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Effect of sample manipulation on the Couette rheometry of copper concentrates

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ABSTRACT

The measurement of slurry rheology is a key factor in ore concentrate pipeline design and operation. In addition to the known effects of the input data, the manipulation and the particular conditions of the experimental apparatus may significantly affect the measured flow curves. In the present work, an experimental study of the influence of some of these factors in copper concentrate rheology was performed. In particular, the impact of sedimentation, hydrodynamic segregation and inertial effects is assessed through wide-gap Couette rheometry experiments using an automated, variable-speed sample-loading mechanism. Two types of experiments were performed by controlling the sample loading speed: the generation of an angular velocity ramp and individual torque measurements with constant angular velocities after an impulsive start. Two concentrations, on the order of those corresponding to typical turbulent transport of ore concentrates, were tested. The flow curves obtained from the first set of experiments show slight differences for loading times between 1 and 10 s. However, differences close to 100% were observed when the results obtained with loading times in the order of 1 s were compared with the results obtained with loading times of approximately 1 min. The individual torque measurements, which were represented by the maximum value within the corresponding time series, show some previously documented features of hydrodynamic segregation in monodisperse suspensions; however, consistent effects were obtained with the different sample loading times and angular velocities tested. Using scaling arguments, a set of five dimensionless groups including gravity, momentum, particle concentration diffusion and flow inertia, assessed by means of the sample loading time and the angular rotation of the inner cylinder, is proposed. Fitting such dimensionless numbers to the relative difference between the measured torques and those that should be obtained under purely homogeneous and inertialess conditions revealed a decreasing dependence of the sample loading time and increasing effects of hydrodynamic segregation and angular velocity. In particular, the present findings suggest that hydrodynamic segregation has a significant influence on the effective measurement of torque; this finding is consistent with previous results obtained with neutrally buoyant monodisperse suspensions. The experimental data suggest that the dimensionless torque may be interpreted as the result of a nonlinear interaction between the sample loading and the transient start of the rotation of the inner cylinder.

1. Introduction

The measurement of ore concentrate rheology is a crucial procedure in mineral transport and thickener design and operation. In the case of long-distance pipelines that transport many different types of ore slurry, including copper, iron and nickel concentrates, the rheology is commonly one of the first inputs that are used to define the pipeline diameter and the thickness distribution along the tube [1–5]. In addition, the slurry rheology is often relevant in the definition of operational characteristics of pipeline systems, such as maximum stop and start-up times [6]. The accurate measurement of rheology is becoming increasingly important in systems that operate at relatively high concentrations, especially those in places where water is scarce. This type of systems is often found in many Chilean and Australian desert locations, where it has been shown that slight inaccuracies in the rheological characterization may propagate to significant errors in slurry pressure ratings and power consumption in pipelines that are a-few-hundred-kilometers long [7]. The design of systems with increasingly higher solid concentrations requires the assessment and control of different measurement parameters in the laboratory, which include the concentration, the particle size distribution and the weak inter-particle forces through chemical characterizations of the slurry [8–11], as well as the geometrical and manipulation conditions, that may severely distort the experimental output [12].

Rheometry in mining operations has the inherent complexity of liquid–solid flows: solids are likely to re-distribute in their containers and depart from their initially homogeneous suspensions. Ore

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concentrates and tailings witness the combined effect of a solid concentration [13], weak electrokinetic forces [13,9], thixotropy [14] and polydisperse particle sedimentation [15]. The impact of these processes is mostly observed on the viscosity measurements. Despite the fact that they present nonzero yield stress values, the latter are often low compared with those that exist at the thickening stage due to the typical volume fraction range at which these concentrates are transported, i.e., close to 0.3. The corresponding yield stress values are on the order of 1 Pa or lower for typical values of d_{50} that are close to 50 μm [16–18]. It was recently identified that sheared dense suspensions tend to segregate according to the prevailing exerted shear stress ([19,20,10], and references therein). These elements pose different restrictions on rheometry. Thixotropy has been frequently found to be responsible for the time-dependent behavior of many fluids, including suspensions, such as clays [21]. This process has been explained as an elastic deformation when the stress is lower than the yield value and a subsequent destructuration to ultimately obtain an equilibrium state between destructuration and restructuration [22,23,14,21,24]. For instance, using a 6% w/w bentonite suspension (yield stress of approximately 6 Pa and d_{50} value close to 10 μm), Bekkour et al. [25] obtained local maxima in the shear stress–time curve before the first second of shearing at shear rates commensurate with 1 s^{-1}, whereas local maxima were found to occur at times commensurate with 10 s for shear rates lower than 0.1 s^{-1}. A similar trend was observed by Klein and Hallbom [20] with nickel laterite suspensions with a d_{50} value of approximately 5 μm. In contrast to the referred measurements, copper concentrate and tailings have larger average particle sizes. In general, the mineral extraction process requires average particle sizes on the order of 50 μm (and not below 10 μm) for close-to-optimal copper recovery [26]. At this particle size range, Brownian forces are negligible, and weak inter-particle forces become progressively less important compared with those of a hydrodynamic nature. In the transportation of solids at Reynolds numbers that exceed approximately 10^4, colloidal forces are usually unimportant [27,28], as can be demonstrated through dimensional analysis [29,10,30]. In this regime, the aforementioned shear-induced particle migration effect, which can be described through a hydrodynamic diffusion process, becomes increasingly relevant at greater mean particle sizes and shear rates [31,19]. Despite the relevance of the ore rheological output at high concentrations, this mechanism is often overlooked in the definition of the measurement methodologies and in the post-processing phase.

In the present paper, the effect of hydrodynamic segregation through sample manipulation and rheometer sequencing was analyzed through a set of experiments with commercial copper concentrate samples. This work was motivated by the conjecture that sample loading, ramp sequencing programming and/or individual torque measurements in the cup and bob geometry affect the shape of the flow curves and/or the time dependence of the torque signals. It is also known that different measurement techniques yield different results [32], which indicate that the apparatus setup and procedure are critical for repeatability and accuracy.

As a result of some preliminary measurements using a standard Couette cell, several flow curves were obtained using copper concentrate at random cup loading speeds in the range of a few to several seconds. After obtaining the rheological parameters, differences on the order of 10% or higher, depending on the solid concentration, were found for both the Bingham plastic viscosity and the yield stress with the same samples during the execution of identical pre-conditioning procedures. In contrast, when the same sample loading times were re-examined, the typical differences in the triplicate measurements were less than 3%. To gain insight into this mechanism, a set of well-controlled measurements of the time progression of shear stress at different slurry concentrations in a cup and bob Couette cell was performed. The results are interpreted in light of different phenomena that may have a relevant impact on the results; as described by Chow et al. [33], these phenomena include particle migration due to sedimentation, hydrodynamic segregation and inertial effects induced by the non-negligible flow accelerations triggered by sample loading [33].

2. Particle migration

Although copper concentrates and other non-colloidal high concentration slurries handled in the mining industry are most commonly modeled as homogeneous mixtures, these slurries are prone to segregation, which induces local solid concentration distributions and thus density and viscosity fields. This segregation can occur due to the effect of gravity [34] and can be induced by the flow itself via various mechanisms, including self-diffusion [31,35] and shear-induced particle segregation [36,19]. The latter case occurs in the presence of local gradients of concentration and shear stress. Different from sedimentation, particle segregation occurs even in the presence of neutrally buoyant particles, and its effect is much stronger than that of self-diffusion, which is relevant in polydisperse sedimentation [35]. In the presence of an imposed mean shear rate, particles do not strictly follow streamlines and are instead exposed to hindrance effects due to the presence of their neighbors, which alters their trajectories and diameters commensurately. This interaction is irreversible, and the resulting dynamics lead to chaotic trajectories of the solid matter, despite the fact that the corresponding Reynolds numbers are often very low [37]. Because the number of particles is very large, their motion can be described by a Gaussian function, where the typical particle radius a scales with diffusion coefficient $\hat{D}$ and the time t through the relationship $2\sqrt{\hat{D}t/D}$, where D is a dimensionless function of the concentration. In the case of an imposed Couette flow, both the particle size and the imposed shear rate $\dot{\gamma}$ remain relevant variables. In particular, the diffusion coefficient scales as $\hat{D}{\sim}a^2\dot{\gamma}D$. Leighton and Acrivos [19] proposed that $D=K\phi^2/\eta(d\eta/d\phi)$ for neutrally buoyant monodisperse spheres, where K is an order 1 function of the solid concentration and η is the viscosity of the suspension, which is a function of the solid volume fraction ϕ. Using neutrally buoyant sphere suspensions, the diffusion coefficient was fitted in the context of a shear flow induced in a parallel plate device using the following relation [38]:

$$D=\frac{1}{3}\phi^2\left[1+\frac{1}{2}exp(8.8\phi)\right], \tag{1}$$

which is also commonly considered a measure of the dimensionless particle diffusion coefficient. Although the diffusion process is better represented by an anisotropic tensor [39,40], the relation (1) gives a reasonable overall approximation of the hydrodynamic diffusion process. Based on the concepts described by Leighton and Acrivos [19], Phillips et al. [41] proposed an empirical, two-parameter mixture model — later extended by Shauly [42] to polydisperse suspensions — to describe the evolution of the concentration field, which includes a term to account for particle collisions. On the other hand, to describe the dependence of the viscosity on the concentration of a dense suspension of spheres, the Krieger model [43,12,44] is most commonly used:

$$\frac{\eta}{\mu}=\left(1-\frac{\phi}{\phi_m}\right)^{-X}, \tag{2}$$

where ϕ_m is the maximum settled concentration of the suspension, μ is the dynamic viscosity of the liquid and X is an empirical parameter. Other approaches, including empirical and semi-empirical models for the viscosity of multimodal particle distributions, are discussed by Qi and Tanner [45]. Ovarlez et al. [46] analyzed suspensions of nearly neutrally buoyant spheres and found that hydrodynamic segregation occurs at very short times. Based on their measurements, these researchers proposed that the mixture behaves as a purely viscous mixture beyond a critical shear rate and concluded that, even after