Impact of Excess Synthetic Flocculent on Security Filtration

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Abstract

Synthetic polymers derived from acrylic acid and acrylamide are used in the Bayer process to assist the separation of bauxite residues from the liquor.

These contemporary flocculents have replaced starch mainly because of their high efficiency found at relatively low dosage. However usage of excess flocculent can lead to reduced performance in liquor filtration and/or operational problems due to build-up of compacted mud onto the vessel's internals such as the rake.

This work outlines the results obtained from an extensive investigation carried out in one of RTA alumina refineries after the commissioning of the new high rate decanter and security filter facilities. The impact of carried-over flocculent and other operating parameters such as filteraid (TCA) dosage and total suspended solids (TSS) on liquor filterability have been evaluated and are discussed. The paper also includes the basis of a laboratory method to quantify flocculent in Bayer liquor at sub ppm level.

Introduction

As part of the expansion of one of its alumina refineries, Rio Tinto Alcan (RTA) installed four new high rate decanters (HRD) together with self dumping filters. Based on previous plant experience, a specific filtration rate greater than 1.0 $m^3/h/m^2$ was expected from the new filters.

Following initial commissioning, it was observed that the performance of the filters was significantly below the expectations with filtration rate on the order of 0.6 $m^3/h/m^2$. Poorer liquor clarification and filteraid (TCA) preparation were first suspected to have a negative impact on filtration performance. Various combinations of synthetic flocculent, method of preparation and dosage were also suspected to contribute to the problem.

A series of tests were carried out to monitor the quality of the new TCA make-up facilities and many changes to the dosing strategy of TCA to feed-to-filter liquor were made. The flocculent make-up solution was corrected as per standard which resulted in decreasing considerably the total amount of flocculent added to HRD which in turn led to increase the average lifetime of the units.

Despite a better tank stability and a reduced TSS supernatant, the filtration performance of the new filters was still far from the design target, even with an increase of TCA to feed-to-filter liquor.

Facing this situation, an extensive investigation program was undertaken consisting of plant monitoring coupled with laboratory pressure filtration tests. The paper describes how the excess flocculent carried over in the HRD supernatant is quantified and discusses its major detrimental effect on security filtration.

Theory

The behaviour of filtration under constant pressure can be described by Darcy's law as follow in its integrated form:

$$t = \frac{\mu \alpha W}{2 \Delta P A^2} V^2 + \frac{\mu R_s}{\Delta P A} V$$

equation (1)

where:

t	= filtration time (s)
V	= filtrate volume at (t) in (m^3)
μ	= filtrate viscosity (Pa.s)
W	= mass of filter cake per unit volume of filtrate (kg/m^3)
ΔP	= filtration pressure (Pa)
A	= filtration area (m^2)
R_s	= filter resistance (1/m)
α	= filter cake resistance (m/kg)

Equation (1) can be re-arranged in a linear form as follow:

$$t = a\left(\frac{V}{A}\right)^2 + b\frac{V}{A} \Rightarrow \frac{t}{\left(\frac{V}{A}\right)} = a\left(\frac{V}{A}\right) + b$$

equation (2) and (3)

with:

$a = \mu W \alpha / 2\Delta P$ and $b = \mu R_s / \Delta P$

Thus, plotting [t / (V/A)] as a function of (V/A) will describe a linear relation having "a" for slope and "b" as ordinate. This approach was used earlier a reported by Malito [1].

Experimental

The determination of the filter cake resistance (FCR) by mean of controlled laboratory tests allows relating the filtration performances to variations taking place in the process. Changes in TCA, TSS and flocculent dosage can provoke a quantifiable and sensitive change of FCR as it will be shown in the next sections. The analysis of the results obtained in using controlled laboratory conditions can lead to conclusions which can then be translated to the process by extrapolation.

In this work, the filtration tests were performed at 95°C and 2.7 bars (40 psi) using a 2.5L laboratory filtration cell and Whatman no.42 membrane of 13.3cm diameter. In a typical filtration sequence, precisely 2500 mL of liquor containing filteraid (TCA) is poured into the filtration cell. The lid is closed, the pressure is set on and immediately filtration time of the filtrate volume is recorded by increments of 200 mL until empty. The values of "t / (V/A)" are plotted against "V/A" to yield a linear curve with its "a" and "b" coefficients. The data are fitted by least-squares analysis for the linear portion. The cake is assumed to be incompressible throughout this work. Figure 1 displays a typical filtration curve obtained with pregnant liquor (PGL) containing a known amount of flocculent.

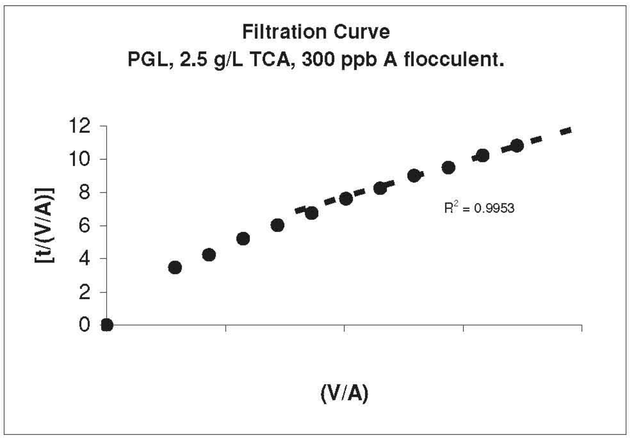

Figure 1. Filtration curve of pregnant liquor (PGL) containing 300 ppb of flocculent A.

Controlled addition of flocculent (liquor spiking)

A series of filtration tests were undertaken to study what effect the residual flocculent by itself may have on the filtration cake resistance. Both flocculents were prepared at an active concentration of 0.1500 g/L in 25 g/L Na_2O aqueous solvent. Precise amounts of flocculent are added to PGL to obtain excess flocculent in the range of 0-300 ppb active. Prior to filtration 2.0 g/L of plant TCA are added to the PGL doped with flocculent. Figure 2 shows the negative impact of flocculent

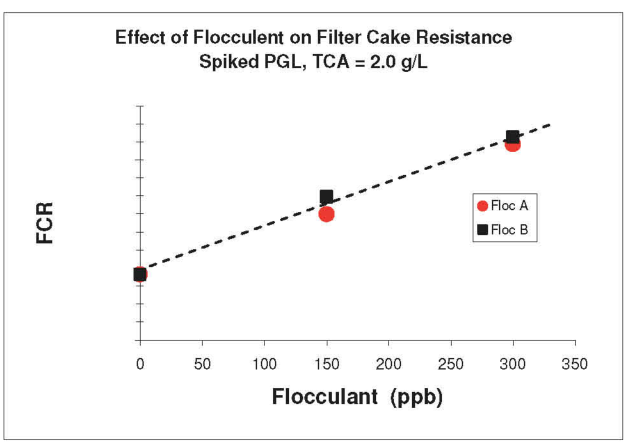

Figure 2. Increase of FCR for pregnant liquor (PGL) containing 0-300 ppb of flocculent.

added to PGL. It can be seen that only 150 ppb of flocculent A or B causes FCR to double in value. This effect is emphasized with the presence of 300 ppb of flocculant where FCR almost triples.

Laboratory determination of FCR for plant samples.

Several samples of decanter overflow and feed-to-filter (overflow with TCA) slurry were collected simultaneously in the plant and analyzed in the laboratory. The turbidity and the total suspended solids of the decanter overflow were determined and varying amounts of plant isolated TCA were added to the overflow to imitate plant feed-to-filter slurry. The filtration test results yield a plot of FCR as a function of TCA dosage. Figure 3 provide an example of such a curve obtained for one decanter overflow on a particular day.

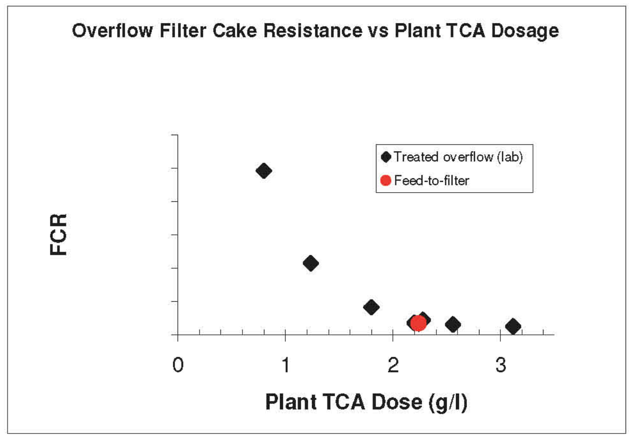

Figure 3. Variation of filter cake resistances of decanter overflow with dosage of TCA.

The influence of TCA dosage on FCR is clearly emphasized by the results of Figure 3. In this particular case, optimum filtration rate would be obtained with TCA dosage greater than 2 g/L. It is also possible from these results to estimate the precision of this filtration method. Indeed, duplicate analysis of overflow samples were made by adding 2.2 and 2.3 g/L of TCA. These results are compared to the FCR of the plant feed-to-filter slurry containing 2.24 g/L TCA. Table 1 shows the reproducibility of the filtration method below.

Table 1: Reproducibility and accuracy of the filtration test method.

	TCA dose (g/L)	FCR (m/kg)
First test	2.20	4.56E+10
Second test	2.28	5.36E+10
Plant "Feed-to-Filter"	2.24	4.35E+10
Average	2.24	4.76E+10
Standard deviation	0.04	0.53E+10

Then FCR data were collected over a period of seven consecutive days while the operation of the HRD was very stable and the flocculent dosage maintained between 50-60 g/ton active. Figure 4 displays the FCR values obtained for one decanter overflow to which controlled amounts of TCA were added. Additionally, on the same graph, FCR values of plant feed-to-filter slurry samples are compared against the TCA dosage.

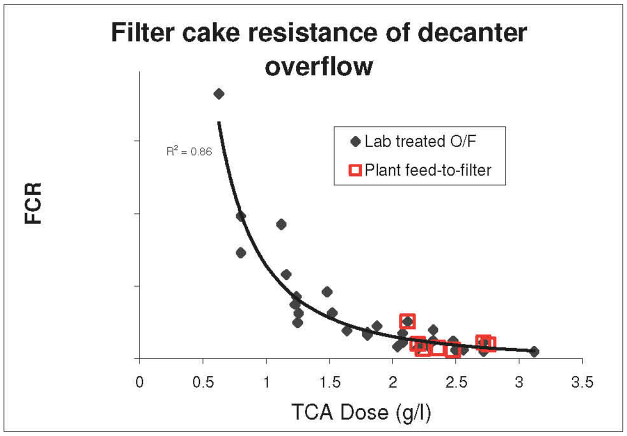

Figure 4: Baseline curve for filter cake resistance of decanter overflow. Comparison of laboratory prepared with plant feed-to-filter samples.

As in the previous example, there is a good relationship between the filtercake resistance and TCA dosage. There is also an excellent correspondence between the FCR of the plant feed-to-filter samples with the FCR obtained with overflow liquor treated in the laboratory with the same amount of TCA. These results have reinforced our confidence in the laboratory filtration method to be able to predict the performance of the plant security filtration.

Once the filtration response to TCA dosage was established as shown above during stable plant conditions, another campaign was initiated to monitor FCR of feed-to-filter slurry and to compare the results to the base case. Several feed-to-filter samples were analyzed daily using the method described above.

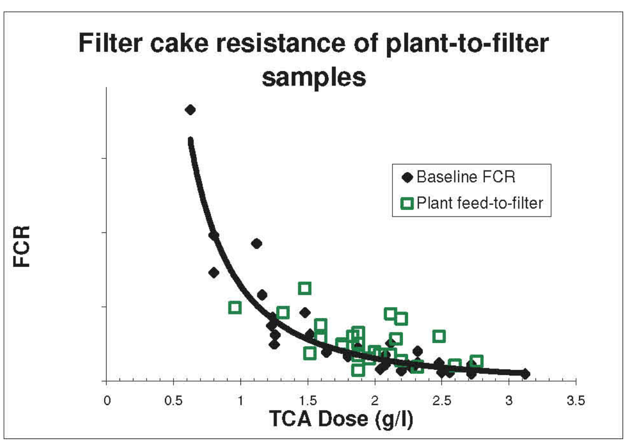

Figure 5: Daily filter cake resistance of feed-to-filter samples plotted against baseline curve.

Figure 5 shows the FCR values obtained over a period of about 5 weeks. These values are plotted together with the FCR baseline curve presented previously. It can be seen from these results that TCA dosage of feed-to-filters results varies between 1.0-2.7 g/L for the period and hence FCR values vary significantly.

A closer examination of Figure 5 results obtained with TCA dosages between 1.9-2.2 g/L was made and the values are reported against the active dosage of flocculent in use at the time of taking the samples. These results are displayed in Figure 6.

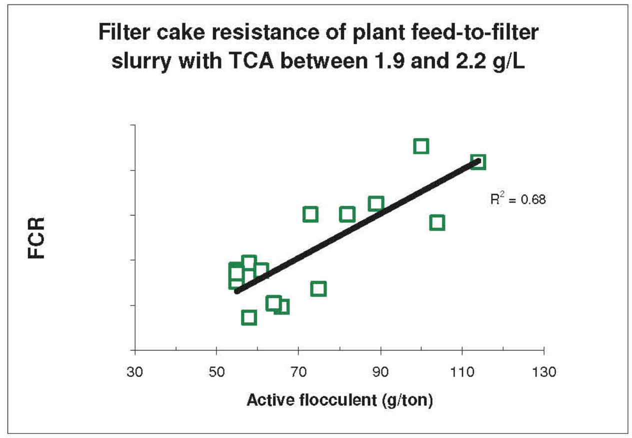

Figure 6: Filter cake resistance of feed-to-filter samples containing between 1.9 and 2.2 g/L TCA reported as function of total active flocculant.

Impact of flocculent and of total suspended solids on filter cake resistance

Results shown in Figure 6 indicate a relationship between the filter cake resistance and the total dosage of active flocculent used to settle the mud. Because the concentration range of TCA was chosen between 1.9 and 2.2 g/L, this parameter should not significantly influence the FCR. Results in Figure 6 suggest that FCR increases by at least a factor of 3 when the total flocculent increases from 55 to 115 g/ton. Besides TCA and flocculent, total suspended solids (TSS) may also play a role in increasing FCR.

To study the effect of TSS on FCR, another set of data is plotted in Figure 7 and shows simultaneously the dependence of FCR and TSS on the total active flocculent. These values were obtained over a period of several days by sampling HRD overflow and by adding 2.0 g/L of TCA prior to the filtration test. Determination of TSS was made separately using Millipore vacuum filtration on a different portion of the overflow sample. The flocculent dosage values were those prevailing in HRD, sixty minutes prior to sampling the overflow liquor.

Results from Figure 7 again show a strong dependence of FCR on the total flocculent as in Figure 6, but TSS values are not consistent with the general understanding. It is well known that TSS should be decreasing with an increase of flocculent but this clear relation is not present in Figure 7. Based on this evidence, the authors are suggesting that flocculent is not properly reacted with the mud particles because of a lack of mixing and that a fraction of the flocculent remains dispersed in the overflow liquor affecting FCR proportionally. It can be observed that even when TSS is very low but the dosage of flocculent reaches its highest value, the FCR is at its maximum value. Based on these results, we have good reasons to believe that the dosage of flocculent is a major factor impacting FCR. On the other hand, total suspended solids (TSS) at a concentration of less than 400 mg/L affect FCR to a much less extent if TCA dosage is greater than 2.0 g/L.

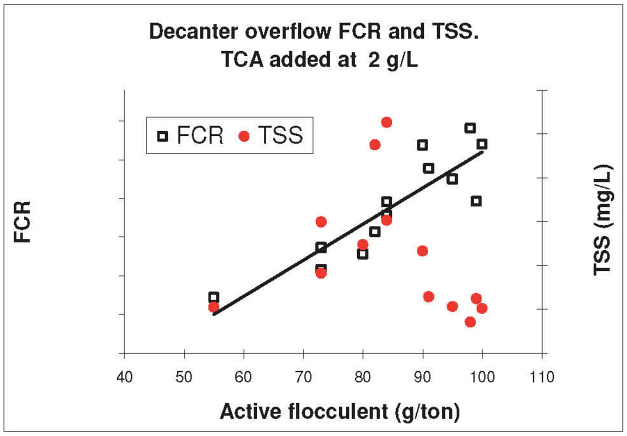

Figure 7: Decanter overflow's filter cake resistance and total suspended solids dependence on flocculant dosage.

It can be argued that it is not the total amount of active flocculent that shows an impact on FCR, but rather the excess flocculent carried over in the overflow. It is reasonable to believe that the more flocculent added, the greater the risk that some flocculent remains unabsorbed by the mud particles and end up free in the liquor. Even at fixed dosage, an overdose of flocculant can happen because slight changes in the operations such as bauxite quality, digester charge, mixing, feed rate, etc… can vary rapidly. Linking back to the first results obtained with pregnant liquor doped with known amount of flocculent (Figure 2), FCR values increase by a factor of 2 in the presence of 150 ppb of residual flocculent and by a factor of 3 with 300 ppb. When converting the g/ton dosage to active flocculent in solution, these residual amounts represent an un-reacted fraction of flocculent of only 3-6% of the total dosage.

Means to reduce the impact of residual flocculent

Two potential means to reduce the adverse effect of residual flocculent on filtration were investigated: ageing time and TCA dosage.

Pregnant liquor (filtrate) was collected in the plant and controlled amounts of flocculent were added to simulate overflow containing 300 ppb of residual flocculent. The doped samples were kept in an oven at 95°C and portions of 2.5 liters were filtered at different time using 2.0 g/L TCA as filteraid. Figure 8 displays the FCR obtained for the solutions doped separately with two different flocculents used by the plant. The experiment was carried out over a period of four hours. Within the precision of the method, FCR remains more or less constant over 200 minutes for flocculent B showing little if no degradation of this polymer as a function of time. On the other hand, the adverse effect of flocculent A on FCR is reduced by a factor of two after 255 minutes. However, even after 4 hours of holding time, the doped solution still displays a FCR which is approximately twice the FCR of the original (undoped) pregnant liquor. Because of the plant constraints, the total residence time of the overflow after the mud has been separated cannot be increased to more than 2 hours. Therefore, holding time by itself is unlikely to be sufficient to improve FCR if residual flocculent still exist in the overflow.

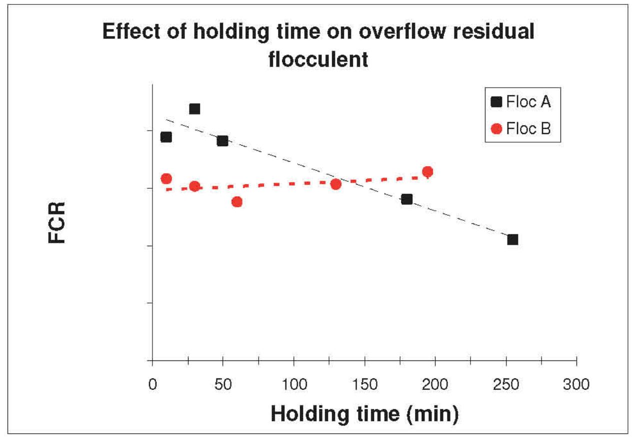

Figure 8: Effect of increasing solution ageing time on filter cake resistance.

One last series of experiments was carried out to investigate the effect of TCA dosage on the improvement of filtration rate. Again pregnant liquor was sampled in the plant and doped with 150 and 300 ppb of flocculent A. Filtration tests were carried out using varying amount of TCA as filteraid. The results are displayed in Figure 9 where it can be seen that TCA plays a major role in adsorbing residual flocculent and hence reducing FCR. For this specific case, it can be extrapolated that about 2.6 g/L of TCA filteraid would be required to almost neutralize the effect of any residual flocculent and then match the FCR of pregnant liquor.

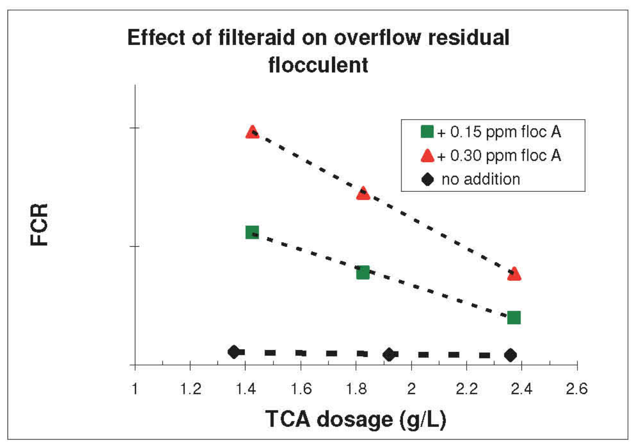

Figure 9: Effect of increasing TCA filteraid on filter cake resistance.

Unfortunately, this mean of improving FCR is not very attractive on both a cost and an operation point of view. Therefore we feel that other work is required to develop a more efficient method of controlling/avoiding the presence of residual flocculent in primary decanter overflow because it has one major negative impact on filtration rate, based on the understanding of the fundamentals examined in this paper.

Conclusions

The laboratory method using pressure filtration as described in this paper is capable of correlating the plant security filtration performance to some quantitative data such as filter cake resistance (FCR), residual flocculent at sub ppm level and to TCA filteraid dosage.

Decanter overflow filtration rate is primarily driven by the amount of TCA filteraid added to decanter supernatant. With the specific liquor and TCA used in this work, the optimal dosage of filteraid is found around 2 g/L.

Residual flocculent in decanter overflow is one of the major contributors to increased filter cake resistance. As little as 3-6% of total active dosage of flocculent can increase FCR by 2 or 3 times if it ends up in the overflow.

Total suspended solids (TSS) in overflow liquor have little or no significant detrimental effect on FCR in the range of 50-400 mg/L provided that sufficient TCA filteraid is added.

Security filtration performance is directly linked to the control of the flocculent addition. Excessive dosing of flocculent in an attempt to decrease TSS is not indicated since this will result in creating unwanted residual flocculent in the overflow, which will deteriorate performance and irremediably blind the filters.

Increasing overflow residence time will not improve FCR within reasonable process limits. Results have shown slight improvement with residence time of more than 4 hours, which is not accessible for actual modern plant configuration.

The dosage of filteraid (TCA) appears to be critical to FCR as it may adsorb some of the residual flocculent. However, the required TCA dosage to neutralize completely the effect of the residual flocculent is high (>2.4 g/L) and, assuming lime availability, may not be cost effective.

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