INCREASE IN THE STABILITY OF GRAVIMETRIC CLASSIFICATION SYSTEM OF PRECIPITATION AT HYDRO ALUNORTE

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Abstract

After the installation of coarse seed filtration at lines 1, 2 and 3 at Hydro Alunorte, the precipitation productivity was increased, however the effect of higher concentration of solids in the circuit generated some operational problems. Small interruptions in the feed of the filters were responsible for significant production losses resulting from the overload of solids on the classification system caused by the addition of liquor to the chain. In order to improve circuit stability, the underflow streams from the secondary classifier were divided between the first cementator, where the control of supersaturation occurs, and second cementator, where it is fed excess seed. When the seed filter stops, its feed reduces and the flow to second cementator increases, eliminating the flow variation in the chain. Furthermore, statistical modeling was performed to regulate the concentration of solids in the feed of the thickeners, reducing the overload to it.

Introduction

Alunorte started operation in July 1995. The plant had two production lines and a nominal production capacity of 1.1 Mtpa. This production rate was reached in 1997 and exceeded in the following years. A number of process improvements were implemented so that the production capacity could be increased to 1.6 Mtpa in 2000. In eight years only from 2000 to 2008 the plant production capacity increased three times. Work on Expansion 1 started in 2000 and in the first quarter 2003 the third process line was commissioned. The plant design production capacity was increased to 2.325 Mtpa. This production rate was reached and even exceeded during the first year after the expansion. In 2006 process lines 4 and 5 were commissioned as part of Expansion 2. The production capacity increased further to close to
4.4 Mtpa as a result of the installation of coarse seed filtration on the lines 1-3. Finally, in the fourth quarter of 2008, lines 6 and 7 started operation as part of Expansion 3. The plant has now a nominal production capacity of close to 6.3 Mtpa.

The coarse seed filtration area aims to increase the productivity of the precipitation circuit by reducing the spent liquor re-circulation through the precipitation chain allowing higher residence time. This increase in residence time raises the productivity by approximately 3,8% and consequently the plant production by 90,000 tpy.

Precipitation circuit

The hydrate precipitation process of Hydro Alunorte refinery consists of the following steps: pregnant liquor cooling, precipitating tanks, filtration and classification of seed.

The pregnant liquor cooling consists of two steps: the vacuum cooling and plate exchangers using water as cooling liquid, in order to achieve the supersaturation necessary for the precipitation process occur.

The precipitation process consists of the following steps: agglomeration, cementation and precipitators of dense bed and end chain. In the agglomeration stage, a fraction of cold pregnant liquor is mixed with fine seed directly from the tertiary thickeners. In cementation, the agglomerated slurry is mixed with the rest of the pregnant liquor and fine seed, plus the coarse seed from the secondary classifier that was previously filtered in disc filters. The dense bed and end of chain precipitators basically help to increase the productivity of the precipitation, by increasing the hydrate residence time through the cooling obtained by indirect contact with water that passes through the coolers inside the tanks.

The classification process is achieved by gravimetric separation and consists of three stages: primary classification, where the product is separated by underflow, secondary classification, where the coarse seed is separated and tertiary classification, where the fine seed is separated from the spent liquor that will again be used in digestion after concentration, heating and adjustment of the caustic concentration.

Figure 1 illustrates the precipitation and classification circuit at Hydro Alunorte. Both precipitation Lines 1 and 2, consist of 14 tanks each, with the first three being agglomerators, where a portion of the pregnant liquor and fine seed are added, the following three tanks are the cementators, where the agglomerated pulp is mixed with remaining fine seed and coarse seed, and the other has the function to promote crystal growth and increasing yield. The classification section follows the principle of gravity separation, as is divided in primary, secondary and tertiary classifiers (or “thickeners”).

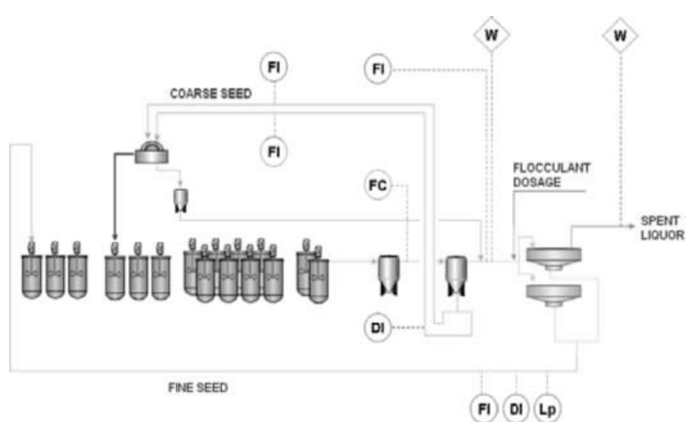

Figure 1. Overview of Hydro Alunorte Precipitation Process.

Improvement description

With the start-up of the coarse seed filtration area, an increase in the precipitation productivity was achieved, but this was accompanied by more difficulties to respond to disturbances in the process. Due to the limitations in gravimetric classifiers characteristics, poor underflow density control it was observed during the third quarter until the end of campaigning, causing interruptions in the operation of the coarse seed filters. With that, the return of the volume of liquor to the process while the filter returns to operation causes a large overload in secondary classifiers and thickeners, generating successive reductions of flow in the Plant. Thus, it became necessary to identify mechanisms to react for the oscillation process in order to obtain less disturbances in the filters.

As shown in Figure 2, the secondary classifier underflow was totally interrupted during coarse seed filters shutdowns.

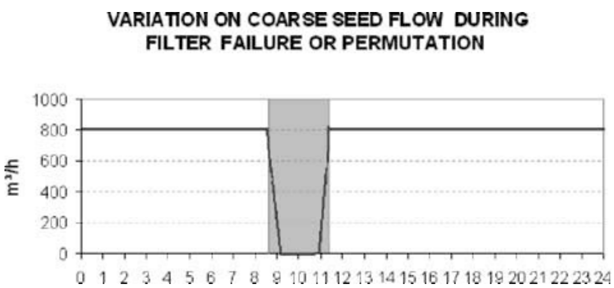

Figure2. Theoretical profile of secondary classifier underflow.

Therefore, as a first step, it was tested the operation with the coarse seed flow divided between:
a) first cementator, after passing for the coarse seed filtration,
b) second cementator, without passing by the coarse seed filtration.

Based on simulations, it was observed that maintaining a total coarse seed flow rate slightly higher than usual, the negative effect of the addition of liquor to precipitation by reducing the residence time was not only compensated but also there would be a potential increase productivity of the precipitation, as shown in Figure 3..

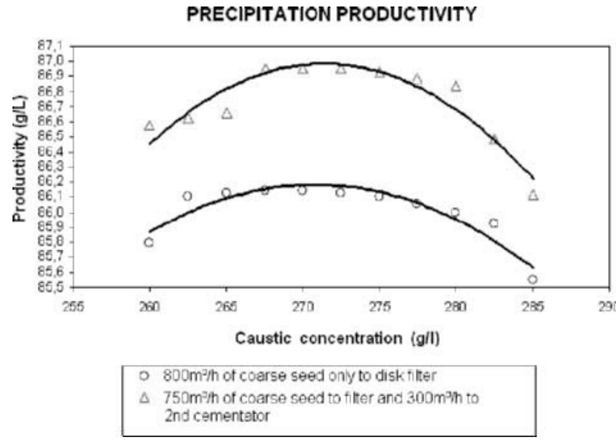

Figure 3. Simulation showing the increase in precipitation productivity with the increase in coarse seed charge.

Figures 4 and 5 show the behavior of the secondary classifier underflow when operating with coarse seed flows divided. It is possible to observe that at no time the tank runs out of underflow, which allows for more stable operation and a long-time campaign.

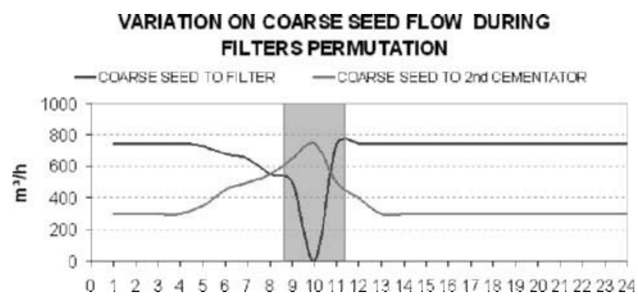

Figure 4. Theoretical profile of secondary classifier after the change in the process concept.

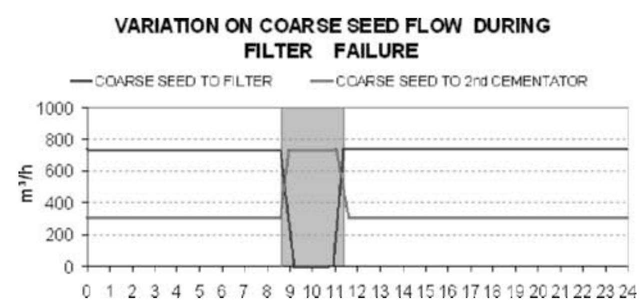

Figure 5. Theoretical profile of secondary classifier after the change in the process concept.

After eliminating the disturbances caused by the stopping of the coarse seed filters as well as the accumulation of solids in the secondary classifiers, it becomes necessary to act on change the operations around the tertiary classifiers. To do it, we used the 6 sigma methodology in order to define the scope of the studied process, measure the reliability and analyse the interaction between the input and output variables, and to propose alternatives to achieve better results, using statistical tools.

Initially, in order to evaluate the influence of other parameters that can not be changed in the plant tests for causing impact on the process control, we performed a DOE (Design of experiment). The results obtained are shown in Figure 6 where it is possible see, at the studied conditions, major influences of the concentration of solids in the pulp to be settled, the median particle size and the flocculant dosage.

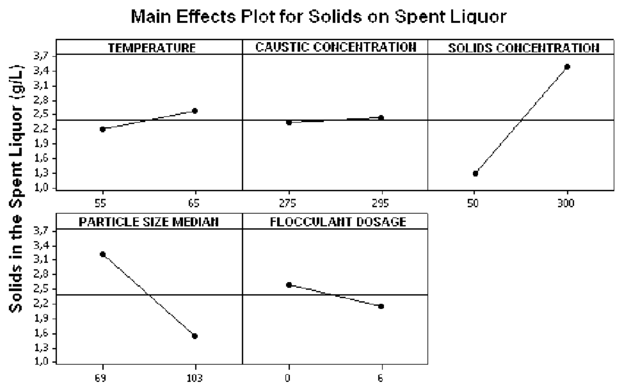

Figure -6. Main effects graph for solids in the spent liquor

Additionally, an analysis of the input variables impacting the concentration of solids in the thickener overflow was performed, in order to identify the mechanism in which these parameters interact to control the response variable,For this, a multivariate regression was performed, using Plant data colected in each two hours during one week, and a summary of the results are presented in Figures 7 and 8.

Regression Analysis: SOLIDS CONCENTRATION AT SPENT LIOUOR (W_{SL})

The regression equation is
W_{SL} = 12,0 + 10,7 dsec + 0,00385 F4 + 0,0248 F5 + 2,90 dfs - 0,448 T - 0,155 Mfs

Predictor	Coef	SE Coef	T	P
Constant	11,958	7,555	1,58	0,145
dsec	10,726	2,130	5,03	0,001
F4	0,0038479	0,0005896	6,53	0,000
F5	0,024771	0,004701	5,27	0,000
dfs	2,895	1,239	2,34	0,042
T	-0,4478	0,1700	-2,63	0,025
Mfs	-0,15478	0,06735	-2,30	0,044

S = 0,239800   R-Sq = 96,6%   R-Sq(adj) = 93,9%

Where:

dsec - density at secondary classifier overflow
F4 - Flow at the thickener feed
F5 - Flow at the thickener underflow
dfs - Fine seed density
T - Liquor temperature
Mfs - Fine seed particle size median

Figure 7. Output from Minitab showing the regression for solids concentration at spent liquor.

The results show that the thickener overflow solids is strongly correlated to the feeding flow in the thickener, the thickener underflow rate, fine seed (U/F) density, temperature, median particle size of the fine seed and the density of the classifier secondary overflow. The Figure 8 shows the regression obtained for the density of secondary classifier overflow, wich was a function of the coarse seed flow rates to the first and to the second cementator, the densities of these flows as well as the feed flow rate of the secondary classifier. The analysis was made using data collected directly from the process and treated in Minitab software, and achieved excellent correlation coefficients for both.

Regression Analysis: DENSITY AT SECONDARY CLASSIFIER OVERFLOW (dsec)

The regression equation is
dsec = - 1,72 - 0,000366 F1 - 0,000949 F2 + 2,16 Dcs + 0,000154 F3

Predictor	Coef	SE Coef	T	P
Constant	-1,7236	0,5963	-2,89	0,013
F1	-0,00036562	0,00007675	-4,76	0,000
F2	-0,00094865	0,00007801	-12,16	0,000
Dcs	2,1580	0,3723	5,80	0,000
F3	0,00015395	0,00001316	11,70	0,000

S = 0,0113323   R-Sq = 97,3%   R-Sq(adj) = 96,5%

Where:
F1 - Coarse seed flow to disk filter
F2 - Coarse seed flow to second cementator
Dcs - Coarse seed density
F3 - Flow at the feed of the secondary classifier

Figure 8. Output from Minitab showing the regression for density at secondary classifier overflow.

Based on regression, it was possible to know the behavior of the process under test conditions, and consequently the operation control limits for these parameters in order to achieve the goals of operation. Thus, it is possible to set the operating range for the density of the overflow secondary classifier to not exceed the limit of solids concentration in the spent liquor, which is shown in Figure 9. It is essential that the mass of solids being fed into the thickener does not exceed these limits, because this will cause an overload of solids in the tertiary classifiers , as well as a loss of hydrate production as a result of high solids levels in the spent liquor. Furthermore, it also means that the fine seed will be contaminated with coarser particles, which will affect the agglomeration process and, thus, the quality control of the final product.

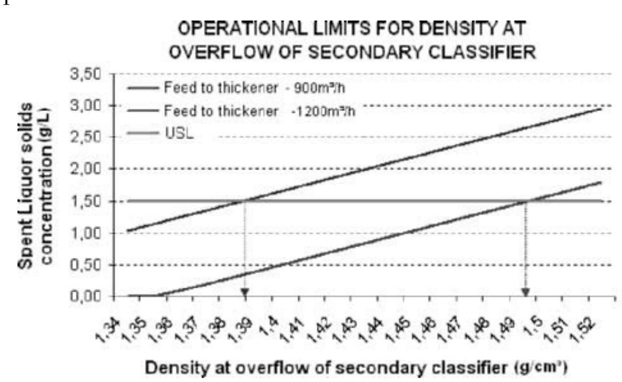

Figure 9. Operational limits based on the regression for solids concentration at spent liquor.

Known the importance of the concentration of solids in secondary classifier overflow and based on the linear regression, it is possible to control this parameter through coarse seed flow to the second cementator, since the other parameters of the regression must follow the precipitation control goals and should not be changed. Figure 10 shows how this control can be achieved, and Figure 11 illustrates the process according to the new design.

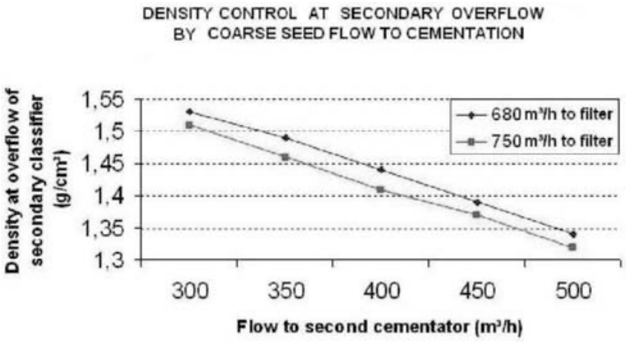

Figure 10. Operational limits based on the regression for density at secondary classifier overflow.

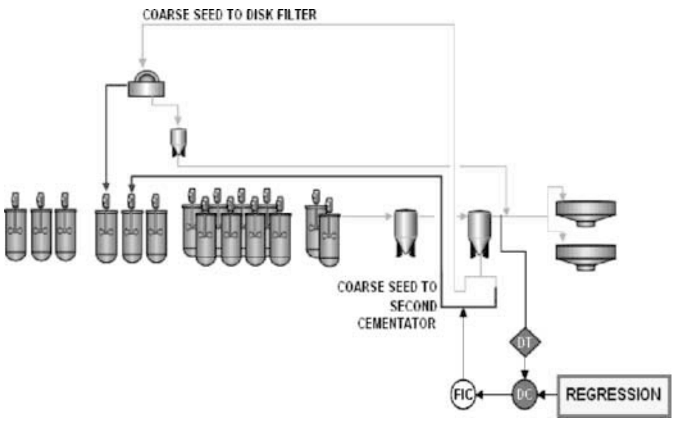

Figure 11. New process concept.

The result of this new operation design of the classification can be seen in Figure 12, where the loss of solids in spent liquor was substantially reduced. Moreover, the negative effect of these solids deposition on the tubes of heat exchangers of evaporation, vacuum cooling and heating spent liquor at digestion, can be considerably reduced.

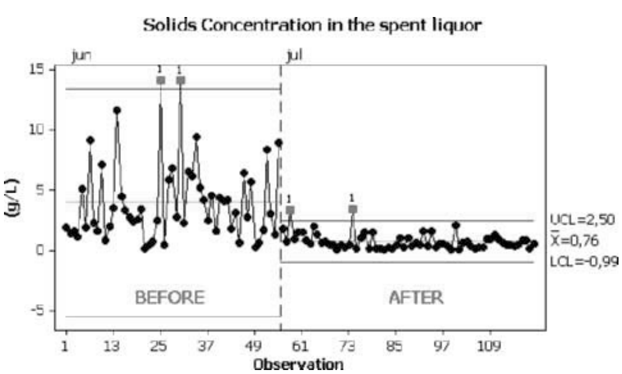

Figure 12. Profile of solids concentration in spent liquor before and after the improvements.

Conclusion

Based on the foregoing, it is clear that production losses caused by overload of solids on the secondary and tertiary classifiers derived from coarse seed filters stops was eliminated. Currently, both specific disturbances as well as filters relays routine can be performed without the need to interrupt the secondary classifier underflow, which makes the operation more stable, secure, and still allow a longer campaign tank. The negative effect of the increased volume of liquor in the precipitation chain due to the addition of the unfiltered seed on the second cementator was fully offseted by the use of total coarse seed, that was fed into the tertiary classifier, causing a overload on it. Based on the simulations, there were no productivity losses on precipitation productivity.

With the change in the design of the distribution of coarse seed charge was possible to create a mechanism to prevent overload on thickeners. Using multivariate regressions, it was observed that the solids concentration in the overflow from secondary classifier was function of the mass of solids transferred by the tank underflow and the mass entering, identifying that the density in the feed of thickeners could be controlled by varying the coarse seed charge to the second cementator.

By the use of multivariate statistics, there was obtained also a regression for the behavior of solids concentration in the spent liquor which allows to set operating limits in order to minimize loss of productivity of the circuit. Laboratory experiments have also allowed it to be identified the potential for solids control in spent liquor controlling the density overflow in the secondary classifier.

As a result, we developed a new operating concept based on statistical models obtained and the distribution of coarse seed charge, allowing an increase in process stability without impairing the productivity of the precipitation.

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