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The effect of process variations on slurry rheology, washer performance and control strategy

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Abstract

Washing circuits at lateritic ore refineries can experience operational issues associated with the end of the washing circuit, where washer overflow becomes excessively turbid, the underflow thickens to the point that the rake cannot move it to the outlet, operation becomes unstable and underflow pumping becomes an issue. In this study, suboptimal flocculation was assumed to be the cause of these operational issues. On site flocculation testing, rheology testwork and particle size analysis, combined with analysis of washer operating data, shows that the effects of process variations on slurry flow properties are significant. Comparison of the control strategies employed by different washers highlighted opportunities to improve process control and subsequently stabilise washing train operation and performance.

1 Introduction

Efficient washing of slurries in a counter current decantation (CCD) circuit requires control of the overflow clarity, the underflow solids concentration and, importantly, the underflow rheology. An understanding of the underflow rheology is prerequisite to ensuring that the washed slurry is able to flow or be raked to the washer outlet and pumped downstream.

In an attempt to understand and mitigate operational issues resulting from high yield stress washer underflow streams in the washing of lateritic slurries, Rheological Consulting Services conducted a series of site tests and follow-on investigations to isolate the factors affecting the rheology of flocculated slurries in the washing trains. As suboptimal flocculation was originally reported to be the cause of the issues, the testwork focused on measuring the shear yield stress of flocculated slurries to determine the efficacy of various flocculants, the dose response and the effect of process variables such as particle size distribution and feed rate. Washer operational data was analysed following the site visit in the light of the outcomes of the site work.

2 Methodology

2.1 Materials and sampling procedures

Samples were gathered from the washing circuit of a lateritic ore refinery. Samples were collected from the penultimate and last washer underflow streams, as these washers had been experiencing operational issues. Three polymers were investigated over the course of the site visit.

The initial primary aim of the site work was to investigate the effect that flocculation conditions have on the rheology of the resultant washer underflow. In order to investigate as many variables as possible in the time available, a general methodology was devised in which a data set was gathered over a two day period, with the first day being used to gather 'baseline' data, and the second day being used to vary an experimental parameter to acquire additional data sets.

The 'base case' yield stress profile was determined for unflocculated washer feed on three different days. On these days, a further yield stress profile was determined for the washer underflow (WUF) and the washer feed was flocculated at the feed solids, dosage rate and with the polymer type being investigated the following day. The sediment from this procedure was concentrated and used to develop an additional yield stress profile.

This general procedure was followed for each of the three polymers investigated. The effect of feed solids was also investigated for one of the polymers, although due to time constraints the baseline data from two days prior was used for comparative purposes.

2.2 Flocculation and sample preparation procedures

Washer feed samples were prepared using the underflow from the penultimate washer and diluting to the last washer feedwell solids concentration for flocculation. Flocculation was undertaken using a bucket-plunge method. A washer feed sample was added to a 20 L bucket and homogenised with multiple rapid plunges. At this point the requisite amount of dilute polymer solution was added to the bucket and dispersed throughout the feed sample with one rapid plunge, followed by three slower plunges to facilitate further mixing and assist floc formation.

The flocculated washer feed was allowed to settle for approximately 30 min., after which time the supernatant was decanted and the flocculated sediment centrifuged to high solids to facilitate shear yield stress profile determination. The high solids flocculated feed was sheared with an overhead stirrer and anchor impeller for approximately 5 min. The resulting slurry was in a partially sheared state at this point.

For the unsheared shear yield stress measurements (discussed in Section 2.4), 1.5 L of washer feed was measured and poured into the test cylinder and an appropriate volume of flocculant was measured in a syringe and set aside. The material in the cylinder was homogenised by plunging rapidly five times. After this step, the polymer was added to the top of the material slowly and in a circular fashion. The polymer was dispersed throughout the material with one rapid plunge, and floc formation was facilitated with three slower plunges. Once flocculated, the cylinder was covered and allowed to rest at ambient temperature for at least eight hours.

2.3 Shear yield stress measurements

Shear yield stress measurements were conducted using the vane shear method (Nguyen and Boger, 1983; 1985) with a Haake VT550 controlled strain rheometer. Samples to be tested were placed in a plastic container and tapped on the laboratory bench to remove entrained air bubbles. A 50 mm long vane with a diameter of 15 mm was rotated at 0.20 rpm and the maximum torque response recorded. All measurements were taken in triplicate to ensure reproducibility of data and to ensure that shear history effects were not evident within the timescale of the measurement.

2.4 Unsheared shear yield stress measurements

The shear yield stress of the unsheared sediments formed from flocculating washer feed samples with various polymers and dosage rates was measured using a custom made detachable settling cylinder, shown in Figure 1.

Once the slurry has been flocculated and allowed to settle, the top half of the cylinder is removed and the supernatant carefully removed with a syringe, enabling a vane to be inserted into the unsheared sediment and the yield stress measured. All measurements were conducted on sediments formed from flocculating 1.5 L washer feed and allowed to settle and rest for at least eight hours before the unsheared sediment yield stress was measured.

Once the initial measurement had been taken, the sediment was sheared a number of times by hand or using an overhead stirrer, with the shear yield stress measured intermittently, in order to give an indication of the effects of shear on the shear yield stress of the sediment. Finally, the solids concentration of the sediment was measured via oven drying.

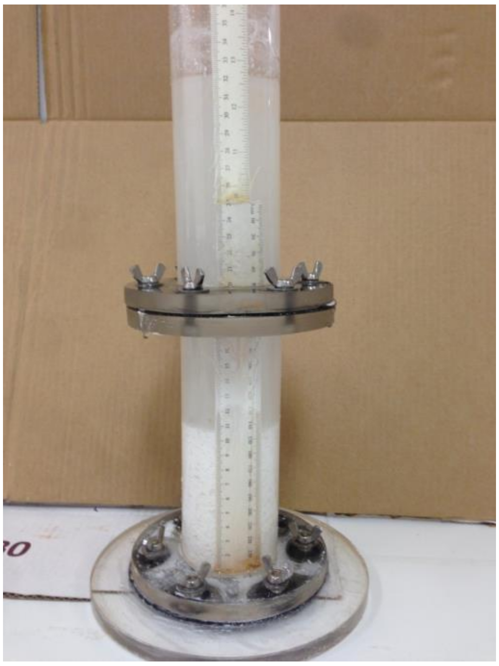

Figure 1 Custom made detachable settling cylinder used during the site visit

2.5 Shear history dependence analysis

Over the course of the site visit, several fresh washer underflow samples were obtained and inspected for thixotropy (whereby the shear yield stress decreases reversibly as a function of time of shear) or rheomalaxis (whereby the shear yield stress decreases irreversibly as a function of time of shear) (Barnes, 1997).

By measuring the shear yield stress of samples from the suction and discharge sides of the underflow pump from the same washer, it can be ascertained whether the flocculated material undergoes yield stress reduction as a result of pumping. However, due to the limited availability of sampling points in the washing train, it was not possible to obtain both suction and discharge side samples across the entire washing train. In instances where discharge side sampling was not possible, the suction side material was used to test for shear history dependent behaviour.

Upon receipt of a sample of underflow (UF) material, the sample was inverted several times to ensure homogeneity, and a sub-sample taken for shear yield stress and solids concentration measurements. Following the initial measurements, the sub-sample was sheared using an overhead impeller for a period of 5 min, and the shear yield stress measured again. This process was repeated until the shear yield stress did not change appreciably between consecutive measurements.

2.6 Particle size distribution measurements

Particle size distribution (PSD) measurements were conducted using a Malvern Mastersizer 2000 laser diffraction particle size analyser. This apparatus has a particle size measurement range of 0.02 μm to 2,000 μm.

Stable samples of washer feed and underflow were obtained by pressure filtration of samples to remove process liquor and prevent sample degradation. The resulting filter cake at was oven dried at 80°C. The dried, stable samples were transported to RCS in Melbourne for laser PSD measurements.

In order to ensure that the material was fully dispersed, the dried samples were re-slurried using laboratory prepared liquor at a similar pH to process conditions, and re-homogenised in a high shear mixing device to form a thick paste. Sample was then added to the Malvern Mastersizer 2000 loading cell until the desired laser obscuration was achieved.

3 Results

3.1 Particle size distribution

Across the course of the site visit, samples were taken for PSD measurements as outlined in Section 2.6. Key particle sizing parameters are given in Table 2, and reflect the variation in sample particle size distribution encountered. The samples are grouped into last washer feed (LWF) and last washer underflow (LWUF) samples.

As seen in Table 1, the d_{50} of the slurries remained relatively stable across the course of the site visit, although the coarse fraction varied significantly.

The high variations in the PSDs of sample were not discovered until the site testwork had been completed. Given that the testwork programme relied upon comparing shear yields stress profiles to illustrate any differences in the behaviour of flocculated samples, such direct comparisons could not be made if the PSDs of the samples differed significantly.

Figure 2 illustrates the fluctuations in the coarse component of the process stream with time, whilst Figure 3 gives differential particle size distributions for three samples with widely differing polydisperse distributions. It has since been confirmed that this level of variability is typical during normal operations.

Table 1 Particle sizing parameters for samples taken during site visit

Sample ID	Mean particle size (µm)	d_{50} (µm)	d_{80} (µm)	d_{90} (µm)
Day 1 LWUF (PM)	27	8	33	77
Day 2 LWF (AM)	63	16	124	198
Day 2 LWF (PM)	48	9	86	160
Day 2 LWUF (PM)	52	13	88	155
Day 3 LWF (AM)	34	8	41	92
Day 4 LWUF (AM)	63	11	110	195
Day 7 LWF (AM)	45	9	65	138
Day 7 LWUF (PM)	45	8	64	128
Day 8 LWF (AM)	50	10	87	161
Day 9 LWF (AM)	46	8	81	147
Day 9 LWUF (AM)	31	7	45	98
Day 10 LWF (AM)	41	8	69	130
Day 11 LWUF (AM)	61	8	122	195

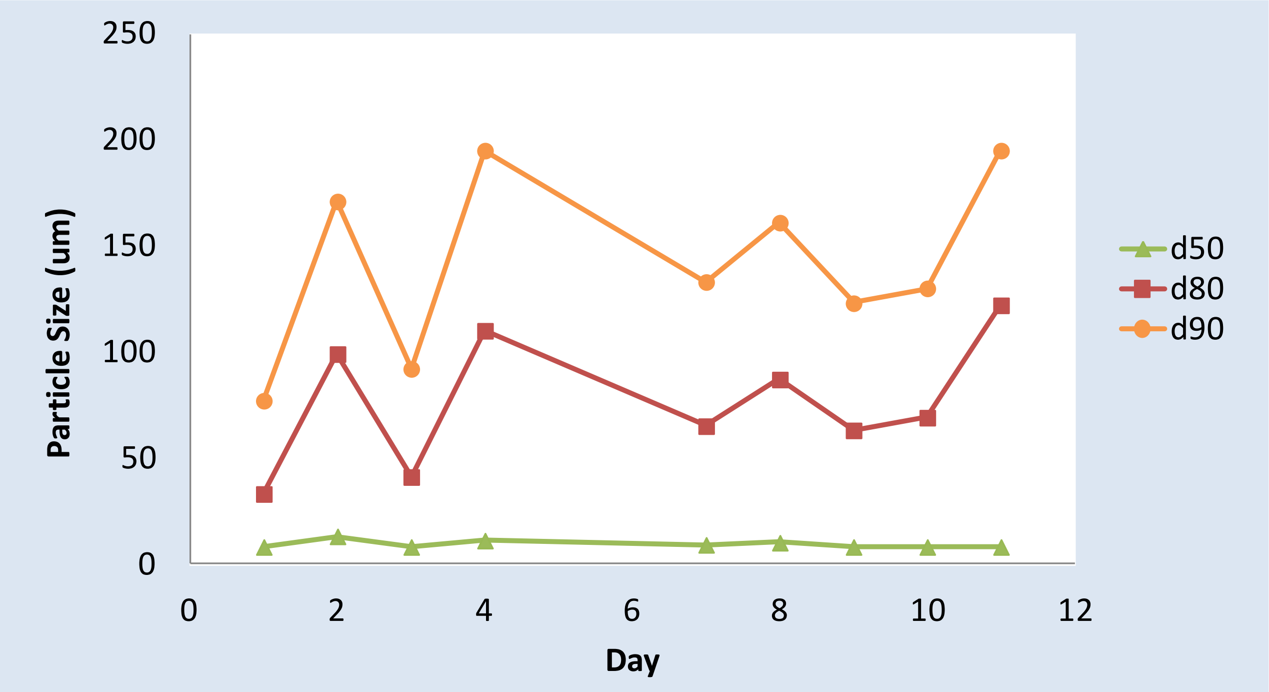

Figure 2 Fluctuations in washer feed and underflow sample particle size distributions with time

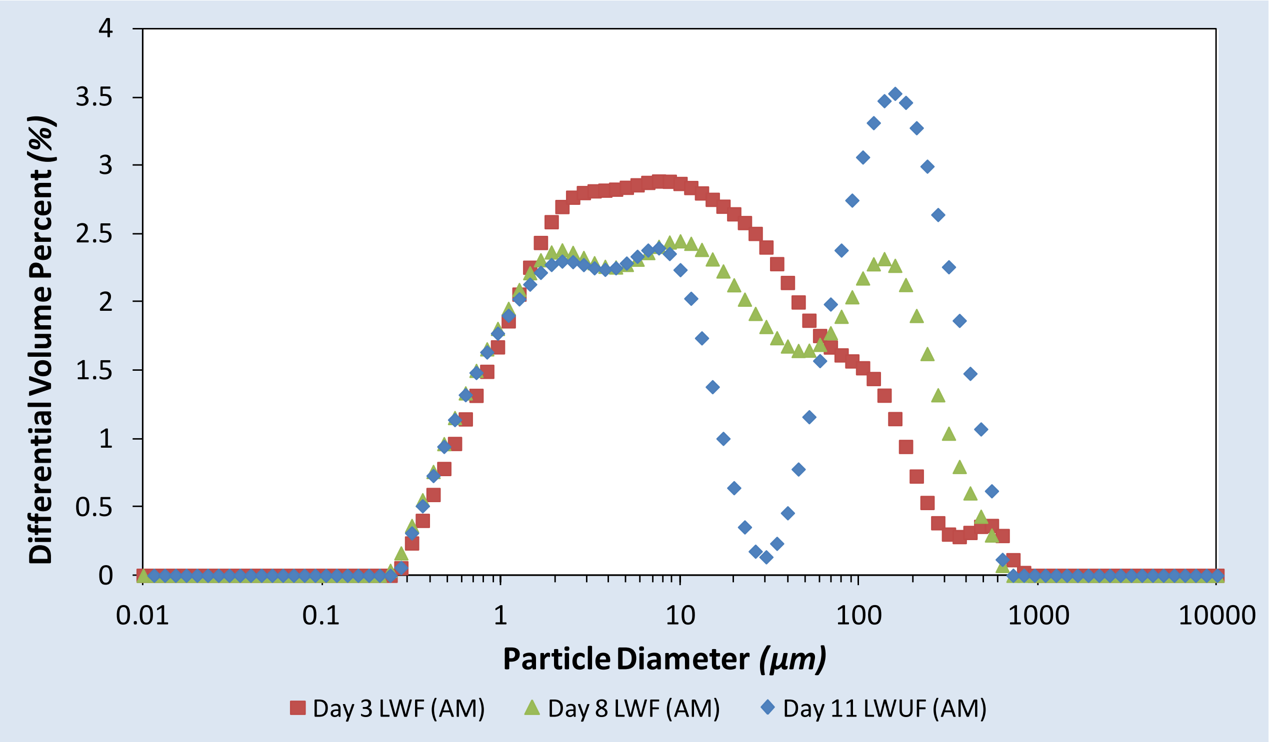

Figure 3 Differential PSD for three samples taken over course of site visit

3.2 Effect of flocculant type, dosage, feed solids

Due to the varying particle size distributions of samples prepared and flocculated on different days, it was not possible to use shear yield stress profiles to directly compare the effects of varying flocculation parameters in many instances, as the particle size distribution of a sample heavily influences the net inter-particle forces and hence the shear yield stress (Johnson et al., 2000). Taking this into account, the shear yield stress profiles generated nonetheless suggest that there is very little effect of flocculant dosage rate or feed solids concentration on the behaviour of partially sheared flocculated material. Where any differences occurred, they did not follow a distinct trend due to flocculant type or dosage. The effect of flocculant dosage rate on the sheared sediment behaviour is shown for samples flocculated with Floc 1 in Figure 4. Flocs 2 and 3 gave similar results.

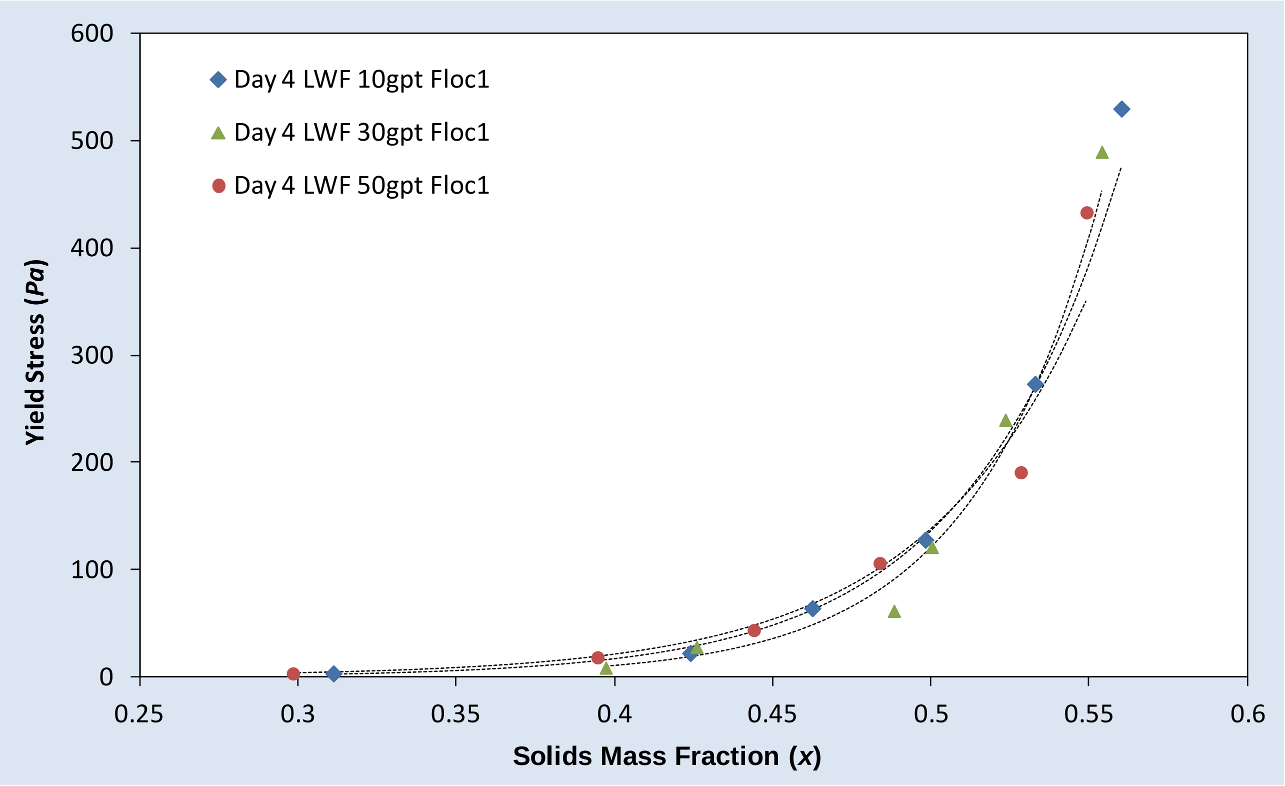

Figure 4 Shear yield stress as function of solids concentration for sheared samples flocculated with Floc 1 at various dosage rates

The variable PSD precluded comparison between the behaviour of samples flocculated with different polymers and at different feed solids concentrations. An example of this is seen in Figure 5. The shear yield stress profiles suggest that, at this dosage rate, the shear yield stress of material flocculated with Floc 2 will be significantly less than that of material flocculated with Flocs 1 and 3 at a given solids concentration. However, analysis of the PSD data indicates that the samples used with Flocs 1 and 3 had similar particle size distributions, with mean particle sizes of 34 and 41 µm respectively. The sample used with Floc 2 had a larger mean particle size of 50 µm and, importantly, a d_{90} of 161 µm, indicating a significant coarse fraction. The coarser nature of this sample when compared to those treated with Floc 1 and Floc 3, rather than any polymer effect, is most likely responsible for the behaviour seen in Figure 5.

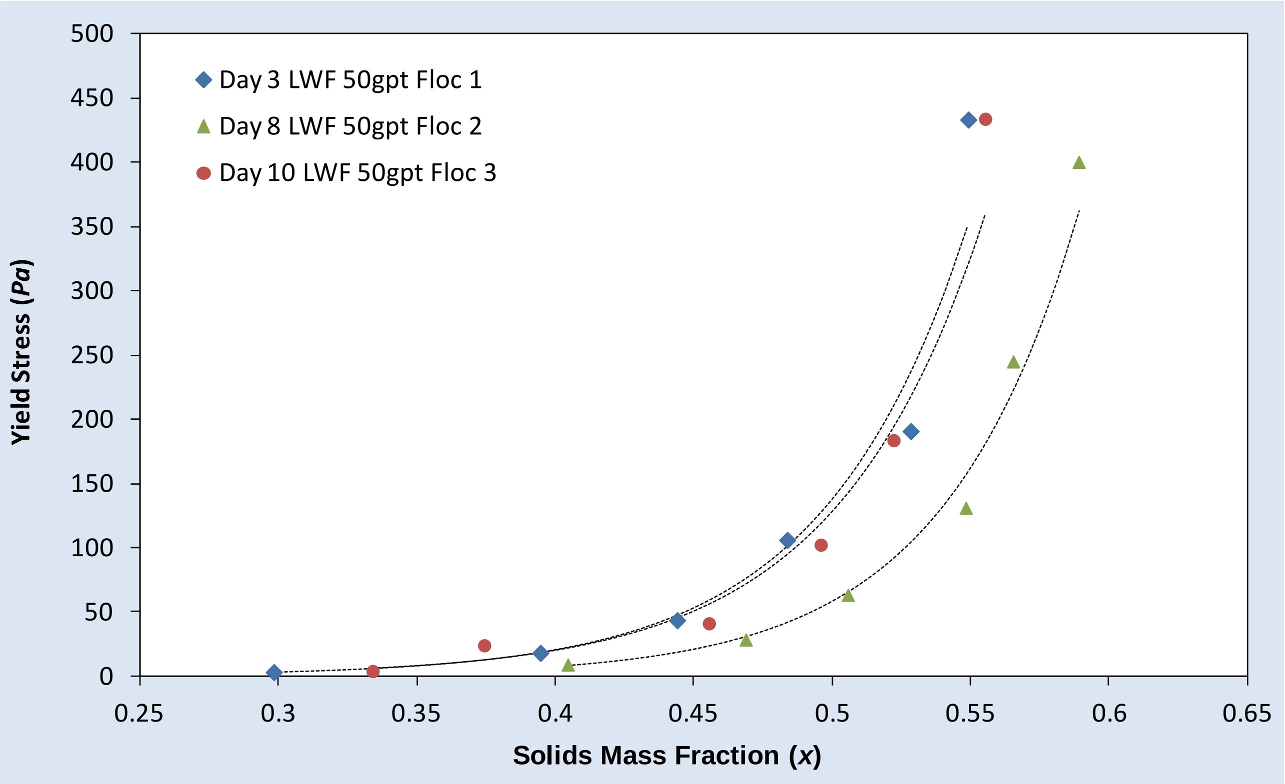

Figure 5 Shear yield stress as function of solids concentration for sheared samples flocculated with various polymers at 50 gpt

3.3 Shear history effects

The shear history analysis of the last washer underflow samples indicated that all samples tested on both the suction and discharge sides of the pump were not fully sheared, with each sample displaying a shear induced irreversible reduction in yield stress in the approximate range of 30–50% of the initial shear yield stress. The results of the shear history testing are given in Table 2.

Table 2 Shear yield stress and solids concentration data for thixotropic analysis of LWUF samples

Sample ID	Solids concentration (wt%)	Initial yield stress (Pa)	Final yield stress (Pa)	Yield stress reduction (%)
Washer 1, Suction	39.9	20.9	12.8	38.8
Washer 2, Suction	42.7	37.5	17.6	53.1
Washer 3, Suction	40.5	18.4	12.7	31.0
Washer 4, Suction	38.7	20.8	12.1	41.8
Washer 4, Discharge	39.1	16.2	11.4	29.6

Table 2 indicates that, as expected, the suction side samples taken showed the greatest decreases in shear yield stress with shear, with up to a 50% reduction being seen for the suction sidpe sample from Washer 2. This indicates that the material on the suction side of the underflow pump is not fully sheared, and the flocculated network structure is still partially intact. The effect of the discharge pump is evident on the Washer 4 sample. Both the suction and discharge samples have a nominal solids concentration of 39 wt% solids, and the initial shear yield stress of the discharge sample (16.2 Pa) is lower than that of the suction sample (20.8 Pa), indicating that the shear imparted by the discharge pump has induced thixotropic breakdown. However, in this instance, the discharge sample underwent further thixotropic breakdown, with the shear yield stress decreasing by a further 30% with additional shear. This suggests that the shear imparted by the discharge pump alone is not sufficient to fully shear the flocculated sediment. These shear induced rheology changes are typical of those generally observed in thickener underflows (Sofra and Boger, 2002).

3.4 Unsheared shear yield stress results

Table 3 shows the results of the unsheared sediment yield stress testwork undertaken on flocculated samples of last washer feed. Once again, variations in the PSD of the feed samples used for each test disallow direct comparisons between the relative performance of each of the floc types. However, each floc type showed a trend of increasing yield stress of the unsheared sediment with increasing floc dosage rate across the range of conditions investigated. This trend is illustrated graphically in Figure 6.

For all floc types investigated, the shear yield stress of the sediment bed decreased dramatically with shear. As shear breaks down the flocculated network structure, destroying bonds between flocs and particles within the network and hence lowering the shear yield stress of the sediment, this result was expected. The magnitude of the difference between the shear yield stress of unsheared and sheared sediment samples indicates that any dead zones or regions of low shear within the washer could lead to a region of high yield stress material, leading to significant raking and pumping issues, as well as detrimental flow phenomena such as rat-holing.

Table 3 Shear yield stress and solids concentration data for unsheared shear yield stress tests

Flocculant	Dosage (gpt)	Unsheared yield stress (Pa)	Sheared yield stress (Pa)	Sediment solids (wt%)
Floc 1	10	79.24	6.941	31.0
	20	90.81	2.892	27.7
	50	137.1	2.314	26.3
Floc 2	10	42.22	2.892	27.7
	20	103.5	3.471	28.6
	50	114.8	2.892	27.4
Floc 3	10	63.05	2.892	29.8
	20	85.61	2.314	26.8
	50	153.3	3.471	29.8

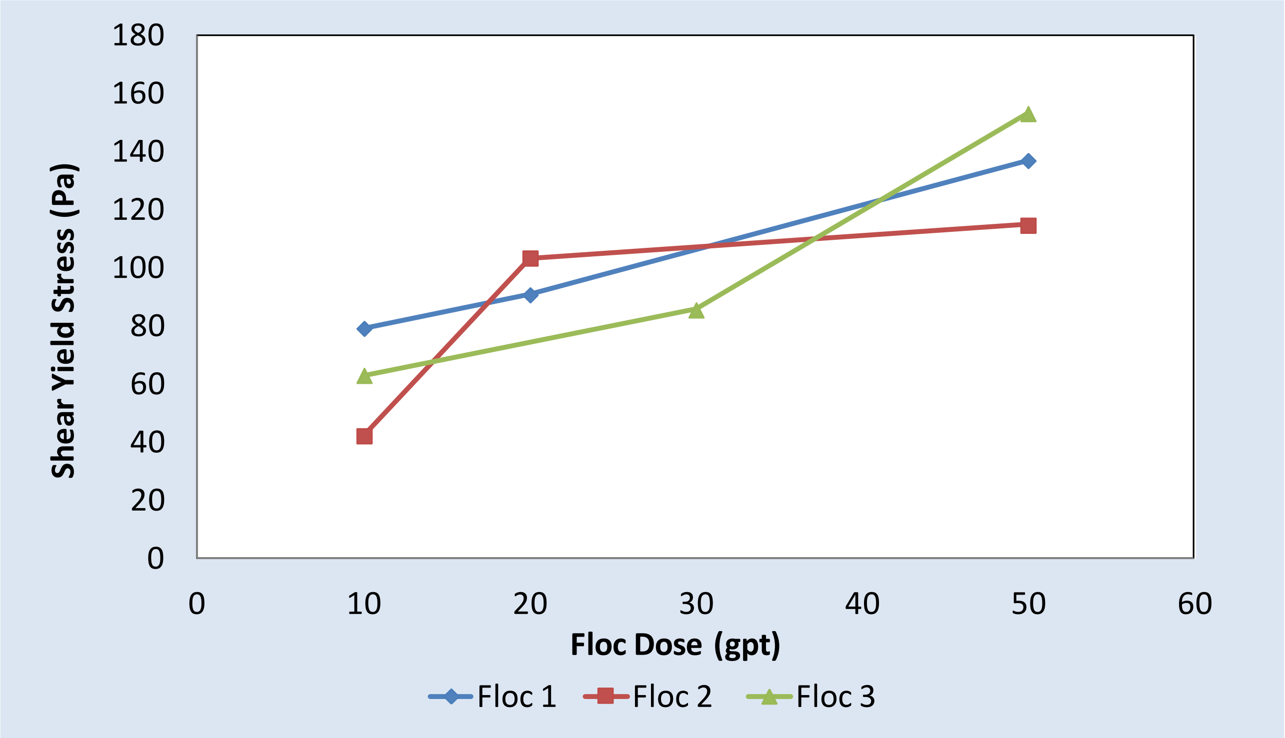

Figure 6 Unsheared sediment shear yield stress for LWUF samples flocculated at various dosage rates

4 Washer operating data analysis

Following the site work, RCS requested the operational logs for the penultimate and last washers, around which the site work was focussed. It was hoped that, in the absence of any conclusive results from the site work, the operating trends could shed light on the causes of the operational issues. For the purposes of this investigation, the most pertinent operational parameters are as follows:

- Feed rate (tph).
- Floc dose (gpt).
- Sediment bed height (m).
- Rake pressure (bar).

The variations in these parameters for each of the second last and last washers are given in Figures 7 through 10. PW represents penultimate washer data and LW represents last washer data.

The data indicates that both the penultimate and the last washer experience similar deviations in both the feed rate and the floc dose, however only the last washer is experiencing dramatic variations in bed height and frequent rake over-torque. This observation led to an investigation of the control regimes used in each washer. It was discovered that the penultimate washer uses the sediment bed height as its primary control parameter, whilst the last washer uses rake pressure as its primary control parameter. The issues encountered in the last washer most likely stem from the significant fluctuations in feed rates coupled with a control strategy which does not allow the washer to reach approximate steady state operational conditions.

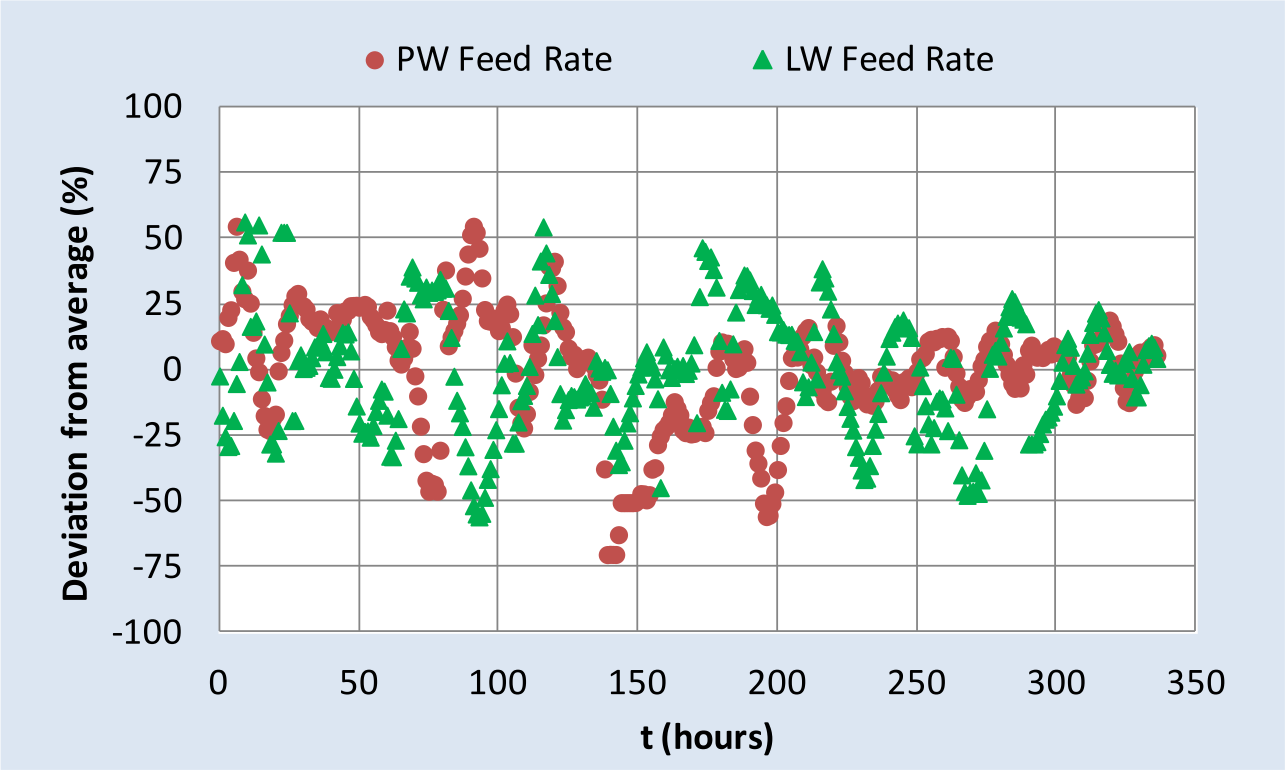

Figure 7 Variations in feed rate for penultimate and last washers across course of site visit

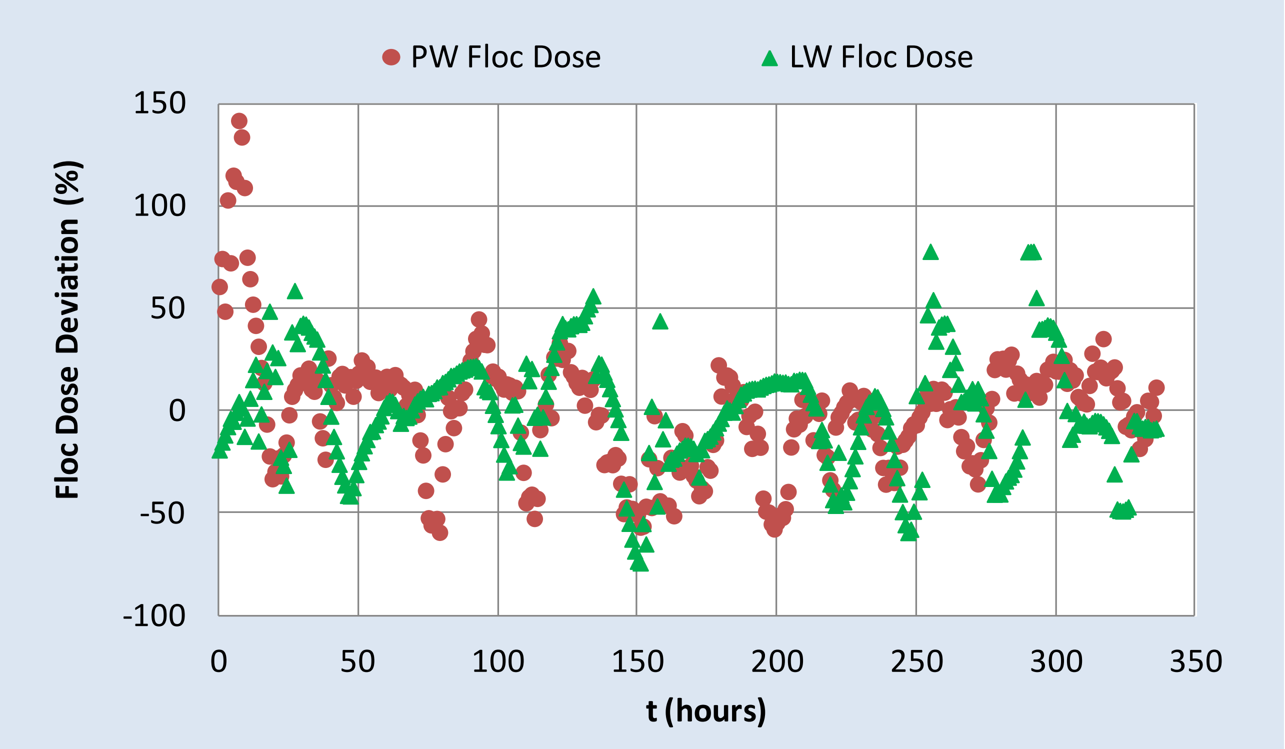

Figure 8 Variations in floc dose for penultimate and last washers across course of site visit

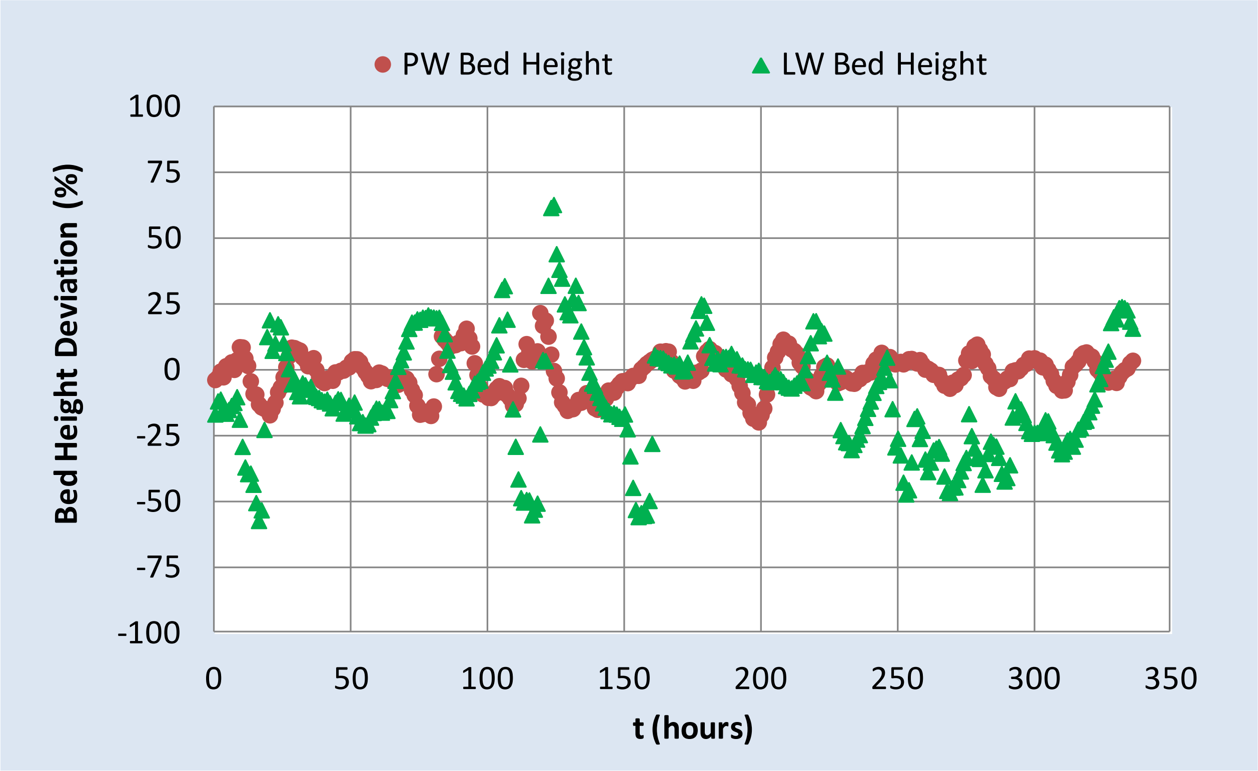

Figure 9 Variations in bed height for penultimate and last washers across course of site visit

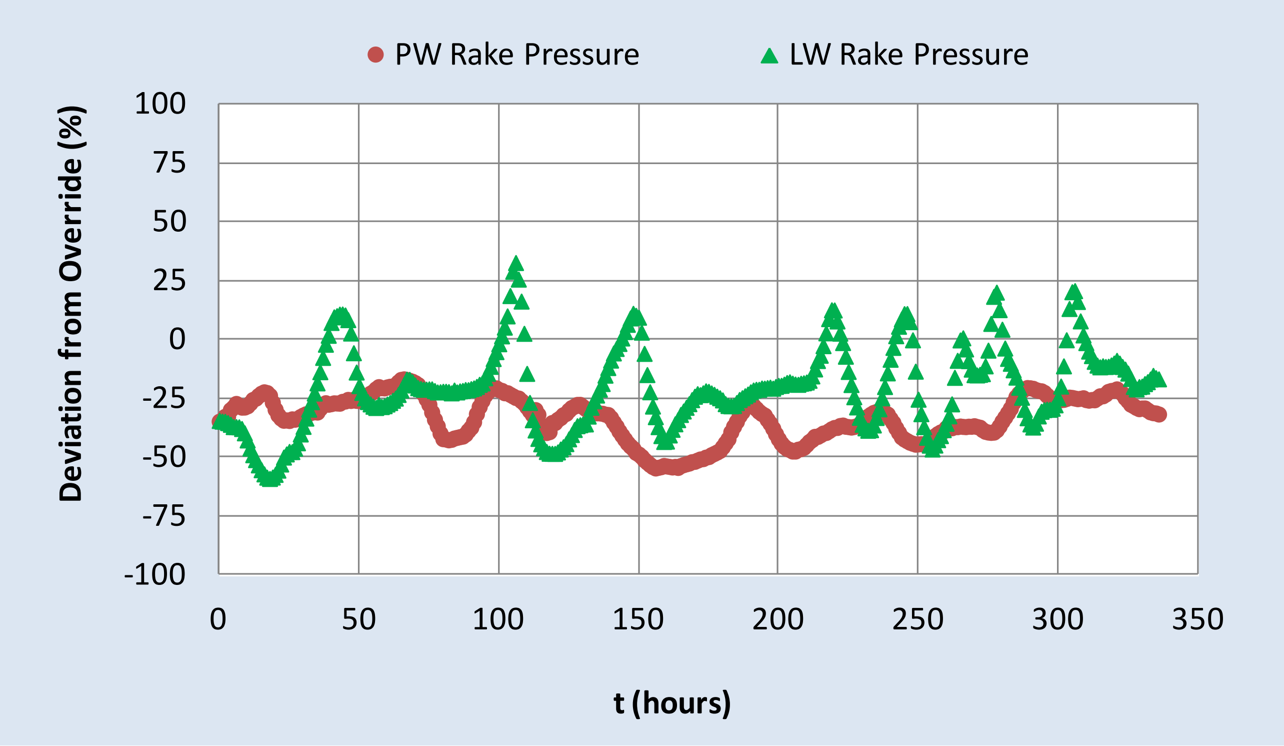

Figure 10 Variations in rake pressure for penultimate and last washers across course of site visit

The primary control parameter for the last washer is rake pressure. An increase in rake pressure is met with a decrease in floc dosage rate in order to reduce this pressure, which then falls below the set point, triggering an increase in the floc dose. The bed pressure then rises to an unacceptable level and the cycle continues, with bed pressure and floc dose constantly fluctuating in an opposing and extreme manner. This cycle is highlighted by considering the plot of rake pressure vs floc dosage for the last washer, as given in Figure 11.

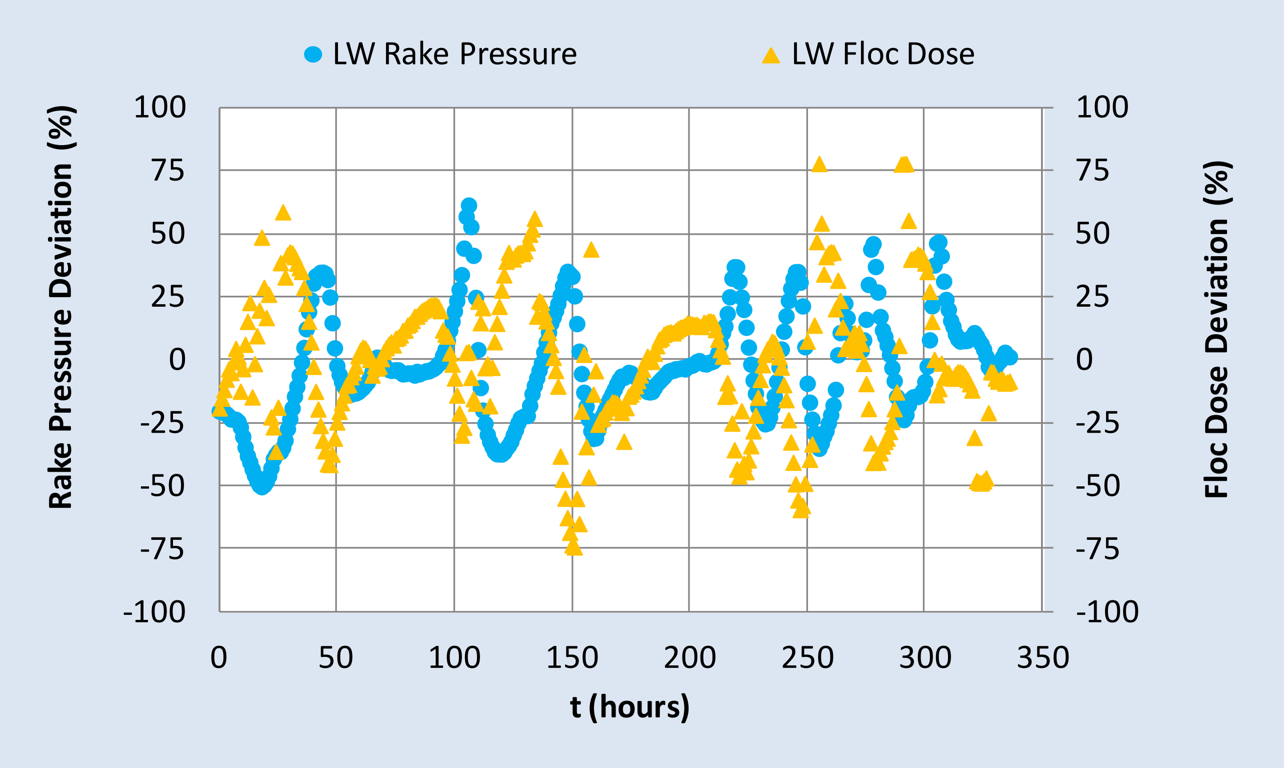

Figure 11 **Last washer rake pressure and floc dose deviations across the course of the site visit**

The increase in floc dosage rate increases the settling rate and causes the bed height to rise. The over-flocculated material has a higher network strength (yield stress) at a given solids concentrations than material flocculated at lower dosage rates. As such, an increase in floc dosage rate increases rake pressure and causes the floc dosage to be reduced before a steady state is reached. The result is that only a portion of the bed is over-flocculated, and once this portion reaches the rakes it will become sheared, lose its strength and collapse, causing a decrease in bed height and rake pressure, and prompting the cycle to begin again.

In contrast to the last washers, the penultimate washers are operated to a constant bed height. The solids concentration profile in the bed is monitored and a pre-determined solids concentration is chosen to represent the surface of the bed. Under this control strategy, an increase in bed height is seen as excessive turbidity in the region above the bed, and the flocculant dosage rate is increased to improve supernatant clarity. The response of floc dose to changes in bed height is shown in Figure 12.

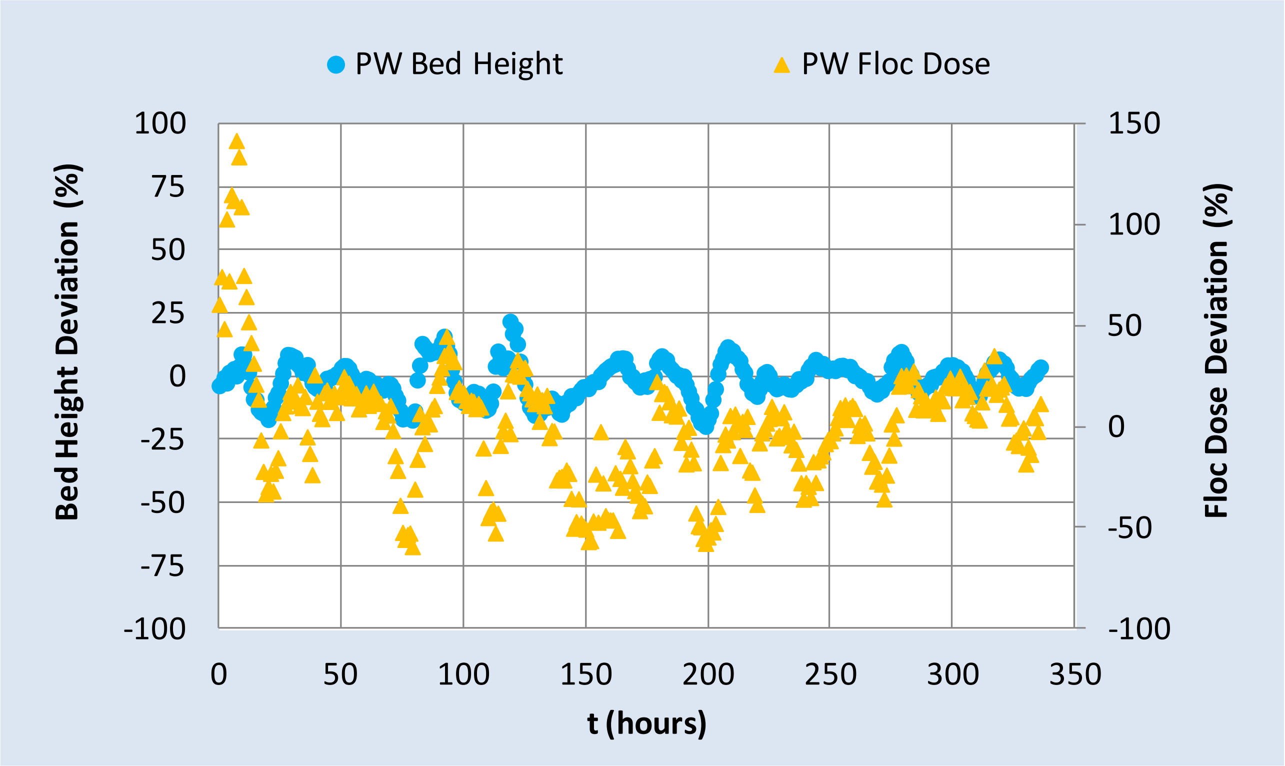

Figure 12 **Penultimate washer rake pressure and floc dose deviations across the course of the site visit**

Under this control regime, the bed height and rake pressure are far more stable than for the last washer, as seen in Figures 8 and 9. Based on the operating data provided, the control strategy implemented in the penultimate washer is more effective at maintaining rake pressure (and hence steady operation) than the strategy used in the last washer. By using the floc dose to control the bed height rather than the rake pressure, the temporal lag between the adjustment of the floc dose and the resultant process response is minimised. However, using floc dose to respond to rake pressure, which could mean that up to one residence time may pass before the changes are seen in the raked zone, is ineffective and leads to the kind of extreme fluctuations seen in Figure 11.

In light of the flocculation study results and upon analysis of process data it is evident that fluctuations in process conditions (most notably feed rate and feed particle size distribution) dominate the effects of suboptimal flocculation. Attention is now focussed on improving control strategies to stabilise operation. Once operation is more stable, flocculation conditions can be more effectively optimised.

5 Conclusions

The effects of flocculant type, flocculant dosage rate, feed solids and liquor viscosity on partially sheared, flocculated samples were investigated. The results of the testwork indicated that none of the parameters investigated had a significant effect on the rheology of partially sheared, flocculated material, with neither polymer performing more favourably than others on the basis of the rheology of the resulting flocculated mud.

The shear yield stress of unsheared, flocculated washer feed was investigated for all polymer types as a function of dosage rate. The results of these experiments indicate that, for all flocculants investigated, the shear yield stress of the unsheared, flocculated mud increases with increasing flocculant dosage rate. The networked structure of all flocculated samples broke down dramatically with the application of shear. The magnitude of the difference between the shear yield stress of unsheared and sheared sediment samples indicates that any dead zones or regions of low shear within the washer could lead to a region of high yield stress material, leading to significant raking and pumping issues.

The underflow streams from the penultimate and last washers were investigated for evidence of shear history effects. For all streams investigated, the analysis showed that thixotropic breakdown occurred with the application of shear, with the shear yield stress of the underflow decreasing by more than 50% in some instances. Furthermore, the shearing imparted by the underflow pumps does not appear to shear the underflow to an equilibrium state, implying significant improvements in pumping performance could be gained by recirculating a portion of the underflow discharge mud back to the suction side.

Across the course of the site visit, multiple samples were taken for laser PSD analysis in the RCS laboratories in Melbourne. The d_{50} of the slurries remained relatively stable across the course of the site visit, although the coarse fraction varied significantly. The variable PSD precluded comparison between the behaviour of samples flocculated with different polymers and at different feed solids concentrations. Such analysis should be a core component of future work in situations with a significant variation in process PSD, as it allows for better planning of testwork to ensure proper sampling techniques are formulated and results are more easily compared.

Subsequent analysis of the penultimate and last washer operating data indicates that both washers experience similar deviations in both feed rate and the floc dose, however only the last washer is experiencing dramatic variations in bed height and frequent rake overtorque. Analysis of the control regimes used in each washer suggest that the issues encountered in the last washer most likely stem from the significant fluctuations in feed rates coupled with a control strategy which does not allow the washer to reach approximate steady state operational conditions. In contrast to the last washer, the penultimate washer is operated to a constant bed height. Based on the operating data provided, the control strategy implemented in the penultimate washer is more effective at maintaining rake pressure (and hence steady underflow rheology and operation) than the strategy used in the last washer.

In light of the flocculation study results and upon analysis of process data it is evident that fluctuations in process conditions (most notably feed rate and feed solids concentration) dominate the effects of suboptimal flocculation. Attention is now focussed on improving control strategies to stabilise operation. Once operation is more stable, flocculation conditions can be more effectively optimised.

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