test), fluidity (flume test) and yield stress measurements. The best result without the addition of flocculant in the tests, with the recirculation of the pulp and a residence time of 10 min, was reached when the slurry was taken from orifice number 3 to orifice 1. The paste reached 55.6% of solids by weight under this testing condition. Considering the addition of flocculants the best result was achieved with recirculation of the slurry from orifice number 4 to orifice number 1. This paste reached 53% of solids by weight. Employing two modules of the thickener, the final paste with recirculation reached 56.7% of solids by weight.

1 Introduction

According to Araujo et al. (2003) and Jewell (2002), a mineral paste is defined as a fine particle system that presents itself as a homogeneous fluid in which segregation of particles do not occur as well as any significant water drainage is present. As a result, higher angles of disposal are obtained, for example in the range from 2 to 5%, representing an important gain in terms of volumes per area of disposal (Araujo et al., 2003). Some important characteristics that have to be considered for tailings disposal are height of slump, repose angle, viscosity behaviour and shear stress. Currently the production of mineral paste can be carried out by means of thickeners or filters for backfill (underground mining) and surface disposal. The use of paste thickeners represents an attractive alternative to the filtration operation. Thickeners are utilised, for example, for the production of red mud paste in Australia (Slottee et al., 2005). The DeBeers paste thickener of the CTP plant, in Kimberley, is an example of disposal of paste using a surface disposal system known as stacking (Houman and Johnson, 2003). Research programmes to understand the complex mechanisms involved in the production mineral pastes are presently in high demand.

2 Methodology

2.1 Materials

The mineral sample represents a tailing obtained from the mineral processing of the manganese ore and came from western region of Brazil. The specific gravity, the size distribution, the chemical and mineralogical compositions, and specific surface area were determined for the sample.

2.2 Experimental methodology

The samples were received in the form of slurry. The liquid component (water) was separated from of the solid by using filtration. The solid was dried at a 100ºC. Representative samples were obtained for the determination of different physical and chemical properties of the material. Table 1 presents the techniques utilised in this case.

Table 1 Techniques utilised for determination physical and chemical properties of the material

Property	Technique
Specific gravity	Conventional picnometry (CP) Gas picnometry (GP)
Size distribution	Laser diffraction
Chemical composition	X Ray fluorescence (XFR) Scanning electronic microscopy and microprobe analysis by EDS
Mineralogical composition	X Ray diffraction Infrared spectroscopy and SEM/EDS
Specific surface area	BET nitrogen adsorption Air permeability

The residence time necessary to the paste formation was determined by using graduated cylinders (2000 mL) with different initial solid percentages (10%, 15% and 20%). The flocculant selection was carried out utilising graduated cylinder (250 mL) and three types of flocculant: anionic, cationic and non-ionic. The flocculants tested are indicated in Table 2.

The thickener developed herein is made of Plexiglass and has a modular system with two cylindrical modules having each one a length of 60 cm and 10 cm of internal diameter. It has a set of orifices that are used for sampling and/or slurry recirculation.

Table 2 Tested flocculants

Cationic	Anionic	Non-Ionic
SC-492	SA-130	SN-100
SC-496	SA-110	SN-300
SC-494	BA61BT	M-351
BC-630	M338	-
-	M10	-

The orifices have an internal diameter of 1 cm and the distance between them is 9 cm. The orifices are enumerated of 1 up to 10 from bottom to the top. The underflow discharge is carried through a conical surface. An acrylic bar of 9 cm of diameter and length of 1.4 m was used for agitation in batch tests. For the continuous tests, agitation was performed using mechanical agitation of the slurry feed. Peristaltic pumps were also utilised for slurry feeding and recirculation. Figure 1 shows the equipment with two modules in continuous process operation and the system for sampling.

The conditions of the batch tests, when only one module was utilised, were as follows: feed with 15% solids (w/w), different volumes of slurry (1000, 2000, 3000 and 4000 mL), and different residence times (5, 10, 15 min), without flocculant. The influence of the flocculant (SN 300) addition was only verified for total volume of 4000 mL.

The conditions of the batch tests, when two modules were utilised, were as follows: feed with 15% solids (w/w), without and with flocculant (SN 300) addition, different volumes of slurry (5000, 6000, 7000 mL), and residence time of 10 min.

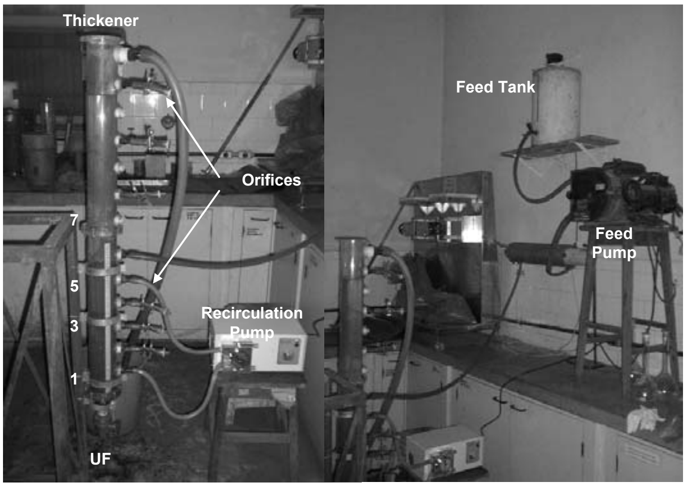

Figure 1 Equipment with two modules in continuous process mode

The conditions of the semi continuous tests when one module was used were slurry recirculation with different combinations, without and with flocculant (SN 300) addition, volume of 4000 mL and residence time of 10 min. The conditions of the semi continuous tests when two modules were used were slurry recirculation with different combinations, without and with flocculant (SN 300) addition, volume of 7000 mL and residence time of 10 min. The continuous tests with two modules (7000 mL) were carried out in presence of flocculant (SN 300) in dosages of 25, 50 and 75 g/t. The slurry was fed at two different heights (orifices 3 and 6) that correspond respectively to 27 and 54 cm height. The total running time was to 180 min.

The mineral pastes were studied by means of slump test, flume test, and viscometry.

The slump tests (Clayton et al., 2003) were performed based on a Brazilian standard procedure (ABNT, 1998). It was utilised a cylinder with height of 10 cm and diameter of 10 cm.

The flume tests were carried out using the apparatus with 100 cm of length, 20 cm of width, and 20 cm of height. The tested slopes were 1, 2, and 3%. The flume apparatus is shown in the Figure 2.

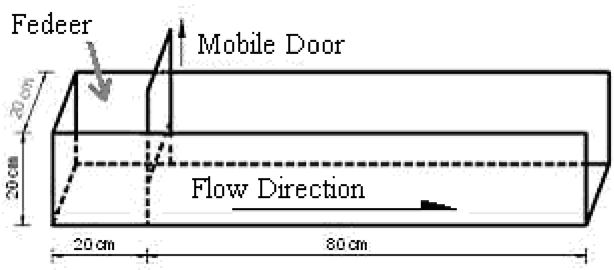

Figure 2 Flume apparatus (modified from Kwak et al., 2005)

A Brookfield DVIII viscosimeter and a Brookfield YR-1 rheometer were utilised for the determination of viscosity and yield stress, respectively.

3 Results and discussion

The values of specific gravity were 2.87 g/cm^3 (water picnometry), and 3.14 g/cm^3 (gas picnometry).

The size distribution of solid particles of mineral sample obtained by means of Sympatec (laser diffraction) equipment is shown in the Figure 3.

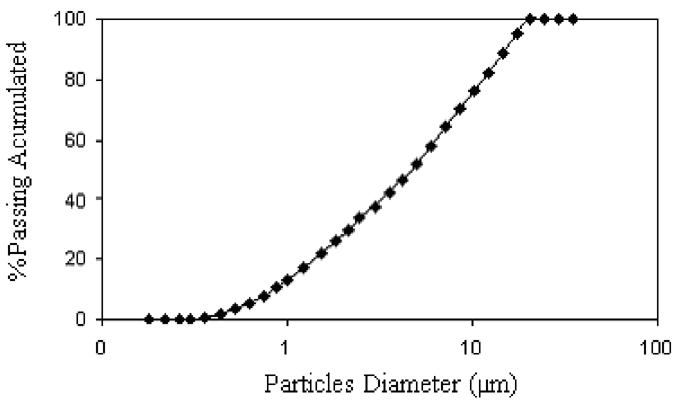

Figure 3 Size distribution of the solid particles of the mineral sample

Approximately 80% of the particles are smaller than 10 µm, that is, the material has high content of slimes.

The chemical analysis, presented on the Table 3, shows that the predominant elements are Mn, Si, Al, and Fe. The minor components are Mg, K, Na, and Ca.

Table 3 Chemical analysis

Element	(% Weight)
Mn	21.87
Si	30.16
Al	28,04
Fe	18.77
Mg	0.00
K	0.82
Na	0.23
Ca	0.11

The X-ray diffraction and the infrared spectroscopy indicated chiefly the presence of pyrolusite (MnO_2) and kaolinite ($Al_2Si_2O_5(OH)_4$).

The specific surface area (SSA) was determined by means of two methods BET and Blaine. The values obtained are shown in the Table 4.

Table 4 Values of specific surface area (SSA) determined by BET e Blaine

Technique	BET	Blaine
SSA (m2/g)	28,963	0.893

The results are not similar as expected. The obtained isotherm in the BET method indicated the multilayer adsorption, isotherm type III, hence that was not considered as a true measurement of this property for this sample.

Figure 4 presents the settling curves obtained from the batch tests (graduate cylinders of 2000 mL) for 10, 15 and 20% solids (w/w). The Table 5 indicates the settling rates obtained from this curves. The results indicate that the time to reach the compression point depends on the solids percentage. For 15% of solids, for example, the time is approximately 33 min. The higher the percentage of solids the higher the settling rate. The value of 15% of solids was chosen for the further tests due to the interface being more clearly observed for this condition.

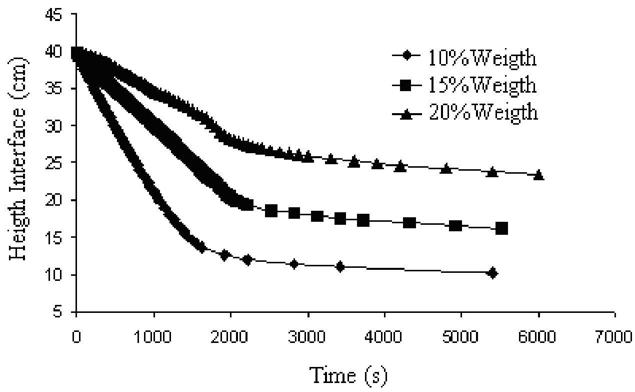

Figure 4 Settling curves obtained from batch tests (graduate cylinders of 2000 mL)

Table 5 Settling rates for 10%, 15% and 20% of solids without flocculant addition

% Solids	Settling Rate (cm/s)	(r^2)
10	2.112 x 10-2	0.999
15	1.067 x 10-2	0.991
20	2.833 x 10-3	0.990

Table 6 shows the values obtained for the settling rate at 15% of solids (w/w) in presence of the flocculants.

Table 6 Settling rate tests for cationic, anionic, and non-ionic flocculants

Cationic Flocculant	Rate (cm/s)	Anionic Flocculant	Rate (cm/s)	Non-Ionic Flocculant	Rate (cm/s)
SC492	0.0332	SA130	0.0775	SN100	0.0587
SC496	0.0335	SA110	0.0407	SN300	0.0622
SC494	0.0239	BA61BT	0.0314	M351	0.0287
BC630	0.0465	M338	0.0322		
		M10	0.0422		

Flocculants BC 630, SA 130 and SN 300 were the best in each flocculant class. SN 300 was selected for further testing. Figure 5 presents the relationship between settling rate and flocculant dosage for SN 300.

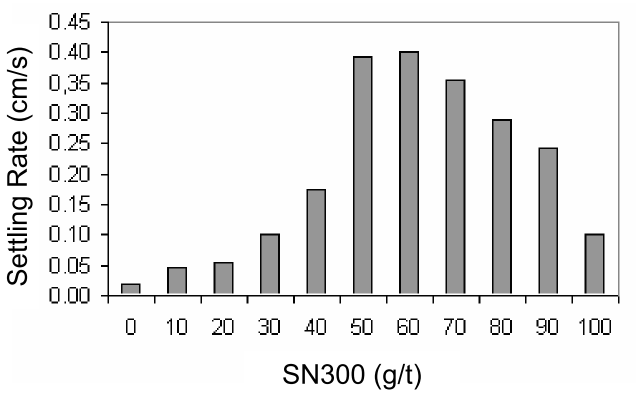

Figure 5 Settling rate as a function of dosage for SN 300

As seen in Figure 5, the highest settling rate is reached when flocculant dosage is the range from 50 to 60 g/t.

Batch, semi batch and continuous settling tests employing the laboratory thickener are presented from this point on. Figures 6a presents results obtained with one module without the addition of flocculant and Figure 6b the results of the influence of flocculant dosage on the final (underflow) percent solids.

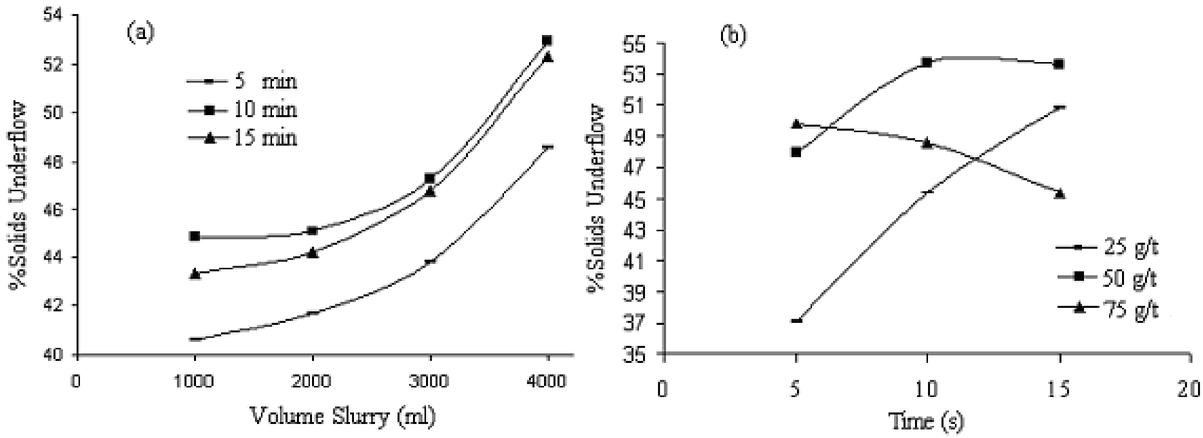

Figure 6 (a) Variation of % solids in the underflow as a function of slurry volume for different values of residence time; (b) Variation of % solids in underflow as a function of time for different values of flocculant dosages for a total slurry volume of 4000 mL

Figure 6a indicates that the % solids of the underflow are larger as the slurry volume increases for different values of residence time. The best condition of operation was reached for a residence time of 10 min at a total slurry volume of 4000 mL, with the underflow reaching 52.9% solids. The increase of the slurry content of 3.53 folds the initial solids concentration. Figure 6b shows that underflow % solids are larger as the residence time is increased for 25 and 50 g/t of flocculant. For a higher flocculant dosage the underflow % solids decreases. With the use of flocculants the highest % solids reached was 53.7%.

Semi-batch tests with one module with and without the use of SN300 flocculant are presented on Tables 9, 10 and 11. From Tables 9 and 10, it is clear that the best condition was achieved for slurry recirculation from orifice 3 to 1 without adding flocculant. With the addition of flocculant, as it can be seen on Table 9, the best condition took place with slurry recirculation from orifice 4 to orifice 1 when a final underflow of 53% solids was reached.

Table 7 Underflow % solids for 4000 mL of slurry and residence time of 10 min

Combinations (Out → In)						
% solids of underflow	1→2	2→1	3→1	4→1	5→1	6→1
	53.5	47.9	55.6	52.6	54.4	51.8

Table 8 Underflow % solids for other combination of orifices, 4000 mL of slurry and residence time of 10 min

Combinations (Out → In)						
% solids of underflow	3→2	4→2	5→2	4→3	5→3	5→ 4
	50.9	52.2	52.9	53.6	54.5	55.3

Table 9 Underflow % solids with the addition of flocculant and slurry recirculation as indicated for 4000 mL of slurry and residence time of 10 min

Combinations (Out → In)			
% solids of underflow	2→1	3→1	4→1
	52.7	47.5	53.0

The results of underflow final % solids for testing employing two modules are shown in Figure 7, with and without adding SN 300 as flocculant. Similar final % solids for underflow were reached. Without flocculant, the final underflow was slightly higher in terms of its solids content reaching 56.2% in comparison to 55.8 with flocculant. The trend of the curves shown in Figure 7 can be related by a fifth order polynomial as shown on Equations (1) and (2).

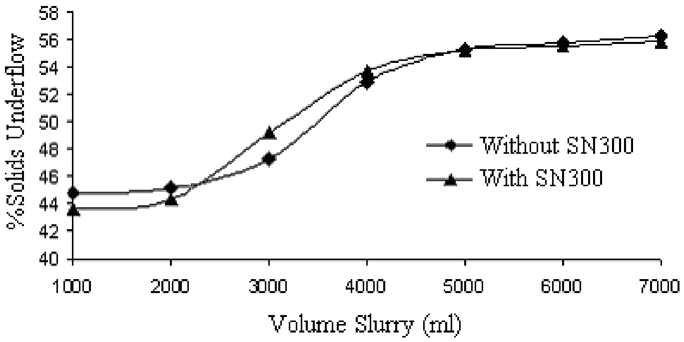

Figure 7 Variation of % solids as a function of slurry volume for modules 1 and 2 together, with and without the addition of flocculant and 10 min of residence time

$$\%S = 4\times10^{-17}\times V^5 - 7\times10^{-13}\times V^4 + 4\times10^{-9}\times V^3 - 1\times10^{-5}\times V^2 + 0.0124\times V + 39.919 \quad (1)$$

$$\%S = -1\times10^{-17}\times V^5 + 3\times10^{-13}\times V^4 - 3\times10^{-9}\times V^3 + 2\times10^{-5}\times V^2 - 0.0263\times V + 57.643 \quad (2)$$

where:

%S = % solids in the underflow.

V = slurry volume in the equipment.

Semi-batch tests with one and two modules, with and without addition of flocculant have their results summarised in Figures 9 and 10, respectively. In Figure 8, one can observe that the best condition in terms of underflow % solids was reached with slurry recirculation from orifice 7 to 1 without flocculant addition. In Figure 9 the highest underflow % solids was reached with slurry recirculation taking place from orifice 4 to orifice 1. Under these conditions the final underflow solids content reached 53.7%. For continuous operation, the highest value reached for the underflow % solids was 56.1. The total testing time was 180 min. Figure 10 shows the results obtained at various operating times for a continuous mode.

Slump tests were performed for pastes from 47 to 59% solids (Figure 11). A slump of 50% was attained at approximately 51% solids whereas a 20% slump was reached at approximately 56% solids.

Figure 12 summarises flume tests. Maximum repose angle was attained for approximately 55% solids.

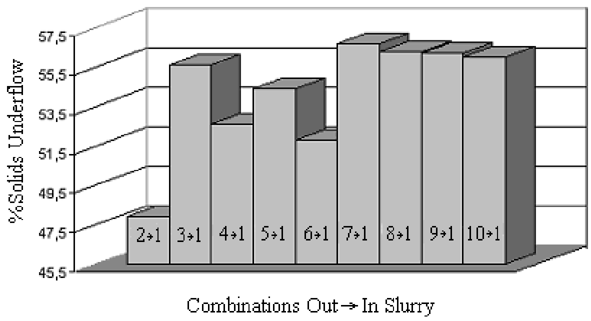

Figure 8 **Variation of % solids of the underflow as a function of different orifice combinations for a total residence time of 10 min and slurry volume of 7000 mL (two modules)**

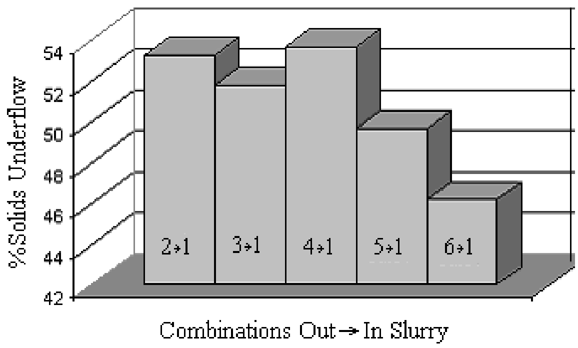

Figure 9 **Variation of underflow % solids as a function of different orifice combinations for 10 minutes of residence time and slurry volume of 7000 mL, with SN 300 addition**

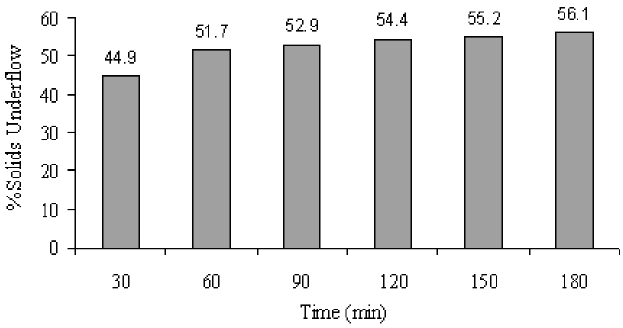

Figure 10 **Variation of % solids of the underflow as a function of total operating time for best overall conditions**

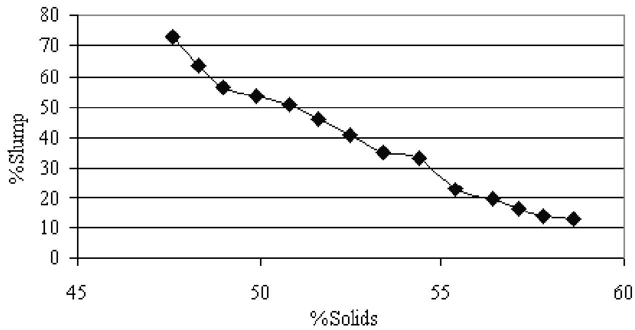

Figure 11 **Percentage of slump as a function of paste % solids**

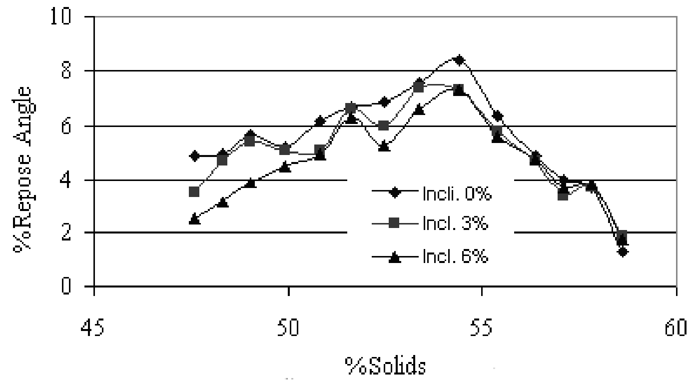

Figure 12 Repose angles as determined by flume tests

Figure 13 shows the rheological cycles for low (1-20-1), medium (1-100-1) and high (1-180-1) spindle rotation. Mixed thixotropic-rheotropic behaviour was observed, especially at the highest % solids tested.

Figure 14 presents the results of yield stress (YS) for paste with 40, 45, 50 e 55% solids. A polynomial equation of second order was found to give a good fit to data shown in Figure 14, as observed in Equation (3).

$$YS = 0.1936 \text{ x } (\%solids)^2 - 14.598 \text{ x } (\%solids) + 315.46 \quad (3)$$

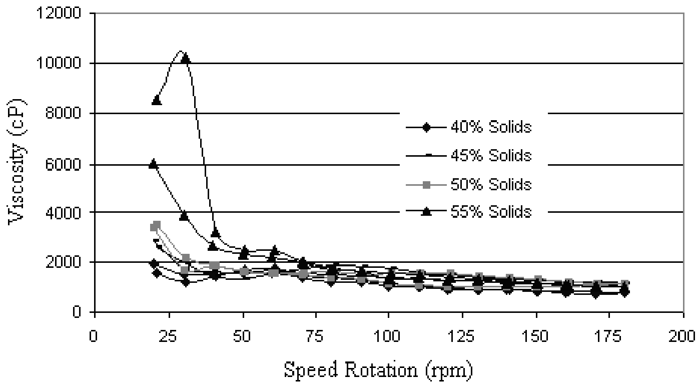

Figure 13 Rheological cycles – viscosity of pastes

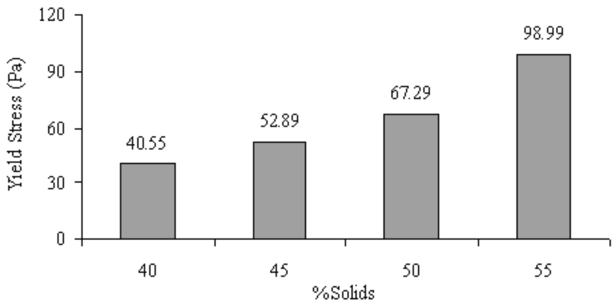

Figure 14 Yield stress for different solid contents of the pastes

4 Conclusions

The minimum time for the slurry to reach compression is 2000 s. The best flocculants for each class were BC 630 (cationic), SA 130 (anionic) and SN 300 (non-ionic).

The highest underflow % solids for tests without the addition of flocculant, was reached for a residence time of 10 min with a total slurry volume of 4000 mL and recirculation from orifice 3 to orifice 1. Under these conditions, the underflow weight percent reached 55.6%. For the same conditions but with the use of flocculant and recirculation from orifice 4 to orifice 1, the final underflow presented 53% of solids.

For semi-batch tests with and without the addition of flocculant and for a slurry volume of 7000 mL the best operational condition was reached when slurry was recirculated from orifice 7 to orifice 1 with the final underflow reaching 56.7%. When recirculation was from orifice 7 to orifice 1, the final underflow reached 53.7% solids.

On continuous operation at a flocculant dosage of 50 g/t, the underflow reached 52.7% of solids.

Pastes are formed when the % solids reaches at least 45% (w/w) for the tailings tested herein and slump tests showed less than 20% slump to occur for pastes with more than 55% solids. Flume tests showed slopes above 8º for 54.4 % solids at an equivalent 33.6% slump.

The pastes presented a mixed thixotropic-rheotropic behaviour. Yield stress increases with % solids by weight.

Acknowledgements

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