EFFECTS OF FLOCCULATION CONDITIONS ON THE DEWATERABILITY OF HEMATITE AND RED MUD SUSPENSIONS

Hulston, J., de Kretser, R. G. and Scales, P. J.
Particulate Fluids Processing Centre, Department of Chemical Engineering, The University of Melbourne, Victoria, Australia

Abstract

Flocculation is commonly used in the minerals industry, such as the Bayer process, as a means of hastening solid-liquid separation. A comprehensive characterisation technique has been developed within the PFPC, which allows flocculation and dewaterability behaviour of suspensions to be investigated. The technique involves flocculating suspensions in a baffle reactor and determining the initial settling rate, equilibrium bed height and supernatant turbidity. The flocculated suspensions are then analysed in terms of the gel point, compressive yield stress, $P_y(\phi)$, and hindered settling function, $R(\phi)$. The information gathered from these experiments can then be used in conjunction with a one-dimensional thickener model to predict the effectiveness of flocculants on thickening.

A variety of flocculation experiments were performed on hematite and red mud suspensions with the aim of making comparisons between the two systems and determining the suitability of hematite as a model system for red mud. The two systems were compared on the basis of their flocculation and dewaterability behaviour. In addition the thickener performance of both suspensions was modelled for the various flocculation conditions investigated.

1. Introduction

Gravity settling is commonly used in the minerals industry as a method of enacting solid-liquid separation. As this process relies on the settling rate of the solid phase, process throughput can be significantly reduced by the presence of a high percentage of fines, which are slow to settle. High molecular weight polymers (flocculants) are commonly added to bind individual particles or aggregates into structures known as flocs, which have a significantly higher settling rate. This process is known as bridging flocculation.

One application of bridging flocculation is the Bayer process, which is used to produce alumina from bauxite. In this process aluminium minerals present in the bauxite such as gibbsite, boehmite and diaspore are digested using concentrated sodium hydroxide to form aqueous sodium aluminate (pregnant liquor), leaving behind an insoluble residue known as ‘red mud’. The red mud is separated from the pregnant liquor, with the aid of flocculants, in a clarification vessel known as a thickener. The red mud fraction is then washed in a multi-stage counter-current washing circuit to remove residual sodium hydroxide. This washing stage is performed in a series of clarification vessels known as washers, to which flocculants are added to aid in solid-liquid separation.

The process can be optimised by increasing thickener/washer underflow solids concentrations, provided that suspensions are still pumpable. Optimisation of the thickening/washing process has the following advantages:

i) Improved throughput
ii) Improved efficiency of washer operation (i.e. higher sodium hydroxide recovery)
iii) Reduction in final residue volume.

Higher underflow solids concentrations are thought to be achievable by manipulating the flocculation conditions in thickeners and washers (e.g. flocculant type, dose, mixing conditions).

Using dewatering theory developed by Buscall and White (Buscall and White, 1987), the Particulate Fluids Processing Centre (PFPC) has developed a comprehensive dewatering characterisation technique, which in conjunction with one-dimensional thickener modelling, allows the effects of flocculation conditions on dewaterability and thickener behaviour to be evaluated. The technique involves flocculating suspensions in a baffle reactor under varying conditions and monitoring the settling rate, supernatant turbidity and equilibrium bed height (Hulston & Scales, 2001). The dewatering properties of the flocculated suspension are then determined by measuring the gel point, ϕ_g, compressive yield stress, $P_y(\phi)$ and hindered settling function, $R(\phi)$. The gel point, ϕ_g, is a measure of the solids volume fraction at which a continuous network structure is formed. This network has a physically measurable network strength denoted as the compressive yield stress, $P_y(\phi)$, which is dependent upon the local solids volume fraction, ϕ (Buscall and White, 1987). The network will remain in its original form until an applied stress exceeds this compressive yield stress. At this point the network collapses, the local volume fraction increases and the system dewaters. The compressibility therefore determines the concentration to which a suspension will dewater to for an applied pressure. The permeability of the suspension is quantified through a parameter known as the hindered settling function, $R(\phi)$ which is a measure of the rate of dewatering (Landman & White, 1994). This parameter accounts for the hydrodynamic interactions between particles, which increase the drag on any given particle in a concentrated suspension and hinder the settling velocity. $R(\phi)$ increases with increasing volume fraction and is inversely related to the permeability.

The solids flux and underflow solids concentration for a particular thickener can be evaluated by the use of a one-dimensional thickener model using $P_y(\phi)$, $R(\phi)$ and ϕ_g as input variables. It should be noted that the model at this stage does not account for the effects of raking within a thickener (Lester, Usher, de Kretser and Scales, 2001).

Due to the complex matrix of red mud suspensions, it is difficult to elucidate the factors controlling flocculation behaviour. As hematite constitutes a significant proportion of red mud it has been used in several studies as a model system (Hulston and Scales, 2000; Jones, Farrow and Van Bronswijk, 1998; Jones, Farrow and van Bronswijk, 1998). Doubt has been cast within the alumina industry over the suitability of hematite as a model system and the purpose of

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the paper is to compare flocculation behaviour of a model hematite suspension with a red mud suspension treated under similar conditions. The behaviour of both suspensions was analysed using the comprehensive dewaterability characterisation technique combined with one-dimensional thickener modelling developed by the PFPC.

Hematite and red mud suspensions were treated with two kinds of flocculants commonly encountered in the Bayer industry. The first flocculant investigated was a medium charge density copolymer of sodium(polyacrylate) and poly(acrylamide) abbreviated as NaAc/ACM. The second flocculant investigated was a hydroxamated polymer, which contained a hydroxamate functional group in addition to the carboxylic acid and amide functional groups present in NaAc/ACM. For ease of communication, the hydroxamated flocculant will be referred to as HXPAM. For each suspension type the mixing conditions (e.g. shear rate and mixing time) were kept constant. These conditions will be referred to as “low shear”. In addition, for HXPAM treated suspensions the shear rate and mixing time were increased to determine the effects of additional shear on dewaterability behaviour. This mixing regime will be referred to as “high shear”.

2. Experimental Methods

2.1 *Materials*

The hematite sample used for the test work was an iron oxide obtained from Aldrich and consisted of 90% hematite and 10% magnetite as determined by X-ray powder diffraction. The sample had a surface area of 3.37 ± 0.01 m^2g^{-1} and was found to be non-porous, as determined by BET (N_2) surface area and porosimetry measurements. Hematite suspensions were prepared at a solids concentration of 120 g/L (2.29% (v/v)) in a background electrolyte solution containing 41.2 g/L of sodium nitrate, 8.2 g/L of sodium hydroxide and 10 g/L of sodium carbonate. The purpose of the sodium nitrate salt in the background electrolyte solution was to replace sodium species, such as sodium aluminate and sodium organics, that are present in a typical downstream liquor. Samples were dispersed by sonication and subsequently mixed for 24 hours on an orbital shaker prior to use. It should be noted that experiments on hematite suspensions were performed at 25°C, while red mud experiments were performed at process temperature (50°C). In order for comparisons to be made between the two systems, the red mud data was corrected to viscosity and density values obtained for the hematite suspension at 25°C.

Red Mud samples were collected from the Alcoa World Alumina Kwinana Refinery site, based near Perth, Western Australia. Experiments were performed on a simulated downstream washer feed, which was prepared by diluting wet sieved (106 μm) washer underflow with washwater. This stock suspension was used for all subsequent flocculation experiments. It should be noted that while the temperature of the washer underflow was 48°C upon sampling, the stock suspension was stored at room temperature. A full liquor analysis of the stock suspension was completed on selected days over the testing period to ensure that no changes in liquor composition occurred. 24 hours prior to flocculation testing, the stock solution was diluted to 63 g/L (2.10% (v/v)) with washwater and stored at 50°C.

2.2 *Flocculation Method*

Flocculation experiments were performed by placing suspensions into a 600 ml baffled, flat bottomed glass vessel fitted with a Rushton impeller. The vessel was based on the standard tank configuration described by Holland and Chapman (Holland and Chapman, 1966), for which the average velocity gradient (G) could be calculated. Samples were covered to minimise evaporation and mixed at the desired shear rate for about ten minutes to allow the system to equilibrate. Flocculant was then injected into the suspension via an inlet tube and the suspension mixed for a given period of time. Upon completion of the experiment the mudline/supernatant interface was monitored until there was no further change in bed height. The settling rate was calculated from the linear region of an interface height versus time graph. The supernatant turbidity was measured using a Hach 2100 AN Turbidimeter thirty minutes after the stirrer had been turned off. Flocculated samples were then collected and the gel point, compressive yield stress and hindered settling function determined (Hulston and Scales, 2000).

2.3 *Gel point determination*

The gel point for each flocculated sample was determined from small scale equilibrium batch settling tests. In these tests a suspension at an initial solids volume fraction, ϕ_0, was allowed to settle to an average equilibrium solids volume fraction, ϕ_{av} for varying suspension heights, h_0. The gel point, ϕ_g, was determined by the extrapolation of ϕ_{av} to zero bed height on a ϕ_{av} versus h_o plot (Green, 1997).

2.4 *Compressibility and Hindered Settling Function determination*

Compressibility and hindered settling function experiments were performed using an automated stepped pressure filtration rig shown schematically in Figure 1. Experiments were performed by carefully loading the sample into a stainless steel cylinder fitted with a membrane, which was supported by a permeable sintered disk at the base of the cylinder. Both the cylinder and base of the filtration rig assembly were fitted with a water jacket to allow experiments to be performed at a given temperature. The sample was compressed by means of a piston, fitted to a double-ended pneumatic cylinder, forcing the liquid to be expressed through the membrane. The delivered pressure at the piston face was measured by a pressure transducer mounted flush in the piston face, while the piston pressure was controlled using a Bronkhorst EL pressure controller. Displacement of the piston was determined using a linear encoder with a spatial resolution of 10 μm.

Compressive yield stress data was obtained by equating the compressive yield stress to the applied pressure when the sample had reached its equilibrium solids volume fraction and stopped compressing. Hindered settling data was determined in a separate experiment by measuring the rate of filtration. Using the stepped pressure filtration technique developed by (de Kretser, Usher, Scales, Landman and Boger, 2001) and validated by (Usher, de Kretser and Scales, 2001), pressures ranging from 1 to 300 kPa could be determined in one experiment.

For pressures below 5 kPa, which is more typical of many washing and thickening type operations, a percolation method was developed to determine the hindered settling function for solids concentrations corresponding to a compressive yield stress of around 1 kPa. In this method, flocculated material was carefully loaded into an 11 cm high water jacketed stainless steel cell fitted with a sintered disk and membrane. The sample was topped up with liquor and the liquor level maintained constant. The mass of liquor passing through the bed of material was logged as a function of time and the average rate of filtration calculated.

Further details on the determination of suspension compressibility and permeability is provided elsewhere (Green, 1997; Landman and White, 1992, 1994, 1995; Usher, 2002).

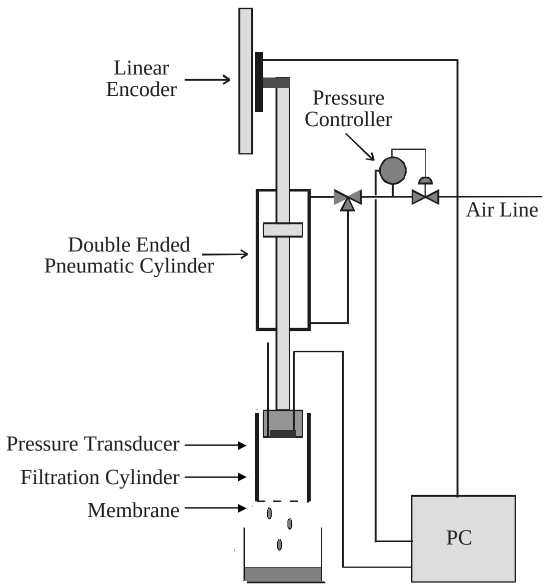

Figure 1 — Schematic of the automated stepped pressure filtration rig used for permeability and compressibility measurements

2.5 *One-dimensional thickener modelling*

Using an algorithm programmed in Mathematica code, the experimentally determined $P_y(\phi)$, $R(\phi)$ and ϕ_g data was used to model the steady state solids throughputs for a range of flat based thickener/washer underflow solids concentrations operating at a bed height of 2 m. It should be noted that the model does not account for shearing, dead-zones or unstable operation and assumes that no solids exit via the overflow (Lester et al., 2001).

3. Results and Discussion

3.1 *Settling and supernatant turbidity behaviour*

The settling rate and supernatant turbidity behaviour of the hematite and red mud suspensions for the various flocculation conditions investigated is shown in Table 1. It should be noted that a significantly higher dose of HXPAM is required to achieve effective flocculation compared to NaAc/ACM. Dose response studies on HXPAM have shown that doses greater than 100 g/t are required to obtain any significant increases in settling rate (not presented here). In the case of NaAc/ACM, polymer doses above about 30 g/t gave rise to a cohesive, gelatinous sediment and very high supernatant turbidities.

In terms of settling rate and supernatant turbidity behaviour, hematite and red mud suspensions showed similar trends with the exception of the low settling rate observed for NaAc/ACM flocculated red mud. While a high settling rate was observed for the NaAc/ACM treated hematite suspension, this could not be reproduced with red mud. To ensure that this effect was not due to incomplete mixing of polymer amongst the red mud suspension, different stirrer speeds as well as doses were investigated. However, no significant further improvement in the settling rate of red mud could be achieved. The reasons for this behaviour are at this stage unclear. Possible explanations may be due to differences in mineralogy, particle shape and size distribution or solution chemistry. Irrespective of the differences observed in settling rate behaviour, NaAc/ACM gave rise to very high supernatant turbidities for both hematite and red mud suspensions, Table 1. This is thought to be due to the break up of aggregates and the steric stabilisation of polymer coated particles or small aggregates.

In the case of HXPAM a high settling rate was observed for both hematite and red mud suspensions, which was significantly reduced when the shear rate and mixing time was increased. This is most likely due to the break up of flocs leading to a reduction in floc size and hence settling rate. Furthermore, an increase in shear rate and mixing time was found to reduce supernatant turbidity, which is believed to be due to the adsorption of particles/aggregates to freshly exposed surface sites created by the break up of floc structures.

The equilibrium bed height data obtained from interface height versus time experiments performed for the various flocculation conditions (Figure 2) showed similar trends between hematite and red mud suspensions. For both suspensions the equilibrium bed height was observed to be higher for HXPAM than either the unflocculated or NaAc/ACM treated suspensions, thus indicating that HXPAM produces a stronger network. Care should be taken when interpreting equilibrium bed height values as the readings are subject to human error and are difficult to determine when the interface is uneven.

3.2 *Compressive yield stress behaviour*

The corresponding compressive yield stress data for hematite and red mud suspensions is shown in Figure 3. The compressive yield stress is a measure of the network strength and increases with increasing volume fraction due to an increase in the number of inter-particle interactions. Results can be interpreted by comparing the applied pressure that is required to achieve a given solids volume fraction.

For both hematite and red mud suspensions similar trends in compressive yield stress behaviour were observed. HXPAM treated suspensions (both high and low shear) were found to have the highest compressive yield stress as suggested from the equilibrium bed height results. This increase in compressive yield stress is thought to be

Table 1 — Summary of flocculation conditions and corresponding settling rate and supernatant turbidity results for hematite and red mud suspensions

Suspension	Polymer	Dose (g/t)	Av shear rate (s^{-1})	Mixing Time (s)	Settling rate (m/hr)	Turbidity (NTU, 30 min)
Hematite	Untreated	0	349	—	1.8	25
	NaAc/ACM	20	349	3	17.3	502
	HXPAM	600	349	3	19.5	167
	HXPAM	600	988	10	8.2	70
Red Mud	Untreated	0	451	—	0.3	144
	NaAc/ACM	20	451	5	2.3	329
	HXPAM	600	451	5	26.9	280
	HXPAM	600	1229	10	5.1	38

due to the higher polymer concentration used. Dose response studies performed on HXPAM flocculated hematite suspensions (not presented here) indicated an increase in compressive yield stress with increasing flocculant dose above 100 g/t. Consequently, as the HXPAM dose is increased the flocs are held together more strongly making it harder for them to be compressed. The increase in yield stress observed for the more highly sheared HXPAM flocculated hematite and red mud suspensions might be due to the formation of a larger number of smaller sized flocs. This is because as the floc size is reduced, the overall surface area is increased giving rise to an increased number of inter particle/aggregate linkages and therefore a stronger network structure. This concept is supported by Zhou et al., (Zhou, Solomon, Scales and Boger, 1999) who investigated the effects of different alumina particle sizes on the shear yield stress, which is a rheological property similar to the compressive yield stress (Zhou, 2000). Zhou et al. found that the shear yield stress and hence network structure is increased with decreasing particle size as a result of an increase in the number of inter-particle linkages, thus supporting the above argument. However, further work is required in this area. A technique to determine the floc size and density is currently under development by the PFPC and it is hoped that valuable information on floc structure will be obtained.

Furthermore, it was found that at higher pressures the compressive yield stress data for the various flocculation conditions investigated converge. This indicates that at sufficiently high pressures the effects of flocculants or floc structure are being overcome and the material starts to behave more like the unflocculated system.

It was also found that the overall compressive yield stress of red mud is higher than hematite. Again, reasons for this trend are difficult to elucidate, but is believed to be due in part to differences in particle size distribution. A particle size distribution (volume %) was performed on both unflocculated hematite and red mud suspensions, the results of which are shown in Figure 4.

The hematite suspension ranged in particle size from 0.74 µm to 9.5 µm, with 4.5% being below a particle size of 1.2 µm. Red mud was found to have a much broader size distribution, with 6.2% of particles being below a particle size of 1.2 µm.

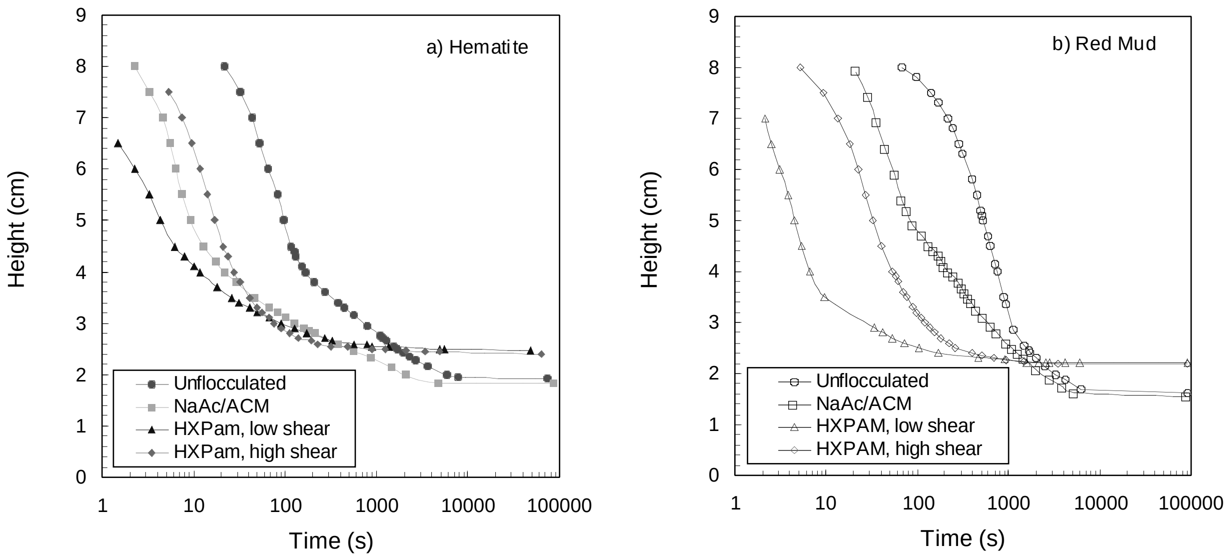

Figure 2 — Interface height versus time results for hematite and red mud suspensions

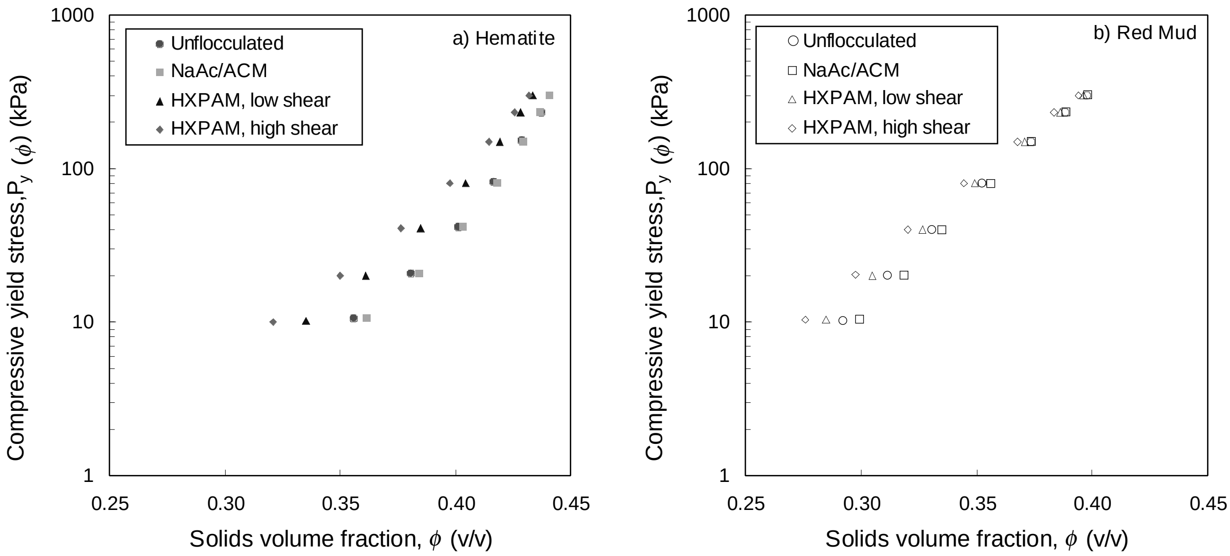

Figure 3 — Compressive yield stress results for hematite and red mud suspensions

While the hematite suspension was analysed using the same background electrolyte solution that the hematite sample was prepared in, the red mud sample was analysed in water. Studies undertaken by Roach et al. (Roach, Jamieson, Pearson and Yu, 2001) indicated that the particle size distribution of red mud slurries is significantly affected by the pH of the suspension. Consequently, when the particle size analysis was performed on the unflocculated red mud sample using water as a medium, the true aggregate/ particle size may not have been captured. Hence, the above particle size results can only be used as a guide. However, particle size analysis performed on red mud by Roach et al. (Roach et al., 2001) at a pH of 11 indicated that ten percent of particles are less than 2.4 μm in size, hence indicating a large percentage of fines. Although the results are not conclusive, the overall higher compressive yield stress of red mud may possibly be due to a greater percentage of fines present in the suspension.

Another possible explanation for the higher compressive yield stress of red mud compared to hematite may be due to an increase in interparticle interaction as a result of red mud suspensions being exposed to higher temperatures. Compressive yield stress studies performed on both unflocculated and NaAc/ACM treated hematite suspensions indicated that the network strength increases with temperature (Hulston, de Kretser and Scales, 2001). The effect was found to be independent of the flocculant used and irreversible under the conditions tested. However, further studies on red mud would be needed to verify this trend.

Other contributing factors leading to a higher compressive yield stress for red mud may include differences in particle mineralogy, particle shape and surface area, particle size distribution as well as differences in solution chemistry (Li and Rutherford, 1996; Roach et al., 2001)

3.3 *Hindered settling function behaviour*

The corresponding hindered settling function data for hematite and red mud suspensions for the various flocculation conditions investigated is shown in Figure 5. For both suspensions the use of flocculants led to a decrease in hindered settling function and therefore an increase in suspension permeability. This is due to the formation of a more porous network structure through which the liquid can permeate. The HXPAM treated suspensions had the highest permeability, where a cross over in permeability behaviour was observed between the low and high shear flocculation experiments. While the more gently sheared HXPAM suspension had a significantly lower permeability than the more highly sheared suspensions, the trend reversed at a solids volume fraction of 4.5% (v/v) for hematite and 7% (v/v) for red mud.

The crossover in hindered settling function for the low and high shear HXPAM flocculated suspensions could be due to differing floc size distributions. For instance the more gently mixed suspension could have a broader size distribution, with the smaller sized flocs filling the voids amongst the larger sized flocs, thus making it more difficult for water to permeate through the bed. In comparison, the more highly sheared suspension could have relatively small and monodispersed sized flocs. This would lead to a low settling rate and hence low permeability at very low volume fractions, but would give rise to a high permeability once a bed has been formed.

It should be noted that the most significant permeability differences between the various flocculation conditions investigated were observed at volume fractions less than about 25% (v/v), which is the region most important for gravity settling devices such as thickener or washers. Above about 25% (v/v) the permeability behaviour for the various flocculation conditions investigated converged.

Furthermore, it should be noted that the overall permeability of the red mud was slightly lower compared to hematite. This is despite the fact that the permeability data was corrected for viscosity differences between the hematite and red mud suspensions. As discussed for the compressive yield stress data, a possible explanation could be the presence of fines in amongst the larger sized particles making it more difficult for water to permeate through the bed.

3.4 *One-dimensional thickener modelling*

In processes where gravity settling devices such as thickeners and washers are used in conjunction with flocculants, a more illustrative method of comparing flocculation behaviour is by means of a solids flux versus underflow solids concentration curve. Using the one-dimensional thickener model and ϕ_g, $P_y(\phi)$ and $R(\phi)$ data as input variables, solids flux curves were modelled for a hypothetical flat based thickener operating at a bed height of 2 m. The solids flux is measured in tonnes per hour per square metre of thickener surface area, where the area is

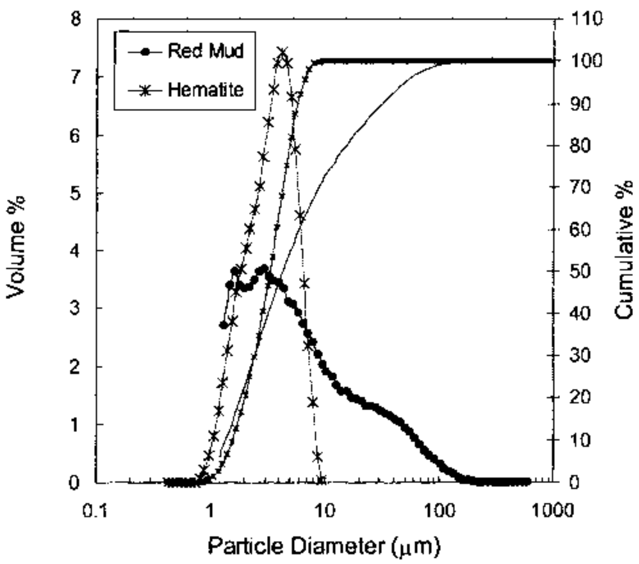

Figure 4 — Particle size distribution of unflocculated hematite and red mud suspensions

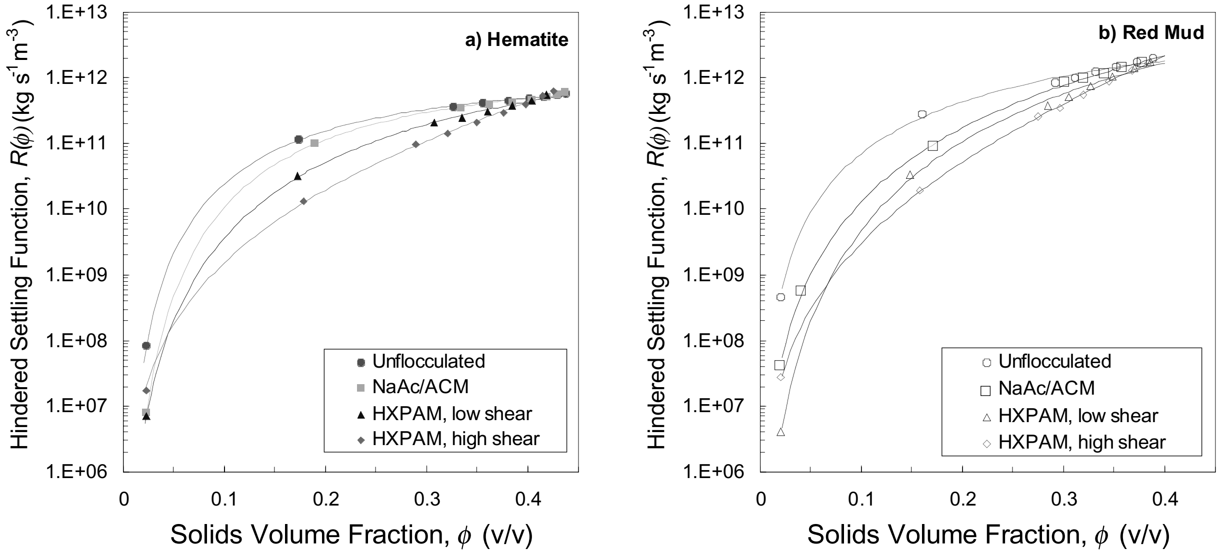

Figure 5 — Hindered settling function curves for hematite and red mud suspensions

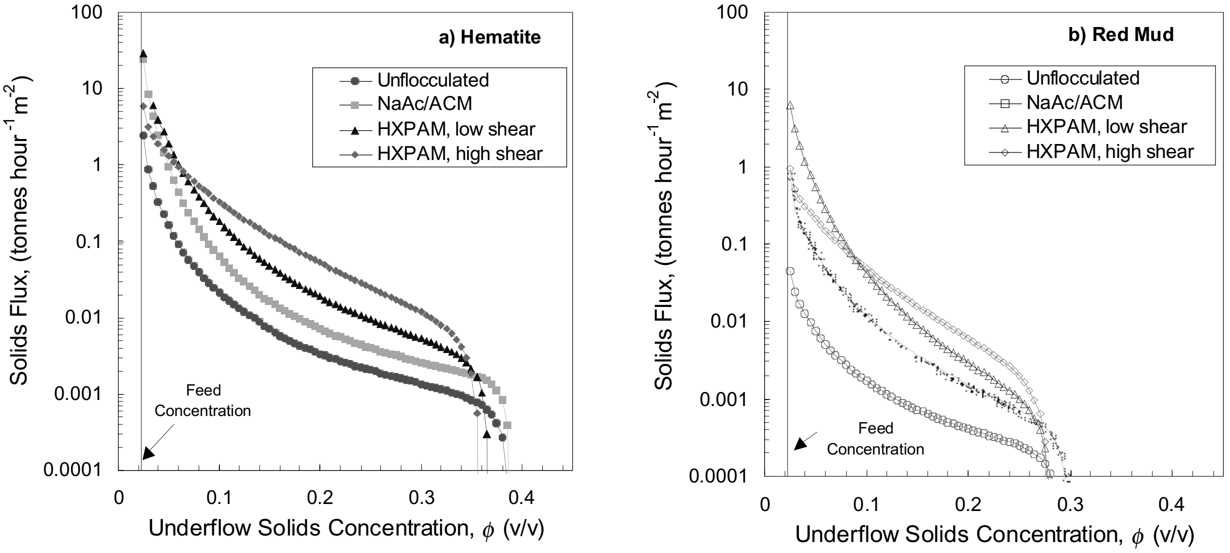

Figure 6 — Solids flux versus underflow solids concentration for hematite and red mud suspensions

the cross sectional area of the thickener at the top of the bed. The underflow solids concentration is expressed as a volume fraction. The fact that hematite and red mud showed similar behaviour in terms of compressibility and hindered settling function data is expressed in the solids flux versus underflow solids concentration curves, Figure 6. The solids flux versus underflow solids concentration graph can be divided into two regions, namely a permeability controlled region and a compressibility controlled region, where the regions are delineated by the inflection point of the bed height being modelled.

If a thickener is operating in the permeability limited regime the achievable underflow solids concentration is controlled by the permeability behaviour of the suspension. Conversely, if a thickener is operating in the compressibility limited region, the achievable underflow solids concentration is controlled by the compressive yield stress behaviour of the suspension. However, most thickeners operate in the permeability controlled region, where the solids flux and underflow solids concentration is improved by increasing suspension permeability. Consequently, while HXPAM flocculated suspensions had a higher compressive yield stress than unflocculated or NaAc/ACM treated suspensions, HXPAM is predicted to yield higher underflow solids concentrations, as it is more permeable. The only exception is for the hematite suspension at very high solids fluxes (e.g. 30 tonnes hr^{-1} m^{-2}) where the NaAc/ACM flocculated suspension is significantly more permeable than the gently sheared HXPAM suspension. For both suspensions a cross over in solids flux behaviour was observed for the low and high shear HXPAM experiments. Consequently, the choice of flocculation condition would be dependent upon the solids flux that the plant is operating at.

Due to the more permeable nature of hematite suspensions, higher underflow solids concentrations are predicted for hematite at the bed height being modelled (i.e. 2 m). For example, at a solids flux of 0.07 tonnes hr^{-1} m^{-2} the gently sheared HXPAM hematite suspension is predicted to reach an underflow solids concentration of 13% (v/v), compared to 9% (v/v) for red mud.

When interpreting the solids flux curve data it should be noted that the one-dimensional thickener model does not include the effects of raking, which may affect thickener performance and hence flocculant choice.

4. Conclusions

The flocculation and dewaterability behaviour of hematite and red mud suspensions were investigated under four different flocculation conditions. Samples were flocculated with HXPAM under low and high shear conditions, as well as with a NaAc/ACM flocculant. Experiments on unflocculated suspensions were also performed for comparative purposes. The flocculation and dewaterability behaviour of the hematite and red mud suspensions were compared by measuring the settling rate, equilibrium bed height, supernatant turbidity, compressive yield stress and hindered settling function. In addition the results were modelled using a one-dimensional thickener model and the predicted model outputs compared.

For the different flocculation conditions investigated, hematite and red mud showed similar flocculation and dewaterability behaviour. The only exception was the low setting rate observed for the NaAc/ACM treated red mud suspension. Furthermore it was found that the red mud suspension is slightly less compressible and less permeable. While the exact cause for this behaviour is unknown at this stage, it is believed that a larger percentage of fines in the red mud may be a contributing factor.

Based on the studies to date, hematite appears to be a good model system for investigating trends in the flocculation and dewatering behaviour of a simulated downstream washer mud. For the flocculation conditions investigated, HXPAM was predicted to yield higher underflow solids than NaAc/ACM, due to the more permeable nature of HXPAM flocculated suspensions.

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