Settling velocities of particulate systems 18: Solid flux density determination by ultra-flocculation

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A B S T R A C T
The scale-up of thickening parameters from laboratory to industrial plant is still an open problem. Several techniques have been in use but none is free of criticism. In order to clarify these issue, in this work flotation tailings from one of the major Chilean copper mines was subjected to flocculation-settling tests with Orficol-2010 polyacrylamide in a Couette type reactor. By varying the shear rate from 100 to 2000 [s⁻¹] the solid concentration from 1 to 15 [% by volume] and the flocculant dosage from 0 to 20 [g/ton] it was shown that an important interaction exists between these variables. At the optimal flocculant dosage, the optimal suspension concentration and the optimal flocculation time, an increase by 50% in the solid flux density function is possible when the shear rate of γ = 100[s⁻¹] is changed to the optimum value of around γ ≈ 400[s⁻¹].

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1. Introduction

In many countries water has become a scarce commodity. The necessity of recovering and recycling water has turned thickening into an important process, requiring the best operational practice. This objective requires improvement in the control system that maximizes thickener underflow concentration, minimize flocculant consumption and, in general, permits the stabilization of the whole operation. Several feedback control systems have been proposed for thickening operations, unfortunately, due to the non-linearity of the process and to the slow response to perturbations, thickening is difficult to control and classical control systems fail to operate adequately. Even more importantly, current methods of thickener control are based on feedback to change operational variables that do not take into consideration the properties of the feed material. Two such parameters are the solid flux-density that expresses, for any concentration of a suspension, the product of the settling velocity and of the solid concentration (and, for a material of constant concentration, represents the momentum of the motion), and the solid effective stress that represents the compressibility of the sediment produced by settling. These parameters permit a phenomenological description of the thickening process. Models using these two parameters permit improved operation and optimal control strategies. Determination of these parameters is still an open problem (Bürger et al., 1999). In this paper we will discuss the determination of one of these parameters, the solid flux-density function.

Several techniques have been proposed to determine the settling velocity in laboratory experiments, the “jar tests” being the most common (Coe and Clevenger, 1916; Richardson and Zaki, 1954; Michael and Bolgers, 1962). The Jar test involves homogenization of suspensions varying solid concentrations in settling cylinders, introduction of the flocculant and mixing by moving a plunger up and down in the cylinders, or by inverting the cylinders several times. This procedure is claimed not to be satisfactory because of the local over-dosing that can occur when the relatively concentrated flocculant solution meets the slurry (Kitchener, 1978), but more important is that the agitation obtained by this method does not produce the optimum flocculation. Farrow and Swift (1996) show that jar test has three main inconvenient: (1) the flocculation efficiency, measured by the sedimentation results, depend strongly on the mixing method (numbers of inversions), (2) that the diameter of the cylinders have a significant effect on the settling rate, diminishing as the diameter of the cylinder increases and (3) the experimental reproducibility associated with the process is low, with standard errors of the mean from 8 to 12% for the settling velocity.

Flocculation is used to aggregate fine particle to maximize the solid flux-density, which is directly related to thickener capacity. Hogg et al. (1993) showed that the choice of a flocculant is determined by chemical factors such as the mineral composition and solution chemistry. But...
floculant adsorption and bridging flocculation take place simultaneously and dynamic variables, such as the intensity of mixing and the mixing time, affect particle-particle linkages (Keys and Hogg; Hogg, 1999). Molecular weight of typical the mixing time, affect particle-particle linkages (Keys and Hogg; Hogg, 1999). Molecular weight of typical flocculants fall in the range of 10–20 million Daltons. The transfer of the floculant from the stock solution into the suspension and distribution within the dispersed system takes some time. An improved procedure to assess the efficiency of flocculation is via the use of a shear vessel, which is similar to a rotational Couette viscometer and has the advantage of quantifying the mixing quality through the shear rate. Several investigators have used the shear vessel in the past in coagulation experiments (Ives and Bhole, 1977; Smith and Kitchener, 1978; Stein et al., 1986) and in flocculation studies (Muhle and Domasch, 1991; Farrow and Swift, 1996; Rulyov, 1999, 2004; Rulyov et al., 2005a, 2005b; Rulyov et al., 2009).

Farrow and Swift (1996) designed their shear vessel with concentric cylinders of 200 and 210 [mm] in diameter and 120 [mm] in length (Fig. 1). At the bottom of the vessel a glass tube 14 mm in diameter and 220 [mm] in length was used to measure the settling velocity. The experiments were carried out at a constant rotational velocity of 200 [rpm]. The outflow of the shear vessel was introduced immediately in the settling column, depicted as A and B in the figure. The authors conclude that the combination of shear vessel and settling column overcame most of the problems associated with the jar test, in particular the strong dependence of batch settling test on mixing rate and cylinder diameter.

It was shown by Rulyov (1999) and Rulyov et al. (2000) that the mixing time in flocculation can be reduced down from minutes to 5–6 s by the appropriate hydrodynamic treatment of the suspension in a shear vessel at high shear rate. This treatment, termed “ultra-flocculation” (Rulyov, 2004; Rulyov et al., 2005a, 2005b), ensures that, not only floculant macro molecules are quickly and evenly distributed within the suspension and adsorb onto the surface of the particles, but also provides for the formation of large and dense flocs. Depending on the size distribution and density of the solid particles in the dispersion, as well as on their volume concentration, the optimum values of the mean shear rate γ may vary in a wide range such as 300–γ ≥ 5000 [s⁻¹]. The significant advantage of ultra-flocculation is that it ensures a good mix of small and large particles in flocs before they get into the settler, thus providing for fast sedimentation and high degree supernatant clarification (Rulyov et al., 2009). It was recently shown that under certain conditions intense agitation for short times may even change the nature of flocculation, from total flocculation to a selective flocculation of only some mineral constituents (Ding and Laskowski, 2007).

In this work an instrument called UltrafloFinder has been used, which combines a shear vessel with variable shear rate and an optoelectronic device that measures the fluctuation via intensity of the light beam passing normally through the transparent tube, while the formed flocs pass through this tube, to (1) analyze the relationship of flocculation efficiency (or mean flocs size) with solid concentration, floculant dosage and shear rate, (2) the effect of the concentration and shear rate on the settling velocity and solid flux density and (3) the effect of the solid concentration on the optimum shear rate for flocculation.

2. Material, experimental set-up and method

Flocculation tailings from one of the major copper flotation plants in Chile were used in all the experiments. Solid concentration was varied over the range from 1.8 to 15 [% by volume] (4.7 and 32.3% by weight); material density was 2700 [kg/m³]. An average particle size x₅₀ = 40μm with size distribution characterized by x₀ = 20μm and x₃₈ = 0.5μm, was determined using a Sympatec Helos-Rhodos laser dispersion instrument. Orifloco-2020 polyacrylamide was used as a floculant with a molecular weight of 9.64 × 10⁶ [g/mol].

The set-up to perform the ultra-flocculation tests is shown in Figs. 2 and 3. It consists of a small shear vessel, referred to as ultra-flocculator in Fig. 2. This Couette reactor, with a rotating cylinder of 28 [mm] in diameter and a gap of 1.5 [mm] was fed continuously with the suspension of tailings by a measuring peristaltic pump. Before entering the Couette reactor the pulp receives continuously a diluted flocculant solution, at a flow-rate to give a pre-determined dosage. After 6 s treatment at a pre-determined shear rate in the Couette reactor, the flocculated suspension is discharged from the ultra-flocculator through a 3 [mm] inner diameter transparent tube equipped with an opto-electronic sensor which registers the fluctuation of intensity of the light beam passing normally through the tube [in accordance with techniques proposed by Gregory and Nelson (1984)]. The electronic signal is processed and displayed in a three digital format, thus showing, in
relative units, the values of flocculation efficiency (or mean flocs size) and the mean shear rate $\dot{\gamma}$.

The operational conditions consisted in changing the flocculant feed rate and the shear rate while maintaining a constant treatment time of the suspension in the Couette reactor (6 s). When the feed suspension concentration exceeded the threshold of 6% by volume of its optical analysis capacity, it was diluted by introducing clean water between the shear reactor and the optoelectronic sensor (shown by a dash line in Fig. 1). In the tests designed to measure settling rate of the treated suspension, dilution was not used. In this case the suspension from the outlet of the tester was continuously fed to a 14 mm diameter settling cylinder and, as soon as the suspension filled the cylinder, it was one time inverted and the suspension allowed settling and the initial settling velocity was measured.

3. Results and discussion

Table 1 shows the operational conditions of the experiments and the output of the instrument.

(In Table 1, $v_{opt}$ and $v_{100}$ are the initial settling velocity of the suspension after treatment, at optimal shear rate $\dot{\gamma}_{opt}$ and at shear rate equal to $\dot{\gamma} = 100$ [s$^{-1}$]. $f_{soc, opt}$ and $f_{soc, 100}$ are the corresponding solid-flux densities.)

### 3.1. Effect of the flocculant dose on the efficiency of flocculation

The flocculation was carried out over 6 s at optimal values of the mean shear rate $\dot{\gamma}$. (for the respective suspension concentrations see Table 1). Fig. 4 demonstrates that the flocculation efficiency increases monotonically with flocculant dosage, reaching the relative value of 90 with a dosage of 10 [g/ton] for the low range of particle concentration, and 20 [g/ton] for the higher range. The observed increase in the flocculant dosage with the increase in the suspension concentration can most likely be attributed to the slowdown of the process of the flocculant macro molecules distribution within the volume of the suspension with the increase in solid concentration.

### 3.2. Effect of the shear rate on the efficiency of flocculation

Fig. 5 shows the effect of shear rate $\dot{\gamma}$ on the flocculation efficiency (or mean flocs size) and clearly demonstrate that a maximum exist between 400 and 600 [s$^{-1}$] with higher values for increasing concentration. The shift of the maximum flocculation efficiency to higher shear values for higher flocculant dosages may be due to the increased strength of the bridges bonding particles within a floc, as shown by Rulyov et al. (2005a, 2005b). (The operational principle of the ultra-flocculator is based on the optoelectronic measuring system proposed and described by J. Gregory and D.W. Nelson. J. Gregory and D.W. Nelson “A new method for flocculation monitoring in Solid–liquid Separation” (J. Gregory / Ed. Ellis Horwood, Chichester, 1984, pp.172–182). The reading of the device is proportional to the average size of flocs and, since these readings are not in length units, they were called «floculation efficiency».

### 3.3. Effect of the shear rate on the settling velocity

Since the shear rate influences the flocculation efficiency (or mean flocs size) in the way expressed in the previous sections, one would expect a similar influence on the settling velocity. This was confirmed as shown in Fig. 6. The results provided in this figure indicate that the optimum shear rate corresponding to the maximum flocculation efficiency also corresponds to the maximum initial settling velocity of the flocculated suspension, thus the ultra-flocculation test is an effective method for identification of the optimal flocculation conditions.

### 3.4. Effect of the solid concentration on the optimal shear rate

It is important to establish the optimum solid concentration for flocculation in a commercial thickener. In the majority of industrial thickeners flocculation is perform in the feedwell where the feed is diluted with up-coming and circulating water. Knowing the solid concentration that gives the best flocculation should permit calculation of the water dilution flow rate.

Fig. 7 shows the effect of the suspension volume concentration on the optimum shear rate for a given flocculation. Interestingly, at a certain solid concentration value, the dependence of optimal shear rate on solid volume concentration has a minimum, which shows that for much diluted and very concentrated suspensions higher shear rate must be used. This relationship between shear rate and solids concentration can be explained using Smoluchowski theory because, at a given suspension concentration, the floc size increases to a maximum within a short time interval. On the other hand, with the increase in suspension concentration the distribution of flocculant macromolecules within the volume of suspension slows down. In particular, this is confirmed by the increased consumption of the flocculant with the increased suspension concentration at a constant time interval. However with increasing shear rate, due to convective diffusion, the rate of flocculant molecules dissemination in the suspension significantly increases, leading to the growth of the dependence of the optimum shear rate on concentration in the region of large concentration values. This may also lead to some decrease in the required flocculant dosage as shown by Rulyov et al. (2005a, 2005b).

![Fig. 3. Photograph of the UltraflocTester: (Model: UFT-TFS-029 Turboflotservice Company).](image)

<table>
<thead>
<tr>
<th>Solid concentration [g/l]</th>
<th>Concentration [%] solid by volume</th>
<th>Settl. vel. $v_{opt}$/v$_{100}$ [mm/s]</th>
<th>Shear rate $\dot{\gamma}$ [s$^{-1}$]</th>
<th>Flocculant dose [g/ton]</th>
<th>$C_{f soc} &gt; 10^5$ [g/cm$^2$·s]</th>
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<td>600</td>
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</table>
3.5. Effect of the shear rate on the flux-density function

The best way to show the effect of shear rate on sedimentation is to plot the flux-density function versus the suspension concentration for several shear rates at the optimum flocculant dosage. This is plotted in Fig. 8.

If one takes into account that flocculation in a commercial thickener is generally performed at shear values lower than $\gamma = 100$ [s$^{-1}$], the results of this work reveals an interesting improvement that could be obtained by ultra-flocculating the feed before entering the thickener.

3.6. Effect of the shear rate on the supernatant clarity

It is well established and known that in the process of ultra-flocculation treatment «supernatant clarity» is directly related to «flocculation efficiency», so in the context of the paper this information has been regarded as redundant.

4. Application to industrial thickeners

Solid–liquid separation of tailings in thickeners relies on the efficient mixing of slurry and dilute flocculant solution in the tube conveying the pulp to the thickener or within the feedwell (Owen et al., 1999). In both cases the mixing, measured in shear rates around 10 to 20 [s$^{-1}$], is too low for efficient flocculation. One of the major recent improvements is thickener feedwell design, for example, Outotec company on its webpage claims that in the “Vane Feedwell the upper zone, into which feed, dilution water and flocculant are added, provides enhanced mixing and energy dissipation. This maximizes flocculant adsorption, eliminates the possibility of coarse-fines segregation and ensures all particles are aggregated together by the flocculant. Efficient operation is maintained in this upper zone over varying feed rates. The lower zone promotes gentle mixing for continued aggregate growth, with the option for secondary flocculant dosing. This zone also enables aggregates to uniformly discharge under low shear conditions”. Unfortunately there is no indication of the shear rate in the feedwell.

Based on its Laboratory Ultra-Floculator, Rulyov et al. (2009) developed industrial sized Ultra-floculators, such as the one depicted in Fig. 9. Fig. 10 shows the flocculation efficiency versus the shear rate of a 200 [m$^3$/h] for the quartz suspension treated by this ultra-flocculation.

5. Conclusions

Proper addition and mixing of a flocculants with the pulp is one of the essential steps in the flocculation process. Optimal dosage of flocculant and the correct suspension concentration are not sufficient to yield a good flocculation. The shear rate at which flocculation is performed may decide on the quality of the process. Up to recently it has been believed that mild mixing for a long time was the preferred mode of flocculation, but work by Rulyov (2004) demonstrate that an intense mixing for a short time gives a better performance.
In this work copper flotation tailings from a major Chilean mine were flocculated with an ultra-floculator yielding the following result:

1. Flocculation at the optimal flocculant dosage and optimal time permitted an increase settling rate by 50% when changing from a shear rate of $\gamma = 100 \text{ s}^{-1}$ to the optimum value of around $\gamma \approx 400 \text{ s}^{-1}$ depending on the suspension concentration.

2. The best flocculation was obtained at the suspension volume concentration in the range of 3–4 [% by volume] (75–115 [g/l]).

3. An increase of the suspension concentration above the optimum value, for example to 15 [% by volume] (400 [g/l]), increased the necessary flocculant dosage from 10 to about 20 [g/ton].

4. The suspension concentration and necessary mixing intensity are interrelated. For example, an increase in the suspension concentration from 1.8 to 10 [% by volume] (50 to 280 [g/l]) requires a decrease in the shear rates from 600 to 280 [s$^{-1}$] to maintain equal flocculation efficiency, but with further increase in the suspension concentration from 10 to 15 [% by volume] (280 to 400 [g/l]) the optimal value of the shear rate increases from 280 to 600 [s$^{-1}$].

5. The “UltraFlocTester: UFT-TFS-029” used in this work allowed to define explicitly the optimum flocculation conditions for maximizing the flux density function for a given thickening operation.

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References


