Understanding Feed Rheology in Nickel Laterite Processing

F. Sofra
Rheological Consulting Services Pty Ltd, Melbourne, Australia

Abstract

The main application of paste technology is tailings disposal. However, paste technology is also being implemented to modify feed characteristics in nickel processing. The mineralogical composition and physical constitution of nickel laterite ores varies between deposits and also within a given deposit and during 'life of mine'. Understanding how these variations impact on the 'processability' of ores is an essential step in identifying and mitigating project risk, and the ongoing sustainable operation of the processing plant. Refining nickel laterites using pressure acid leach technologies requires that the solids concentration of the feed to the autoclave is maximized whilst favourable rheological characteristics are maintained to ensure adequate heat transfer and efficient reaction rates. Maximizing the concentration of the feed also minimises the capital cost of autoclaves and associated unit operations. For nickel laterites, the solids concentration required to achieve the design rheology may vary dramatically depending on the ore mineralogy and the processing conditions. This paper outlines testwork that has been conducted to characterise the rheological properties of nickel laterite feeds in order to define appropriate operating conditions and highlight unique difficulties which may be encountered when processing nickel laterite ores.

1. Introduction

A pressure acid leach (PAL) process is used to extract nickel from ore containing predominantly two mineral types, limonite and saprolite. Typically, ore is blended, screened, crushed, slurried with hot process water, transported by pipeline and thickened prior to being leached in an autoclave where processing takes place at low pH, elevated temperature and pressure.

In many cases, plants have been designed using rheological data obtained for limited or 'representative' samples. Due to a number of rheology related failures and process difficulties encountered by these projects, this is now understood to be a practice that results in excessive risk. Nickel laterite ores can exhibit significant variations in both the magnitude of rheological parameters and the actual rheological phenomena observed. Therefore, failure to define and quantify the range of behaviour evident throughout the ore body may result in the design of feed thickening and handling systems which are unable to meet the autoclave concentration, throughput and/or flow characteristic requirements. To the credit of those involved in projects currently underway and in various stages of investigation, design, engineering and implementation, rheological studies are increasingly detailed and aimed at gaining a comprehensive understanding of the variations in the rheological properties throughout the ore body and the impact of these variations on the successful operation of the plant.

The solids concentration of the autoclave feed should be maximised in order to minimise the size and cost of the autoclave for a given throughput. When designing the key plant areas upstream of the autoclave, the objective is to balance the desire to maximise the feed concentration without compromising (a) the efficiency of the autoclave and (b) the pumping and thickening efficiency of other plant areas. As the autoclave feed concentration is increased, the heat transfer efficiency may be compromised due to the formation of slugs along with an increase in the pumping energy requirements. The aim for designers and operators is to maximise the feed concentration whilst maintaining favourable rheological properties.

Although the solids concentration of the autoclave feed is important from a cost and throughput perspective, the yield stress is the crucial parameter for successful design and operation of the feed thickening and handling systems and the autoclave. For nickel laterites, the solids concentration required to achieve the design yield stress may vary dramatically depending on the ore mineralogy and the processing conditions.

Some nickel laterite slurries possess unique rheological characteristics rarely seen in the minerals industry relating to shear history effects, whilst others possess characteristics commonly observed in mineral suspensions. As both unique and common behaviour may be evident in one ore body, it is essential that the variations in the deposit are identified and appropriate strategies are employed to ensure that the plant will accommodate the feed presented.

Comprehensive rheological characterisation provides input to allow the appropriate design and optimisation of the key plant areas through manipulation of the ore blend ratio, solids concentration, shear history effects and temperature. This paper presents selected results from several rheological studies undertaken for two laterite operations (deposits A and B) and highlights the care which must be taken to ensure that data obtained is relevant to the autoclave feed thickening and handling systems.

2. Rheological Studies Conducted

Since mid 2001, Rheological Consulting Services (RCS) have tested and analysed over 50 nickel laterite ore samples and have been engaged to review rheological data generated by nine other testing facilities. The majority of studies were undertaken on samples from two deposits (deposits A and B) in different parts of the world. Both deposits contained a limonite and saprolite zone, with a transition zone between the two. Samples for rheological testing were produced during pilot plant campaigns and either tested on site or in our Melbourne laboratory.

3. Yield Stress Measurement

3.1. Vane Yield Stress

Yield stress measurements were conducted using the vane-shear method (Nguyen and Boger, 1983). Many workers worldwide have adopted the vane-shear method and confirmed its applicability for numerous types of yield stress materials.

The vane method requires the rotation of a vane consisting of four perpendicular blades immersed in the slurry under investigation. The vane geometry aims to minimise slip occurring between the sample and the instrument fixture. The torque versus time response of the system is recorded at a fixed rotation rate. The maximum torque observed is used to calculate the yield stress.

For samples tested at elevated temperature (up to 90 degrees C), all efforts were made to minimise evaporation, and the solids concentration was measured both before and after testing to identify if evaporation had taken place.

3.1.1. Results

All laterite samples tested were shear history dependent. One way to observe this shear history dependence is to monitor the change in the yield stress with shearing time. In general, this will result in an equilibrium or fully sheared state, characterised by a constant yield stress with increasing time of shear, as shown for a typical sample in Figure 1.

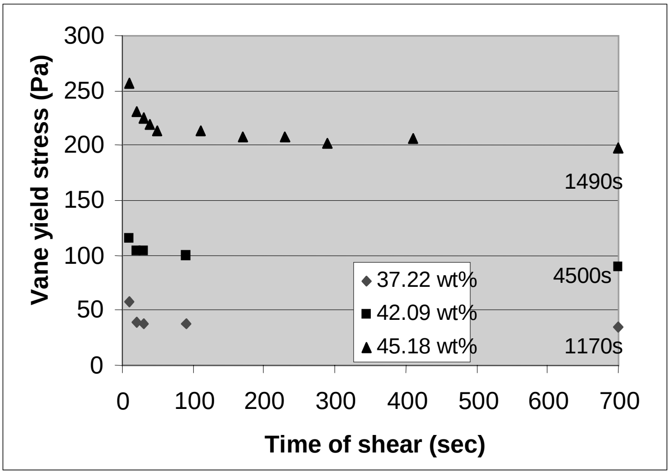


Figure 1: Vane shear yield stress vs. time of shear showing the equilibrium state (time beside final data point shows the total duration of yield stress monitoring i.e. no further change in yield stress from 700s to the time shown).

For one nickel laterite deposit studied (deposit A), samples were not tested as they were produced by the pilot plant, but were collected and transported to the laboratory, for testing some time later. Because of the transport and homogenisation required, the shear history was impossible to quantify. Therefore, the 22 samples to be tested were sheared to the equilibrium state prior to measurement of the yield stress as a function of solids concentration at ambient temperature. Shearing the samples to equilibrium negated the shear history effects on the yield stress. All samples tested had been subjected to the same processing temperature and water quality.

In all cases, the equilibrium yield stress showed an exponential rise with solids concentration. The band shown in Figure 2 highlights the variability in the vane yield stress due to variations of the mineralogy and particle size of the deposit mined.

Although the specific mineralogical compositions and particle size distributions are not presented in Figure 2, what is important to note is the variability in the solids concentration required to produce a given yield stress, even having ensured that all samples were in the same shear history state (i.e. sheared to equilibrium). In this case, the target yield stress for the autoclave feed was 100 Pa, which required thickening to somewhere between 37 wt% and 43.5 wt% depending on the mineralogy and particle size distribution of the ore, a potential difference of 6.5% by weight. It should be noted that these 22 samples were not a total representation of the entire ore body, but of just one set of variables under investigation. For this entire region of the ore body, the variation in solids concentration for a yield stress of 100 Pa was in the order of 30 wt% (McCrabb et al., 2004).

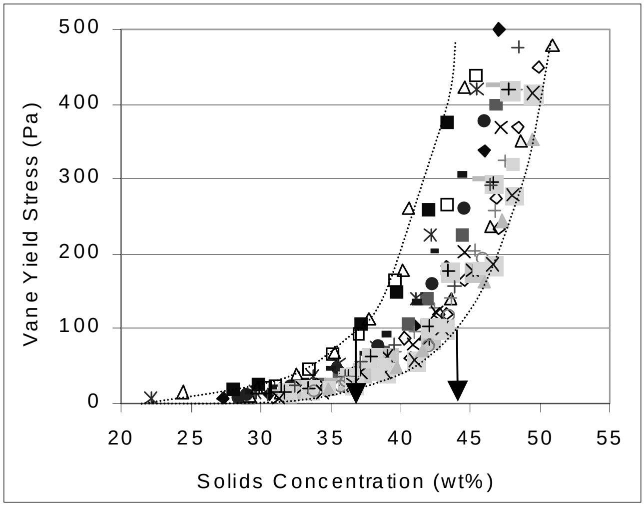


Figure 2: Yield stress versus solids concentration of 22 samples from deposit A indicating the variation in the yield stress profiles due to mineralogy and particle size distribution.

Figure 3 shows similar results for a laterite deposit in another part of the world (deposit B). In this case, samples were tested on site, at the time of production from a pilot scale thickener. The major compositional variable in the samples was the relative proportions of limonite, saprolite and transition zone (the region between limonite and saprolite) ores. The effect of the shear history imparted (from no preshear to shearing to the equilibrium state) was also investigated. As shown in Figure 3, the solids concentration required to achieve a yield stress of 100 Pa ranges from 34.8 wt% to 38.7 wt%, a range of almost 4 wt% depending on the blend ratio and shear history.

Figure 4 shows the yield stress as a function of solids concentration for the sheared and unsheared blends for Deposit B. In all cases the general effect of shearing was to lower the yield stress at a given solids concentration. At 37wt%, the yield stress for Blend 1 reduced by 30% upon shearing, while the yield stress of Blend 2 reduced by 54%. At 38 wt%, the yield stress for Blend 1 reduced by 29%, while the yield stress of Blend 2 reduced by 59%.

From Figure 4, there is a notable difference between blend 1, containing a high saprolite content and blend 2, containing a low saprolite content. Blend 2 generally has a lower yield stress at a given solids concentration. It should be noted that the unsheared blend 2 data may show a yield stress that is higher than expected, especially at high solids concentrations, due to the very high flocculant dosages used to achieve the required solids concentration from the thickener. The difference between the two blends is most evident in the sheared case for each blend as the effect of flocculation was negligible after shearing.

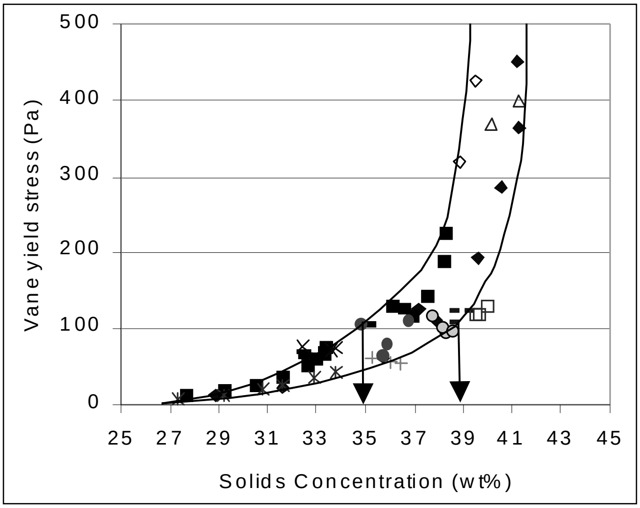


Figure 3: Yield stress versus solids concentration. Samples from Deposit B, indicating the variation in the yield stress profiles due to blend ratio and shear history.

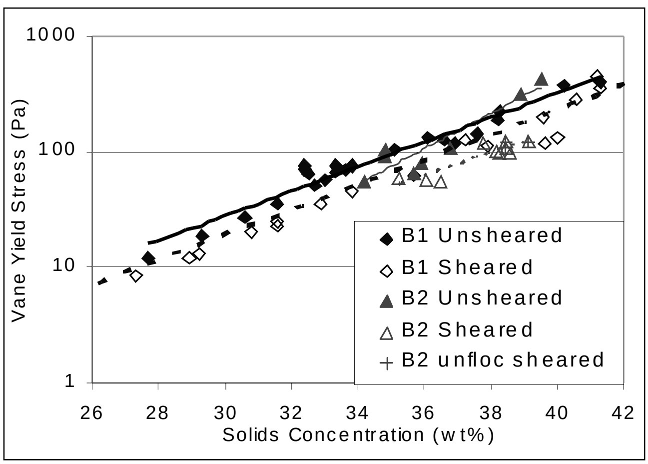


Figure 4: Yield stress versus solids concentration for sheared and unsheared blends 1 (high saprolite) and 2 (low saprolite) from Deposit B.

The large degree of scatter in the unsheared data can be attributed to the frequently changing focculation regime and thickener operating conditions. The flocculant dose was frequently changed in an effort to obtain the required solids concentration from the thickener. Even though the blend may not have changed, strictly speaking the same material was not always tested, especially in the unsheared state where the effect of flocculant would be most relevant. The sheared yield stress of blend 1 is approximately 30% higher than the sheared yield stress of blend 2.

As the thickener underflow is generally pumped using high shear centrifugal pumps, the sheared yield stress value is the relevant design parameter for equipment and processes downstream of the pump. Many modern thickener arrangements now also incorporate recirculation of sheared underflow into the base of the thickener to aid removal of the (now partially sheared) underflow. Although time did not permit rigorous collection of aging data, cursory measurements showed no significant increase in the yield stress following shearing and resting for up to 48hrs.

Although Figure 4 is presented in semi-log format to facilitate distinction between the samples, the semi-log format can be misleading as it masks the severe exponential curvature in the yield stress profiles. The curvature is important as it is not advisable to operate in the region near the ‘elbow’ of the curve, where minimal changes in the concentration relate to significant changes in the yield stress. When viewed on linear axes, as in Figure 3, it is evident that beyond approximately 38 wt%, there is a dramatic increase in the yield stress with only minor increases in the solids concentration. Given the difficulty on operating a thickener within a narrow underflow concentration range, for this material it was advised that the target operating concentration be below approximately 38 wt%.

The yield stress data presented in Figures 2 to 4 were relatively straightforward to obtain, as all of the samples tested showed a progressive reduction in the yield stress with time of shear, to reach a final equilibrium state, irrespective of the shear rate used. Typically, materials are tested in as close to a fully sheared state as possible in an attempt to negate the effect of thixotropy on the yield stress and allow comparison or ranking of materials where the shear history is unknown.

However, for additional laterite samples obtained from Deposit A (‘H class’ samples), this was not possible. Figures 5 and 6 show how the shear rate used to preshear the ‘H’ samples had a dramatic effect on the yield stress. In this case, a low preshear rate resulted in a decrease in the yield stress while a high shear rate caused the yield stress to increase. Because of this combined rheopectic/thixotropic nature of the material under varying shear regimes, obtaining a singular meaningful ‘equilibrium state’ was unachievable. Ordinarily, the fully sheared state is characterised by a constant yield stress reading with increasing time of shear as shown in Figure 1 for a typical thixotropic sample. The Deposit A, H sample, however, did not display such an easily tracked progression of shear breakdown, as Figures 5 and 6 illustrate. Similar behaviour is exhibited by both the –38 mðm and –150 mðm material indicating that the phenomenon is not a particle size specific event but rather a characteristic of the material on the whole. The apparent reversibility and range of the structural states suggests that attempts to define an equilibrium state are purely subjective and shear history dependent.

In cases where this type of behaviour is evident, it is important to ensure that the shear conditions that will be experienced in the plant are well understood and reproduced as precisely as possible for testing purposes.

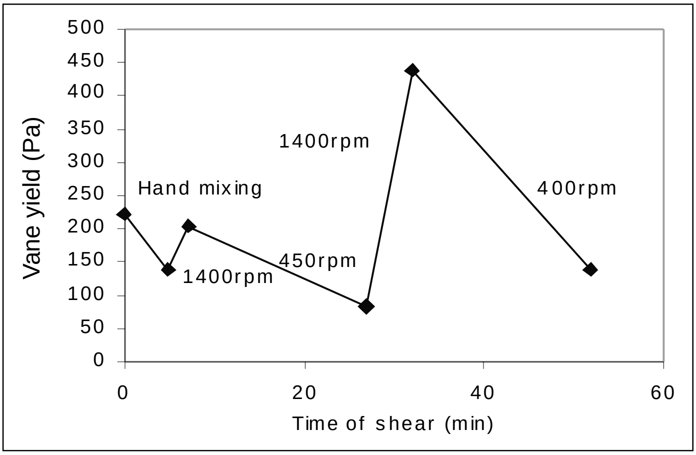


Figure 5: Yield stress progression with varying intensity of applied shear. Sample material: Deposit A, sample H –150 mðm, 60 %w/w solids. Progressive rotational rates (rpm) are indicated.

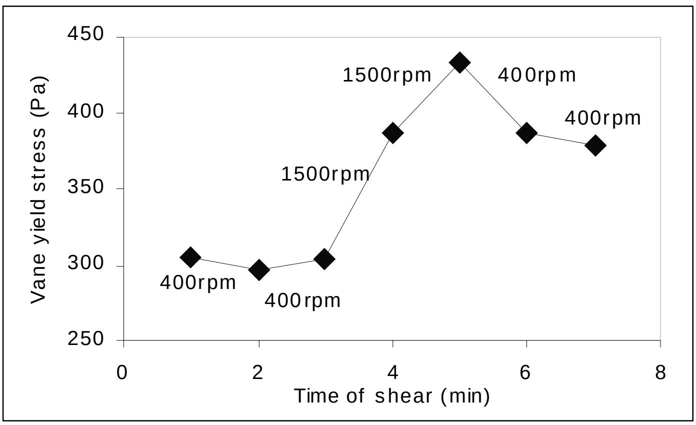


Figure 6: Yield stress progression with varying intensity of applied shear. Sample material: Deposit A, sample H –38 mðm, 60 %w/w solids. Progressive rotational rates (rpm) are indicated.

As Figure 7 illustrates, due to the distinct shear history dependence of the H samples, characterised by the varying yield stress with the rate of preshearing for each of the size distributions investigated, fitting any sort of predictive functional form to the data was deemed inappropriate. Analysed in conjunction with the data presented in Figures 5 and 6, relating the effects of various regimes of applied shear to the resultant measured shear yield stress, it is clear that the material being treated with may be passed through any number of structural states depending on the nature of its shear history.

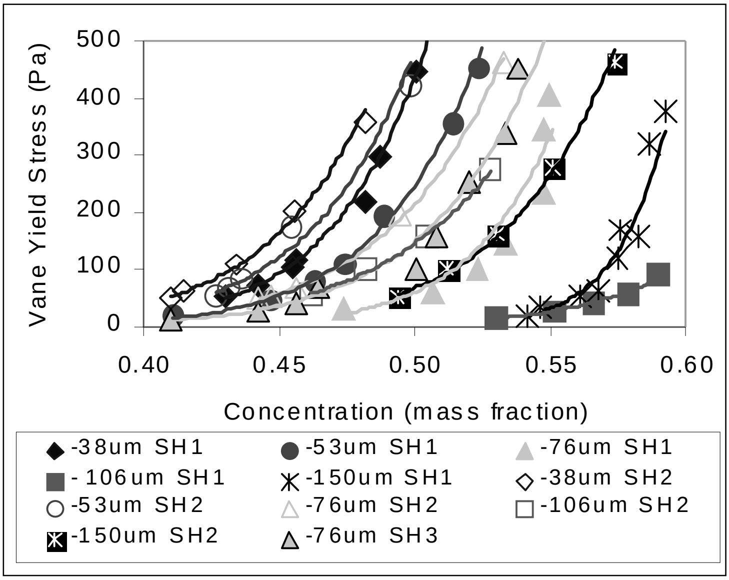


Figure 7: Summary of yield stress profiles for Deposit A, class H samples. SH1 to SH3 indicates sample runs with unique shear histories.

High shear rates were seen to promote structure within material with yield stresses much higher than that of lightly sheared samples i.e. the material is rheopectic, thickening upon shearing within certain shear regimes. However, it was further demonstrated that this structural gain, or thickening, at high shear rates was susceptible to thixotropic breakdown at low applied shear rates. The shear yield stress for these materials as a function of solids concentration is therefore not a static quantity. For any given solids concentration a yield stress value falling anywhere within a 300-400 Pa range might be obtained. The lack of an equilibrium rheological state independent of shear history renders the determination of a solids concentration, corresponding to a design yield stress, case specific and values obtained cannot be presented as a finite material property.

Due to the essentially reversible nature of the rheopectic/thixotropic behaviour observed for some laterites, the phenomenon is believed to be due to particle agglomeration at high shear, whereby discrete particles are brought together to form larger agglomerated units through the energy supplied in shear. In this agglomerated state the forces acting on and between the particles are altered and an increase in the rheological characteristics of the system, specifically, increased shear yield stress and relative viscosity, is observed.

Shear agglomeration is a dynamic process and at lower shear rates the energy supplied to the system is insufficient to produce further agglomerate build-up, with the rate of agglomerate breakdown exceeding the rate of formation. Within these lower shear regimes, thixotropic structural breakdown is then observed as the suspension gradually approaches a dispersed system of more or less discrete particles. The long-range interactions of the latter system produce a weaker network structure than the agglomerated mass and decreased rheological characteristics are observed. Key factors affecting such behaviour but not quantified in this paper are ionic strength, pH, particle size, particle shape, and solids concentration.

Despite the dynamic nature of the material properties for the laterites tested, a number of general conclusions may be drawn from the yield stress characterisation tests.

- In all cases, the yield stress showed an exponential rise with solids concentration and overall tended to be lower at any given solids concentration as the average particle size increased.
- Large deviations in the relative position and slope of individual yield stress profiles are observed, and yield stress values are highly dependent upon shear history.
- High shear rates were seen to result in an increase in shear yield stress for a given solids concentration in each of the materials, while lower shear rates brought about structural breakdown and a decreased yield stress. The H material was seen to exhibit both rheopexy and thixotropy depending upon the applied shear regime.
- For a given solids concentration, yield stresses were seen to differ according to shear history in the range of 50 to 350 Pa. For each of the samples, the effect of shear on the yield stress was observed to increase with increasing solids concentration. Curves for different top-sized samples intersect at various points depending on their shear history.

4. Shear Stress versus Shear Rate Measurement

4.1.Viscometry Methods

Characterisation of the shear stress versus shear rate behaviour was carried out using a Haake VT550 viscometer with either ridged couette (cup and bob) or ‘vane in an infinite’ medium geometry. Initially the vane was given preference on the basis that slip was observed at low shear rates using smooth cup and bob. However, no slip was evident when a ridged couette was used, so both geometries were eventually employed.

For shear stress versus shear rate testing the bob or vane was lowered into the cup or container and is rotated at a prescribed angular velocity. The torque experienced by the bob is measured and recorded, allowing the shear rate and shear stress to be calculated.

4.2. Results

For many of the Deposit A samples tested, the equilibrium shear stress versus shear rate data showed yield stress – shear thinning behaviour. The extent of shear thinning was a function of solids concentration and mineralogy. Typical results are shown in Figure 8. The general characteristics evident in Figure 8 are often observed in many concentrated mineral suspensions.

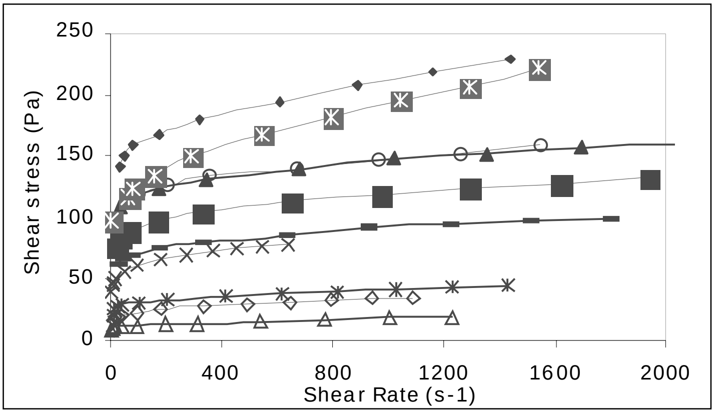


Figure 8: Selected shear stress versus shear rate curves for Deposit A samples.

As with the yield stress characteristics, the viscous behaviour of particular samples from both Deposit A and Deposit B displayed severe shear history sensitivity. One method of obtaining viscosity data for time dependent materials is to measure the shear stress as a function of time for a range of shear rates. Cross plots can then be constructed showing the relationship between the shear stress and the shear rate for various shear histories (ie times of shear). Figure 9 shows these data for selected samples from Deposit A.

For classic thixotropic materials, the stress would reduce from a maximum at low shear times to a minimum, equilibrium value at extended shearing times, while rheopectic materials show the opposite trend. For each successive shear rate tested both the initial and final shear stresses are generally higher than for the previous shear rate, although often by differing amounts as the extent of thixotropy/rheopexy can vary with shear rate. However, as shown in Figure 9, this trend is not observed for some lateritic ores.

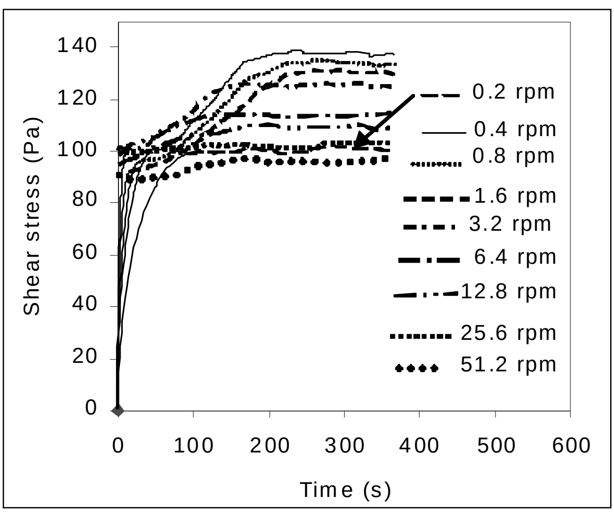


Figure 9: Shear stress as a function of time for various shear rates for Deposit A, samples containing high aspect ratio goethite needles.

The phenomenon of having a yield stress, taken at a rotational rate of 0.2 rpm, so much lower than the shear stress value at a rotational rate of 0.4 rpm and not following the overall trend of decreasing with increasing shear rate is not entirely understood. However, for the testwork shown in Figure 9, and for testwork conducted at additional sites, this phenomenon was completely reproducible. Furthermore, between 21 and 50 rpm, the stress versus time curve actually decreases beneath the yield value.

Figure 10 shows the stress-time data as shear stress versus rotational rate at equilibrium values.

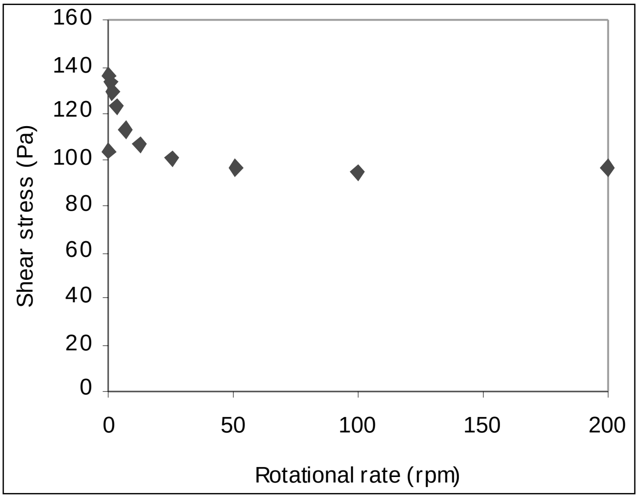
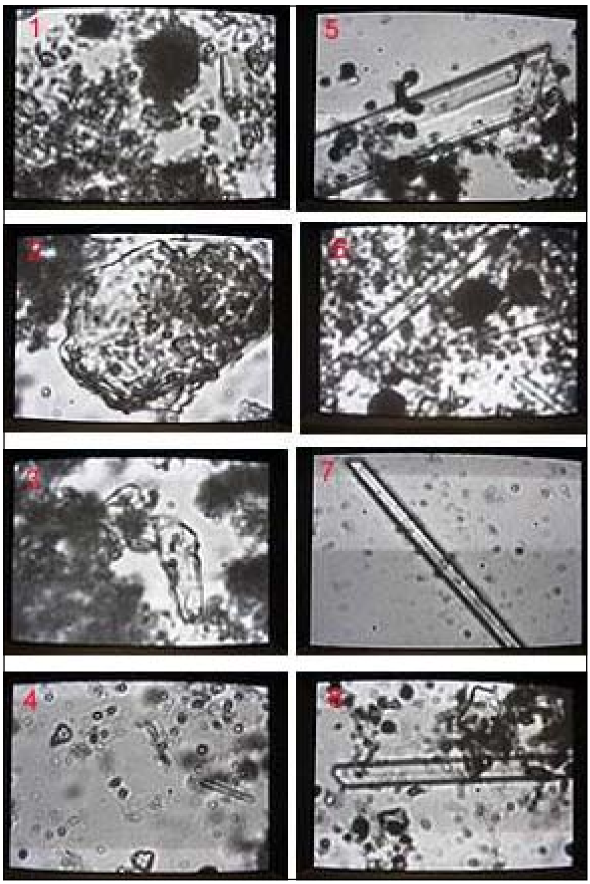


Figure 10: Equilibrium Shear stress vs rotational rate for Deposit A samples containing high aspect ratio goethite needles.

Observation of this complex rheological behaviour and taking into account the ready return of the material to its initial, pre-test state, by hand-stirring is suggestive that the rheopexy is a localised event. The rheopexy promoted in the immediate vicinity of the shear surface is possibly due to the flow-induced orientation or hydrodynamic interaction of microstructural elements (particles or flocs) within the fluid, rather than a strong or irreversible chemical bonding effect. Microscopic comparison of samples shown in Figure 11 revealed that samples with complex shear history effects contained highly asymmetric, needle-like particles which were absent in samples where the shear history effects were limited to consistent thixotropic breakdown. These needle-like particles were also observed by Blakey and James (2003) and were found by X-ray diffraction to be goethite.

Rotational measurements conducted on site at Deposit B showed similar complex shear history effects. In this case, ramped measurements were taken, where the test sequence involved ramping the shear rate from 0 to $600s^{-1}$ in 2 minutes then from $600s^{-1}$ to 0 in the subsequent 2 minutes. This cycle was twice repeated, with the total test time being 12 minutes. However, as the shear stress versus shear rate results in this work and in other works reviewed shows a strong dependence on shear history, it is concluded that the results using this approach are dependent on the programmed rate of shear rate increase and decrease. That is, the stress response at each shear rate will be dependent on the shear rate and time of shear imposed before that measurement. Such results are not useful for design and can only be used for comparative purposes where the shear history is known. In the case of laterites, for shear stress versus shear rate data to be useful for design purposes, the shear history needs to mimic the actual process being modelled. However, provided samples are measured in an identical manner, subject to the same shear history, using the same instrumentation and at the same concentrations, the data may be useful for comparative purposes, that is, to rank the rheological properties of various samples relative to one another.



Non-complex shear history *Complex shear history*

Figure 11: Microscopic images of nickel laterite samples. Samples in the left hand column did not display the complex shear history effects that were observed in samples in the right hand column, where high aspect ratio needles were evident.

As shown in Figure 12, which exemplifies the trends seen for all Deposit B samples tested, a severe initial shear stress overshoot was observed, followed by thixotropic breakdown, as noted for some Deposit A samples. The thixotropy is denoted by the presence of a lower shear stress at a given shear rate for each up/down sweep. The overshoot and subsequent thixotropic behaviour was observed for all samples tested, both unsheared and sheared. It is important to note that the maximum stress reached in the overshoot was generally 50 to 100% greater than the measured vane yield stress.

The overshoot is consistent with the presence of high aspect ratio i.e. needle-like particles and alignment of these particles within the flow field.

The actual mechanics of the microstructural ordering or interactions brought about by these needle-like particles in terms of their effects upon the bulk rheological response is not entirely understood but it is postulated that at low shear rates a certain level of pseudo-elasticity is induced by the random interlocking and interaction of these needle-like networks. This interlocking and interaction promotes long yield times and also leads to increased drag and energy dissipation upon flow as they break up, reorient, interlock and collide. At higher flow or shear rates, an alignment of the asymmetric components within the shear plane may be responsible for the extreme shear-thinning observed. The idea that overall interactions are weak, in a chemical or bonding sense, and more reliant upon hydrodynamic or mechanical considerations is supported by the observation that tested material is readily brought back to its initial pre-test state through a small amount of hand stirring, having the effect of re-randomising the system, removing the flow-induced orientation of the testing.

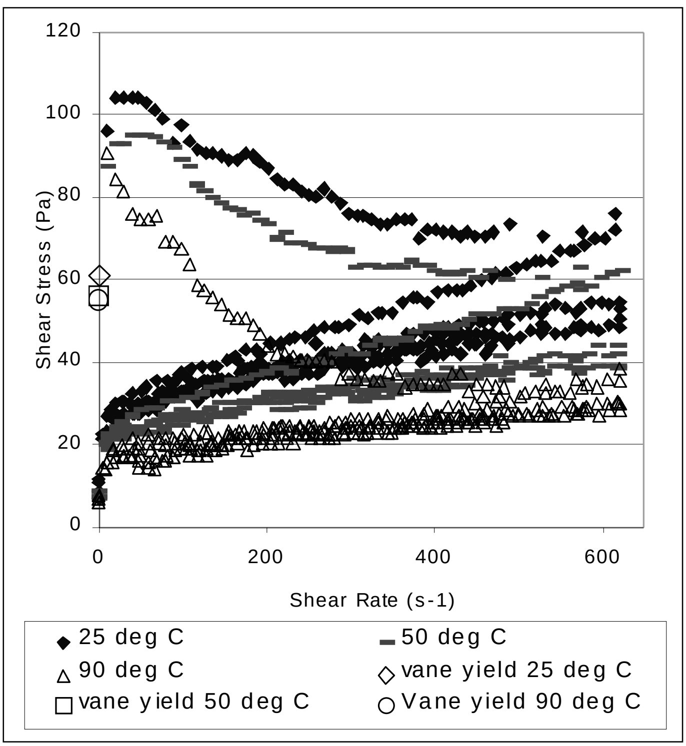


Figure 12: Example of full shear stress versus shear rate ramping curves for Deposit B.

5. Conclusions

Maximising the solids concentration of the autoclave feed is important from a cost and throughput perspective, while maintaining the required rheological characteristics is crucial for successful design and operation of the feed thickening and handling systems and the autoclave itself. For nickel laterites, the solids concentration required to achieve the required rheology may vary dramatically depending on the ore mineralogy and processing conditions. The rheology must be investigated and well understood as major variations in the magnitude of the yield stress and viscosity have been observed. In addition, it has been noted that the shear history related phenomena usually varies dramatically throughout the ore body.

The vane yield stress of freshly produced autoclave feed thickener underflow provides the single most useful rheological parameter for design purposes.

Shear stress versus shear rate results in this work, and in other works reviewed, showed a strong dependence on shear history. For continuously ramped flow curve measurements the results will therefore be dependent on the programmed rate of shear rate increase and decrease. These absolute results should not be used directly as design parameters. However, provided samples are measured in an identical manner, subject to the same shear history, the data is be useful for comparative purposes, to rank the rheological properties of various samples relative to one another.

Recommendations

For the design of laterite autoclave feed thickening and handling systems, it is recommended that the vane yield stress of the unsheared and sheared feed thickener underflow is measured at the process conditions. The thickened samples produced in a pilot campaign should encompass the range of feed materials likely to be encountered throughout the life of mine.

It is recommended that shear stress versus shear rate data be obtained using a capillary rheometer, with stringent control of the shear history to better understand the base rheological properties. The process conditions should be mimicked by preshearing the material at the appropriate shear rate/s for the same residence time/s as the operation being modelled. The vane yield stress should be measured for each sample concurrently, ensuring that the sample is subjected to the same shear history.

A need does exist for further understanding and informed interpretation of rheological data obtained for nickel laterite systems. To this end, our investigations are now focussed on obtaining thickener underflow yield stress (both unsheared and sheared) and rotational rheometry data at the time, temperature and shear history of thickener production and combining these data with capillary rheometry conducted in a controlled environment. The aim is to identify if the combined thixotropy/rheopexy is an instrumental artefact resulting from the short-range flow induced orientation and aggregation of particles or a true material property which will be relevant to the industrial scale autoclave feed thickening and handling system.

Acknowledgements

The author wishes to acknowledge the companies who allowed the use of their data and granted permission to publish this paper.

References

Blakey, B.D. and James, D.F. (2003) Characterising the rheology of laterite slurries. *International Journal of Mineral Processing.* 70 (2003), pp. 23-39.

McCrabb, L., Chin, J. and Miller, G. (2004) A study utilizing vane yield rheometry to predict optimum thickener performance across a range of Laterite nickel ores for the Ravensthorpe nickel project. In *Proceedings of the International Laterite Nickel Symposium 2004* Slurry Rheology, Solution Extraction and other.

Nguyen, Q.D. and Boger, D.V. (1983) Yield stress measurement for concentrated suspensions. *Journal of Rheology* (New York, NY, United States), 1983. **27**(4): pp. 321-49.