

# **Rheology applied to dewatering of mineral pulps to paste production**

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## **Abstract**

The dry disposal of mineral tailings is a better alternative for containment, because it means an increase in the stability of conventional dams, minimizing geotechnical accidents involving tailings dam rupture and consequently the negative impacts, as well as allowing the reuse of available water. Therefore, the rheological properties of tailings, such as yield stress, should be well understood to ensure maximum efficiency of thickening and disposal operations. This study evaluates the properties of nickel tailings to determine its rheological behavior and stability for disposal in dams. Therefore, the main objective is to study the effect of the initial solids concentration and flocculant dosage on the cohesion of the thickened tailings. For this, pulps with initial solids concentration of 10, 15 and 20% (w/w), were flocculated with two commercial anionic polymers and thickened. The sediment was submitted to measurements of rheological properties and slump test to obtain information about static and dynamic yield stress and slump height. The preliminary results showed strong influence of the initial solids concentration as well as dosage and type of the flocculant on the particle aggregation. The sediment solids concentration increased with rising initial solids concentration, while the slump height decreased and the yield stress increased. The dosage and type of flocculant polymers also had a strong influence on the sediment cohesion, reaching static yield stress values of up to 1,590 Pa. The densification process was efficient for particles aggregation/sedimentation, allowing total water recovery up to 90%, and the production of sediment with yield stress much greater than specified for mineral paste, 200 Pa.

Key words: paste tailings; disposition; nickel tailings; rheology; yield stress.

## **1. Introduction**

Inadequate mineral tailings management can lead to the release of millions of cubic meters of slurry and cause serious disasters, with remediless social, economic and environmental consequences. The growing awareness of environmental protection together with the reduction of mineral reserves involves the tailings reuse as an alternative to increase mineral resources. Tailings reprocessing will generate even finer tailings, which will be challenging to dewater and discard, due to the surface properties, making their environmentally friendly disposition even more difficult.

The major factor contributing to increased safety in dams is dewatering of tailings before deposition. With reduced amount of water, the material is well consolidated and even if occur any dam failures, the tailings will not flow, thus avoiding an ecological disaster. The application of tailings dewatering to achieve high consistency requires

significant economic incentives and offers important environmental benefits. The conservation of water, decreased volume of disposed tailings, and consequently reduction of the area required for storage, are some of the advantages of paste technology (BOGER, 2013; SOFRÁ & BOGER, 2002). However, this technology is more complicated than conventional methods, because it needs a high level of technological infrastructure with good understanding of key factors that affect thickening, such as size and shape of the solid particles, temperature, viscosity, solid-liquid weight ratio in feed stream, use of flocculant and method of flocculant application (CREBER et al., 2017; JARVIS et al., 2005; PARSAPOUR et al., 2014; SOFRÁ & BOGER, 2011).

High-density thickened tailings have as advantages low moisture content, homogeneous nature and no segregation of particles. The pastes are non-Newtonian fluids and usually exhibit Bingham fluid behavior, with low yield stress (minimum of 200 Pa) and pseudoplastic features (BOGER, 2013; FITTON & SEDDON, 2013). The yield stress is a crucial component in the rheological characterization of thickened tailings, because beside marking the transition between pulps and pastes, it affects factors such as the transportation energy requirements and the deposition slope (BOGER, 2013; SOFRÁ & BOGER, 2011).

## 2. Objective

This study investigated the rheology of tailings generated in the reprocessing of nickel tailings, submitted to flocculation and thickening under various conditions, evaluating flocculant type and dosage, in order to obtain efficient dewatering operations able to simultaneously achieve multiple properties: high water recovery (qualitative and quantitative) and paste production. The viability of this study will allow the reduction of the environmental impacts caused by the conventional tailings disposal, besides increase process water recycling.

## 3. Material and Methods

The materials used in the experimental study were: a tailings sample produced by reprocessing of nickel tailings, obtained from a tailings dam in the state of Minas Gerais, Brazil, and as flocculants, two anionic polymers manufactured by BASF, referred to as R-10 and R-90. The pulps were prepared and submitted to flocculation and discontinuous sedimentation tests to evaluate the influence of the initial solids concentration ( $C_0$ ), and of the dosage (D-pol) and type of flocculant, on the sediment cohesion. The experiment had a complete factorial experimental design with two variables, three levels and triplicate at the central point (Table 1).

Table 1. Complete factorial experimental design, for both flocculants, R-10 and R-90.

Variables	-1	0	+1
$C_0$ (w/w)	10	15	20
Flocculant dosage (g/t)	40	60	80

### 3.1. Characterization of mineral tailings and flocculants

The flocculants and nickel tailings were characterized according to the surface charge density, with a Malvern Zetasizer Nano series, in the pH range of 2 to 12, and an indifferent electrolyte solution, KCl 0.01 M. The mineral tailings' particle size distribution was measured by laser diffraction with a Malvern Mastersizer 2000.

The flocculants were characterized according to the viscosity-average molecular mass ( $M_v$ ) with a Haake RheoStress 1 rheometer. Viscometry is a technique used to obtain data related to the size and conformation of macromolecules (MELLO et al., 2005). The viscosity of infinitely dilute solutions comes from obtaining the intrinsic viscosity  $[\eta]$ , which can be obtained by several mathematical equations described by Mello et al. (2005). We used the equations of Huggins and Schulz-Blaschke (Equations 1 and 2) to determine this value by graphical extrapolation, and the equation of Solomon-Ciuta (Equation 3) to determine it by the single-point method.  $M_v$  was determined using the equation of Mark-Houwink-Sakurada (Equation 4).

$$\eta_{sp}/c = [\eta]_H + K_H [\eta]_H^2 c \quad (1) \quad \eta_{sp}/c = [\eta]_{SB} + K_{SB} [\eta]_{SB} \eta_{sp} \quad (2)$$

$$[\eta]_{SC} = [2 (\eta_{sp} - \ln \eta_r)]^{1/2} / c \quad (3) \quad [\eta] = K_M M_v^a \quad (4)$$

### 3.2. Flocculation and thickening test

The pulps were prepared with nickel tailings and tap water and flocculated in a jar test device - by Nova Etica, model 218-6. The flocculant solution was then added to the slurry and stirred at 300 rpm for 1 min, followed by slow stirring at 150 rpm for 2 min, to avoid excessive mixing after flocculation, and consequently, the breakage of the flocs. The flocculated pulp was transferred to the 2 L graduated cylinder ( $H = 48$  cm;  $D_{in} = 7.5$  cm) for discontinuous sedimentation test. The supernatant was analyzed with 1h and 2h of sedimentation, and the cylinder was set aside undisturbed for 24 h. After that, the sediment was studied to determine its properties.

### 3.3. Characterization of thickened tailings

The sediments characterization was carried out according to the rheological and slump tests. The slump test is used to determine materials' consistency, and does not require any sophisticated instrumentation. Conventionally, this technique shows good results in literature to evaluate the consistency of mineral suspensions (CLAYTON; GRICE & BOGER, 2003; CREBER et al., 2017). We applied the same technique used by Clayton; Grice & Boger (2003), using a cylindrical geometry of PVC, with  $H = D_{in} = 2$  in.

Yield stress ( $\tau_0$ ) is the minimum stress required for the thickened tailings to flow. Thus, it is an important property to study the dewatering process efficiency (SOFRÁ & BOGER, 2002).  $\tau_0$  can be estimated from different rheometry techniques. However, two types of rheological analysis are commonly employed for this purpose. One is the direct method, in this case the torque vs. time curve is obtained, and the maximum point is when the material yields, equivalent to  $\tau_0$ . The other technique is the regression method based on shear stress vs. shear rate curve (BOGER, 2013; SOFRÁ & BOGER, 2002, 2011). We used both techniques for  $\tau_0$  measurement, by using the vane sensor. The major advantages of using the vane sensor include the lower sample disturbance/breakdown when inserting rotor, minimizing the effects for wall slip, and the larger gap, reducing errors related to large particle size (BOGER, 2013; SOFRÁ & BOGER, 2011). Therefore, the  $\tau_0$  was obtained for the bottom and top of sediment formed in the cylinder, using a Haake RheoStress 1 rheometer, and the Herschel-Bulkley model (Equation 5) to fit the experimental results and obtain the  $\tau_0$  value by regression.

$$\tau = \tau_0 + K \dot{\gamma}^n \quad (5)$$

First, the  $\tau_0$  was obtained by the direct method applying a constant shear rate of  $0.1 \text{ s}^{-1}$  for a maximum time of 300 s. These data are referred to as static  $\tau_0$ . After this, the sediment was subjected to a high shear,  $100 \text{ s}^{-1}$ , in order to destroy the floc structure and release the contained water. This new sediment had its yield point analyzed by both methods and is referred to as dynamic  $\tau_0$ . The direct method was the same as the previous one; and for plotting the flow curve, the shear rate was  $0.01$  to  $100 \text{ s}^{-1}$  and a maximum time of 5 min per point.

The sediment concentration ( $C_{\text{sed}}$ ) was obtained indirectly by a moisture analyzers, OHAUS - MB23. Therefore, after the rheological analysis, the sample was subjected to heating, allowing percent solid determination.

## 4. Results and Discussion

### 4.1. Characterization of nickel tailings and flocculants

Various physical and interfacial chemistry factors influence the evaluation of the rheological properties of sediments. Therefore, they are increasingly exploited to optimize operation and production of paste. The nickel tailings had very fine granulometry: particles smaller than  $28.3 \mu\text{m}$  accounted for 90%,  $5.9 \mu\text{m}$  accounted for 50% and finer than  $1.3 \mu\text{m}$  represented 10% of the total amount of tailings. In general, the smaller the particles, the higher the  $\tau_0$  of the slurry, because small particles have larger specific surface area, and consequently, greater area for inter-particle interaction (SOFRÁ & BOGER, 2011). Thus, the surface chemistry has more influence on rheology. A way to evaluate the surface chemistry is by measuring the zeta potential. It can be seen in Figure 1(A) that in pulp with natural pH ( $7 \leq \text{pH} \leq 8$ ), the particles were negatively charged, leading to strong electrostatic repulsion. As described by Sofrá & Boger (2011), the maximum Van der Waals attraction occurs at the isoelectric point (iep), with a high  $\tau_0$ , and away from the iep, the particles are electrostatically repulsed, leading to a low  $\tau_0$ . This phenomenon is even more pronounced at high solids concentrations, which is the case of pastes. It can also be observed in Figure 1(A) that both polyelectrolytes used as flocculants, R-10 and R-90, have high anionic charge density, thus, the mechanism of particle aggregation involves the formation of bridges (GREGORY & BARANY, 2011). However, it was observed that the R-90 has a higher anionic charge density than R-10. This factor may influence the particle aggregation efficiency, because its high anionic charge density increases the anionic character of the medium and consequently increases the electrostatic repulsion.

The Figure 1 (B) shows the extrapolation graph of Huggins, which exhibits the linear relation for the determination of the intrinsic viscosity of the polymers (MELLO et al., 2005). It can be observed in the Table 2 that all applied equations, showed similar values of  $[\eta]$ . Therefore, the measurement techniques used allowed comparing the viscosity average molecular mass of the polymers. For the calculation of the molar mass by Equation 4, the constants  $K = 3.7 \times 10^{-5} \text{ L/g}$  and  $a = 0.66$  were employed. These values are defined by Clark; Herrington & Petzold (1990) for anionic polyacrylamide, and this being the basis of their chain, these values were used to level comparison. Concluding that the polymers (R-10 and R-90) have similar molar mass, so, the main difference between the polymers must be in the anionic charge density.

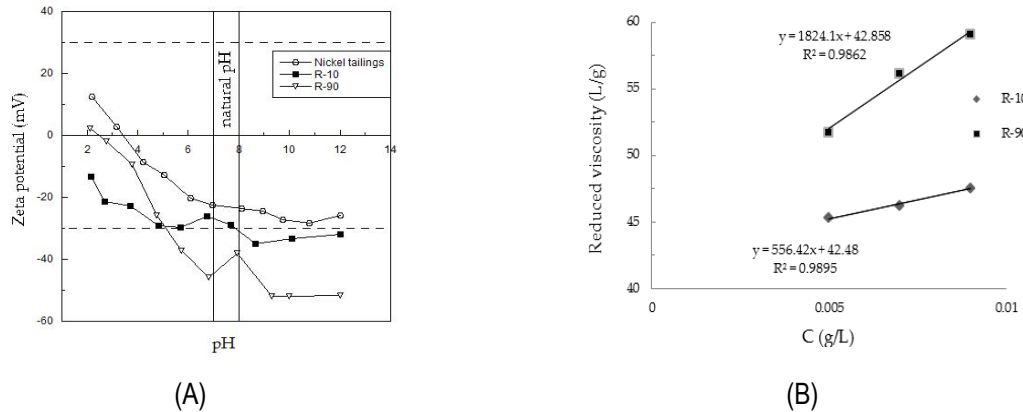


Figure 1. Characterization. (A) zeta potential of the nickel tailings; (B) Huggins viscosity for polymers.

Table 2. Intrinsic viscosity values and molar mass determined for the polymers.

Polymer	$[\eta]_H$ (L/g)	$[\eta]_{SB}$ (L/g)	$[\eta]_{SC}$ (L/g)	$M_v \times 10^{-6}$ (g/mol)
R-10	42.48	42.76	42.30	1.74
R-90	42.86	45.41	47.84	1.76

#### 4.2. Characterization of thickened tailings

As reported in the literature, there is typically variation of the solids concentration in the sediment compaction zone, and the flow properties of concentrated mineral suspensions varied significantly with solids concentration (FRANÇA; BISCAIA & MASSARANI, 1999; SOFRÁ & BOGER, 2002, 2011). Thus, the sediments were analyzed at its extremities (top and bottom), allowing the acquisition of extreme values  $\tau_0$  and  $C_{sed}$  in each case analyzed. Figure 3 (A) shows that the measurement techniques used (direct method and by regression) produced very similar values, mainly for high values, as reported by Boger (2013). Figure 3 (A) also shows the increase of the static and dynamic  $\tau_0$  for the bottom sediment in relation to the top. This behavior is due to the existing concentration gradient in the sediment. In this study, the  $C_{sed}$  presented a variation of up to 38% (w/w) between top and bottom, with the lower and higher observed values of 37 and 60% w/w, respectively. Figure 3 (B) and 3 (C) illustrates the strong dependence of the yield stress on the value of  $C_0$  and the type and dosage of flocculant polymer. The curves presented in Figure 3 (B) show that increase of  $C_0$  led to higher values of  $t_0$  for the sediment. This occurred because the difference in  $C_0$  influences the shape and structure of the flocs formed, and consequently the settling velocity and sediment cohesion (JARVIS et al., 2005; PARSAPOUR et al., 2014). On the other hand, it can be seen in Figure 3 (C) that the increase of the polymer dosage influenced the static  $t_0$  but did not change the values of dynamic  $t_0$ , these being functions of  $C_0$ . Thus, it can be said that the increase of the polymer dosage led to storage of more water inside the flocs, reaching higher values of static  $t_0$ , and after the destruction of the floc structure, the sediments exhibited the same behavior for each flocculant. Between the two polymers, R-10 reached higher values of static and dynamic  $t_0$ , of 1589 Pa and 64 Pa, respectively, for pulps with 20% (w/w)  $C_0$ . The different efficiencies between the studied polymers can be explained by the polymer-slurry interaction difference, i.e., extensive study is necessary for a better understanding.

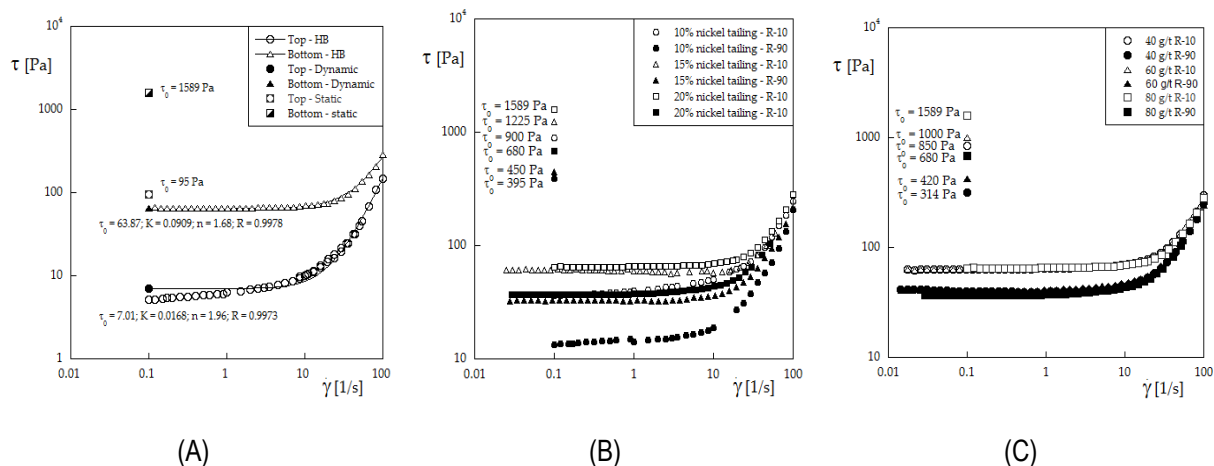


Figure 3. Rheological characterization, static  $\tau_0$  and flow curve. (A)  $C_0 = 20\%$  and  $80 \text{ g/t R-10}$ ; (B) D-pol =  $80 \text{ g/t}$ , varying  $C_0$ ; (C)  $C_0 = 20\%$ , varying the D-pol.

The curves presented in Figure 4 show the exponential growth of  $\tau_0$  with  $C_{sed}$  (SOFRÁ & BOGER, 2002, 2011). The dynamic  $\tau_0$  (Figure 4-A) reached maximum values of 64 and 40 Pa, for R-10 and R-90 respectively, indicating that in the case of complete destruction of the flocs, the thickened tailing do not achieve paste consistency. However, for paste transportation, a positive displacement pump is used instead of a centrifugal pump. Thus, it is certain that the floc destruction will be much less intense, and need to be analyzed in the near future. It can be seen in Figure 4-B that the static  $\tau_0$  has values higher than 200 Pa for the bottom sediment, paste consistency, and illustrates inverse relationship of %SH with  $\tau_0$  and  $C_{sed}$ .

Regarding the water quality, the flocculation/sedimentation of the nickel tailing led to reduction of the overflow turbidity up to 98%, compared to natural sedimentation. Recovered water presented turbidity less than 100 NTU (Figure 4-C) that according to the Brazilian standards, allows its reuse or disposal in the environment.

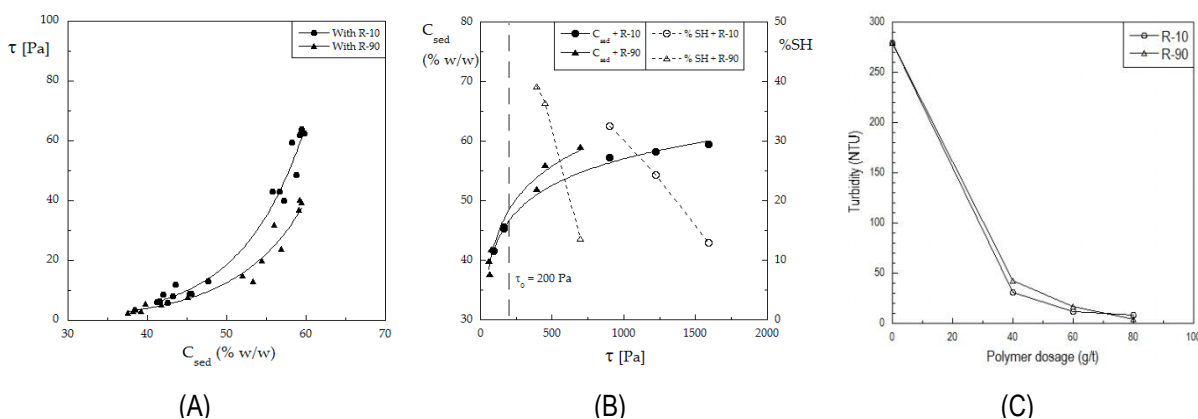


Figure 4. Relationship. (A) dynamic  $\tau_0$  and  $C_{sed}$ ; (B) static  $\tau_0$ ,  $C_{sed}$  and %SH for D-pol =  $80 \text{ g/t}$ ; (C) turbidity and D-pol, for  $C_0 = 10\%$ .

## 5. Conclusion

This paper presented and discussed the effect of flocculation and sedimentation of the nickel tailing using new generation flocculants, R-10 e R-90, which are rheology-modifying agents. In all cases studied the underflow had

static  $\tau_0$  above that specified for pastes. Therefore it can be said that both flocculants were efficient for nickel tailings dewatering. However, the use of polymer R-10 as flocculant allowed the formation of more cohesive sediments, compared to R-90, that is, with higher values of static  $\tau_0$  and  $C_{sed}$ , and lower values of %SH. The results provide the mining company a new alternative for the management of their tailings, i.e., will allow the tailings reuse as source of raw material, together with the safest disposition: as paste, which is more environmentally acceptable than storage behind conventional tailings dams. Therefore, the densification process studied is efficient in particle aggregation, allowing the reduction the amount of free water in the dams, which reduces the risk of environmental accidents, besides allowing the process water recovery.

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