

## Rheology of strongly sedimenting magnetite suspensions

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### ABSTRACT

The flow properties of magnetite suspensions have been investigated with a modified Couette rheometer. The suspensions exhibited instability to sedimentation due to high density and a large particle size. In order to homogenise the suspension for rheological measurements, an additional upward force was applied by pumping the stirred suspensions through the measuring cell.

### INTRODUCTION

Processing of dense medium mineral suspensions involves various sub-processes such as grinding, flotation, filtration and pumping<sup>1</sup>. The wide range of particle concentrations that are used, lead to variable flow properties and makes it challenging to control and dimension the processing apparatus. Typical particle concentrations for e.g., magnetite-processing can be up to 15 % by volume<sup>2</sup>.

Measuring rheological properties of sedimenting suspensions are of great interest for many process applications where a solid is handled as a suspension, i.e. a particulate fluid. Many pigment suspensions, e.g. paper coatings colours, possess short-term stability from several hours up to several days. Minerals from the mining and metal recovery industries, on the other hand, may have such a high density and large particle size that the sedimentation rate is up to several centimetres per second. Measuring

the flow properties of such particle suspensions by conventional rotational shearing techniques is hence more or less impossible.

For colloidal particles ( $< 1 \mu\text{m}$ ) it is possible to stabilize the suspension, i.e. diminish the sedimentation tendencies by addition of polymers (sterically), by increasing the surface charge (electrostatically) or in combination. These interactions of colloidal particles can be estimated with the so called DLVO-theory (from Derjaguin-Landau and Verwey-Overbeek)<sup>3</sup>. The magnitude of the stabilizing repulsive interaction diminishes with increasing particle size and the sedimentation velocity increases with increasing size and density<sup>3</sup>. Due to its high density, the viscosity of magnetite suspensions was mainly governed by surface friction and inter-particle collisions<sup>2</sup>.

Several attempts have been made to measure the rheology of settling suspensions. Sarmiento et al.<sup>4</sup> used in their study a capillary rheometer with different tube lengths and tube diameters in combination with a parallel plate geometry. Their particle size was relatively small ( $d_{50} = 2.5 \mu\text{m}$ ) and of average density ( $3.0 \text{ kg dm}^{-3}$ ), which made it possible to measure the rheological properties with this setup.

Kawatra et al.<sup>5-6</sup> combined a vibrating sphere viscometer and a rotational viscometer. They used silica slurries ( $d_{50} = 30 \mu\text{m}$ , density not mentioned) as model sample. The viscometer had a high shear rate due to the oscillation at 750 Hz. It also had the suspension continuously circulated from a tank to the measuring cell in order to maintain homogeneity of the dispersion. By plotting the apparent viscosity from the two independent methods against each other, they were able to determine the viscosity and also the flow type, i.e. Newtonian or non-Newtonian.

He and Laskowski<sup>2</sup> as well as Klein et al.<sup>7</sup> used a double gap system to investigate the flow properties of magnetite suspensions (density  $4.8 \text{ kg dm}^{-3}$ , size:  $24 \mu\text{m}$ ). The purpose was to place the inner cylinder in the hindered settling zone, below the transition zone. This setup would ensure that the concentration around the bob (the inner cylinder) would remain known and constant and hence not affect the measuring result. A limitation of the approach was that the settling rate of the particles needed to be known and be relatively low. The Casson model was found to best describe the flow properties of the magnetite suspensions<sup>2</sup>. Furthermore, it was concluded that the Casson yield stress was dependent on the size distribution and the solids content of the suspensions.

By taking advantage of the helical flow that arises when the suspension is pumped upwards through a measuring cell, most of the settling problems can be minimized. The theory for the approach and a practical implementation has been developed by Akroyd and Nguyen<sup>8-9</sup>. The theory takes into account both the tangential (around the center) and axial (upward) flow components. With their instrument they were also able to determine the yield stress. They tested their instrument on e.g. fly ash (size  $50 \mu\text{m}$ , settling rate  $0.2 \text{ mm h}^{-1}$ ) and

diamond (size  $10 \mu\text{m}$ , settling rate  $13 \text{ mm h}^{-1}$ ) slurries.

The aim of this work was to clarify the influence of axial flow velocity and solids content on the measured shear stress of magnetite suspensions.

## EXPERIMENTAL

The magnetite ( $\text{Fe}_3\text{O}_4$ ) samples were fractionated by sieving. Five size fractions have been investigated, namely “ $< 30 \mu\text{m}$ ”, “ $30\text{-}146 \mu\text{m}$ ”, “ $43\text{-}63 \mu\text{m}$ ”, “ $63\text{-}146 \mu\text{m}$ ”, and “ $> 146\mu\text{m}$ ”. The density of the magnetite particles was  $5.2 \text{ g cm}^{-3}$ . The range between the lowest and the highest settling rate in water ( $23 \text{ }^\circ\text{C}$ ) is shown in Table 1.

Table 1. The range of the settling rate for the different size fractions of magnetite.

Size fraction	Settling rate $\text{mm s}^{-1}$
“ $< 30 \mu\text{m}$ ”	8.2 – 32.1
“ $30\text{-}146 \mu\text{m}$ ”	10.4 – 29.8
“ $43\text{-}63 \mu\text{m}$ ”	8.7 – 31.0
“ $63\text{-}146 \mu\text{m}$ ”	17.4 – 40.5
“ $> 146\mu\text{m}$ ”	29.3 – 77.2

The shear stress was measured with a modified Haake RV-2, with a concentric Couette system. The cup (outer cylinder) and bob (inner cylinder) diameters were  $42 \text{ mm}$  and  $38.6 \text{ mm}$ , respectively. Data of the torque was gathered as voltage on a PC through a data card (Pico Technology Ltd.). The setup, which was modified according to Akroyd and Nguyen<sup>9</sup>, is shown in Fig. 1.

The suspension was heavily stirred in a separate vessel and pumped with a peristaltic pump from the vessel upwards through the measuring cell. The drain was thereafter led back to the vessel.

Only relatively high shear rates could be used in the measurements, since at times, low shear rates lead to jamming of particles

in the measuring cell. The shear rates used were  $690 \text{ s}^{-1}$  and  $976 \text{ s}^{-1}$ .

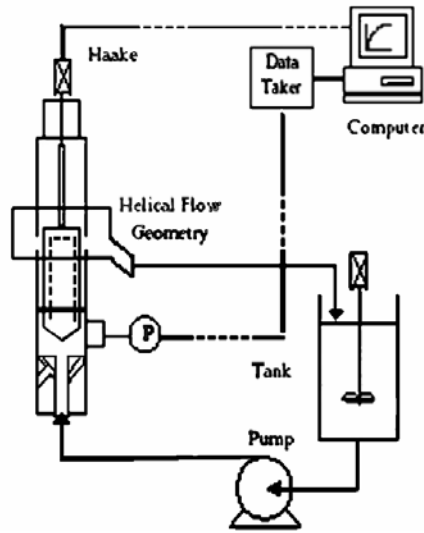


Figure 1. Setup of the modified Haake rheometer<sup>9</sup>.

### Calibration

The shear rate ( $\dot{\gamma}$ ) was calculated from the rotational speed and the dimensions of the Couette measuring cell by using the following equation:<sup>10</sup>

$$\dot{\gamma} = (2\Omega) \left( \frac{\alpha^2}{\alpha^2 - 1} \right) \quad (1)$$

where  $\Omega$  is the angular speed, defined as

$$\Omega = (2\pi) \text{RPS} \quad (2)$$

and  $\alpha$  is the ratio between the radii of the cup and the bob:

$$\alpha = R_{\text{cup}} / R_{\text{bob}} \quad (3)$$

The shear stress,  $\sigma$ , was calculated from:

$$\sigma = \eta_{\text{known}} \times \dot{\gamma}_{\text{set}} \quad (4)$$

The rheometer was calibrated with three Newtonian liquids with known viscosities. The voltage was then related to the calculated shear stress,  $\sigma$ , at two preset shear rates as is illustrated in Fig. 2.

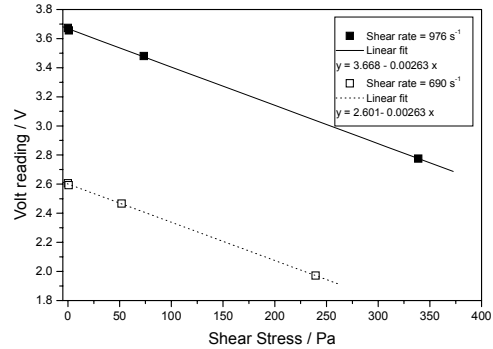


Figure 2. Calibration curve.

The linear dependencies could be expressed for the shear rates  $690 \text{ s}^{-1}$  and  $976 \text{ s}^{-1}$ , respectively.

$$(V) = 2.601 - 0.00263 (\sigma) \quad (5)$$

$$(V) = 3.668 - 0.00263 (\sigma) \quad (6)$$

### RESULTS

The measured shear stress is shown in Fig. 3 as a function of particle concentration for the “< 30  $\mu\text{m}$ ” fraction. The shear rates are  $976 \text{ s}^{-1}$  and  $690 \text{ s}^{-1}$  and the axial flow rate is approximately  $11 \text{ min}^{-1}$ . Each measuring point is a mean value of 100 data points, which were recorded at  $1 \text{ Hz}$ . The measured shear stress increased with increasing volume fraction, due to higher number of inter-particle collisions. The magnitude of the shear stress was in the same range as that of ref.<sup>2</sup>.

The highest shear stress values corresponded to a viscosity of  $25 \text{ mPas}$ , which approached the lower sensitivity limit of the measuring device and as a consequence caused scatter in the data.

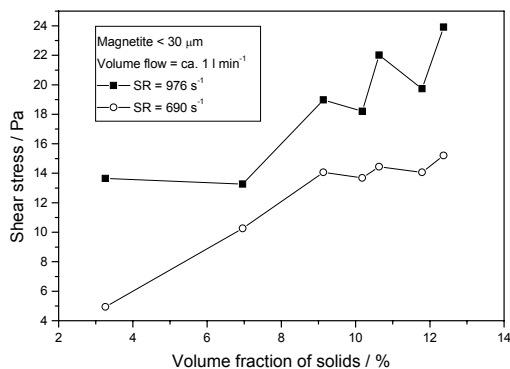


Figure 3. Shear stress as a function of solids content for the “< 30 μm” fraction.

The measured shear stress at  $976 \text{ s}^{-1}$  as a function of solids content for all five size fractions is shown in Fig. 4. The measured shear stress increased with increasing solids content up to a certain solids content. The scattering of the results was thereafter relatively large, and no systematic trend between the size fractions could clearly be found. A contributing factor to this could be local jamming of particles in the cell due to the high solids content.

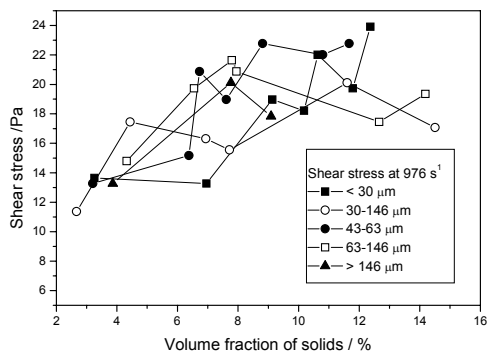


Figure 4. Shear stress as a function of volume fraction of solids for all five size fractions.

The measured shear stress as a function of axial (upward) volume flow of suspension is shown in Fig. 5 for water and two solids contents of the “< 30 μm” fraction. At higher axial volume flow, the shear stress decreased. This was caused by

the decrease in the size of the shear zone in the horizontal plane as the flow rate was increased. The dependency was found to be approximately linear.

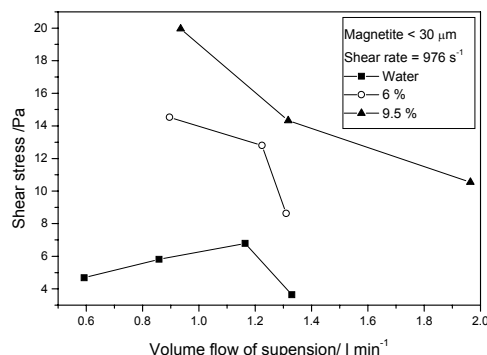


Figure 5. The measured shear stress as a function of volume flow of suspension.

## CONCLUSIONS

The flow properties of sedimenting magnetite suspensions has been investigated with a modified Couette rheometer in which axial flow was used to enable measurements at high volume concentrations and to minimize the effects of sedimentation. It was shown that the measured shear stress decreased with increasing rate of the through flow of the suspension due to a diminished shear zone. The shear stress increased as a function of solids content, but some scatter in the results was found especially at higher solids contents. This was probably caused by local jamming of particles in the measuring cell, especially at solids volume fractions higher than approximately 10%. The technique could be useful in investigations of the influence of chemical additives on the flow properties of sedimenting suspensions.

## ACKNOWLEDGMENTS

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## REFERENCES

1. Shi, F.N., Napier-Munn, T.J. (1996), "Measuring the rheology of slurries using an on-line viscometer", *Int. J. Miner. Process.* **47**, 153-176.
2. He, Y.B. and Laskowski, J.S. (1999), "Rheological properties of magnetite suspensions", *Miner. Process. Extract. Metall. Rev.*, **20**, 167-182.
3. Shaw, D.J. (1992), "Introduction to Colloid and Surface Chemistry", 4<sup>th</sup> ed., Butterworth-Heinemann, London, pp. 21-32, pp. 210-234.
4. Sarmiento, G., Crabbe, P.G., Boger, D.V., Uhlherr, P.H.T. (1979), "Measurement of the rheological characteristics of slowly settling flocculated suspensions", *Ind. Eng. Chem. Process Des. Dev.*, **18**, 746-51.
5. Kawatra, S.K., Bakshi, A.K. and Miller, T.E. Jr. (1996), "Rheological characterization of mineral suspensions using a vibrating sphere and a rotational viscometer", *Int. J. Miner. Process.*, **44-45**, 55-165.
6. Kawatra, S.K. and Bakshi, A.K. (1996), "Online measurement of viscosity and determination of flow types for mineral suspensions", *Int. J. Miner. Process.* **47**, 275-283.
7. Klein, B., Laskowski, J.S. and Partridge, S.J. (1995), "A new viscometer for rheological measurements on settling suspensions", *J. Rheol.*, **39**, 827-840.
8. Akroyd, T.J. and Nguyen, Q.D. (2003), "Continuous rheometry for industrial slurries", *Exp. Therm. Fluid Sci.* **27**, 507-514.
9. Akroyd, T.J., and Nguyen, Q.D. (2003), "Continuous on-line rheological measurements for rapid settling slurries", *Minerals Engineering* **16**, 731-738.
10. Steffe, J.F. (1996), "Rheological methods in food process engineering", 2<sup>nd</sup> ed. Freeman press, East Lansing, pp. 115-139.