REFINERY CARBONATATION: A PRACTICAL APPROACH

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Abstract

Tongaat Hulett Refinery employs carbonatation as its clarification and decolourisation step in processing very high pol sugar. During the past few years attempts have been made to optimise the carbonatation process with regard to impurity removal and filterability. This paper covers in detail the equipment employed to facilitate carbonatation, together with the control philosophy surrounding the gassing of milk of lime, lime dosing rates into melt liquor and filtration throughputs. Practical insights and challenges concerning the management of the saturator equipment, auto-filter station and operations are discussed.

Keywords: carbonatation, filtration, filterability

Introduction

In refining, the two key objectives of carbonatation are (i) to clarify raw sugar melt liquor and (ii) to produce calcium carbonate slurry that has good filterability characteristics (Murray and Runggas, 1975). Carbonatation has been shown to remove turbidity, colour, starches, silica and sulphates (Moodley, 2001) via the use of milk of lime gassed down to the calcium carbonate precipitate. Colour removal using the carbonatation process can vary according to the incoming melt colour, lime dosage and final exit pH prior to precipitate separation. The mechanism for colour removal is via adsorption and absorption into the crystal (Bennett, 1974). In comparison to phosphatation and sulphitation, carbonatation has proven to be a very cost effective and robust process with flexible colour removal achieved by varying the lime dosage on melts solids. Hulref employs carbonatation followed by resin ion exchange as the gross decolourisation step. Over the last decade, the carbonatation station has been subjected to many continuous improvement activities including technical, operations and equipment perspectives. These positive results can be seen in Figure 1. The continuous improvement activities are reviewed in this paper.

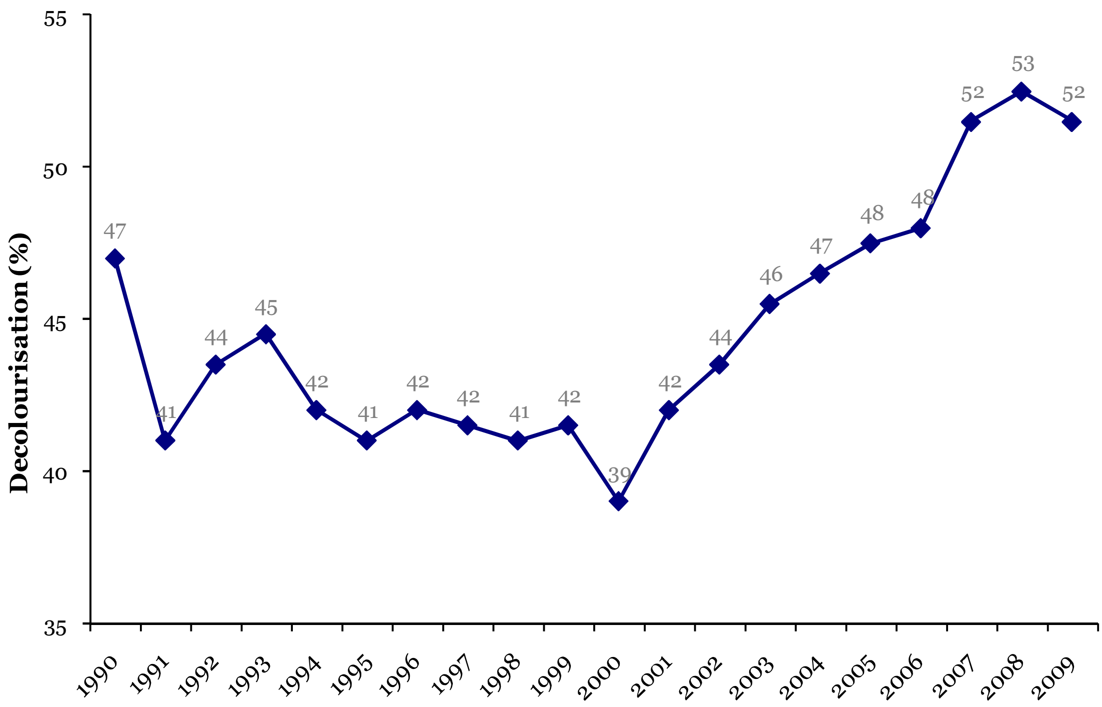

Figure 1. Colour removal across Hulref carbonatation station.

Hulref carbonatation process description

Slaked lime is introduced into melt liquor at dosages between 0.50 to 0.65% on melt brix using a flow ratio variable speed lime wheel (Figure 2). The gassing of the lime with flue gas from the boiler house is achieved in a two-stage process. Approximately 80% of the gassing is complete in the first stage compromising of three A-saturators operating in parallel, achieving an exit pH of 9.0 to 9.5. The remaining milk of lime is gassed down in a B-saturator to a pH of 8.3 for optimum filterability and ash removal. The temperature of the carbonated liquor is then raised to 83°C in a C-saturator using exhaust steam of absolute pressure of 220 kPa. The C-saturator serves the dual function of a heat exchanger and a buffer supply tank for the auto filter station.

The calcium carbonate slurry is separated from the carbonated liquor using thirteen rotary auto-filters operating in parallel. The residual lime carry-over to the resin plant is typically less than 10 ppm on brix. Once the auto-filter throughput is reduced to less than 4 m^3/h, the cake build-up is sluiced using hot water, then desweetened and dried using one of three plate and frame filters. The final mud has a typical pol of 0.20% and moisture in the region of 40%. The carbonatation station has a design capacity of 120 tons/h of melt solids. Typical decolourisation at Hulref is 50% based on a 0.60% lime on melt brix.

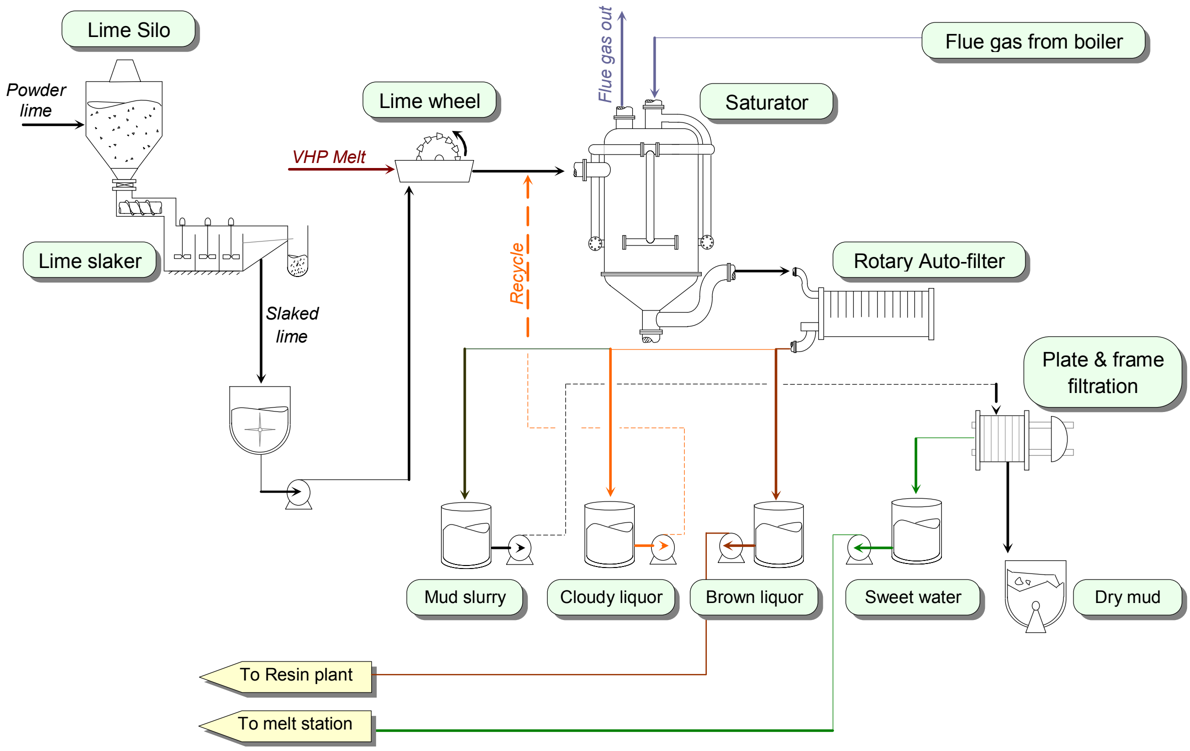

Figure 2. Process flow diagram of Hulref carbonatation station.

Saturator configuration and gas distributions system

The A and B-saturators have sparge pipe gas distribution systems (Figure 3), with each saturator accommodating up to 35 m^3/h of flue gas without adverse effects on entrainment. The disengagement space between the liquor surface and gas exit is 1.5 m. Each saturator has an effective liquor volume of 31 m^3, giving a typical residence time of 80 minutes in the A and B-saturators at melt liquor solids of 145 ton/h.

In 2001, Hulref converted its saturator configuration from 2-2-1 to 3-1-1 as studies by the Technology and Engineering department together with Hulref operations staff revealed that the proportion of residence time split between the A and B-saturators was not ideal for gassing efficiency and producing a $CaCO_3$ slurry with good filterability characteristics (Moodley *et al.*, 2002). In this new arrangement, approximately 80% of the gassing is achieved in the A-saturator, whilst the final liquor conditioning required to achieve the optimum slurry filterability characteristic is completed in the B-saturator. The C-saturator (C-sat) serves only to condition the liquor temperature and act as a buffer supply tank for the auto filter station. The change in saturator configuration did improve colour removal, as indicated in Figure 1. The chief benefit from this modification is the stability of the pH control arising from the increased residence time in the A-saturators.

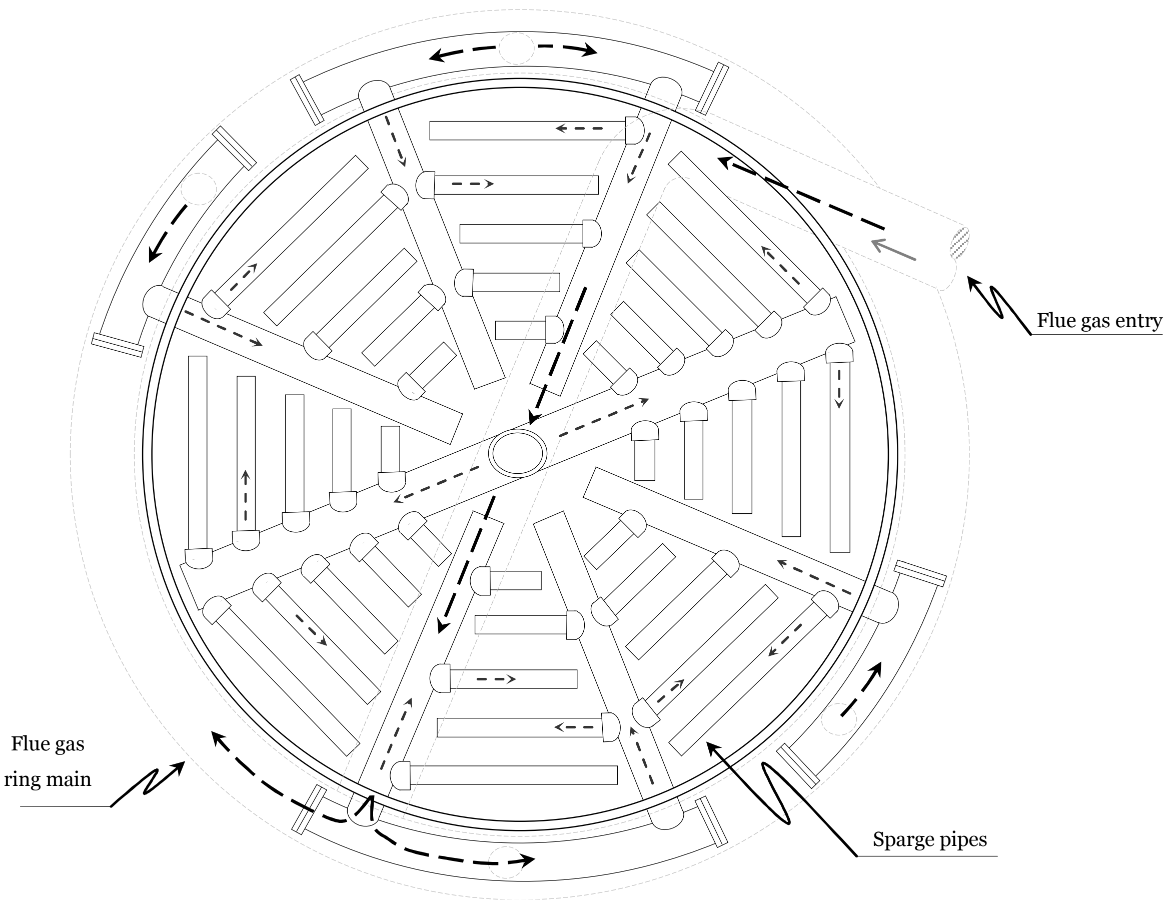

Figure 3. Saturator internal arrangement of sparge pipes.

Hulref's saturator pH control system consists of a primary control loop, which governs the output pH from each saturator, and a secondary control loop, which is the gas input required to maintain the pH. In this cascade control philosophy, the deviation of the set pH from the actual pH generates the set point for the gas flow rate into the saturator. The gas control philosophy is represented in Figure 4. This control system has proved to be very effective, as revealed in Figures 5 and 6 showing the 24-hour pH control trends of A and B-saturators.

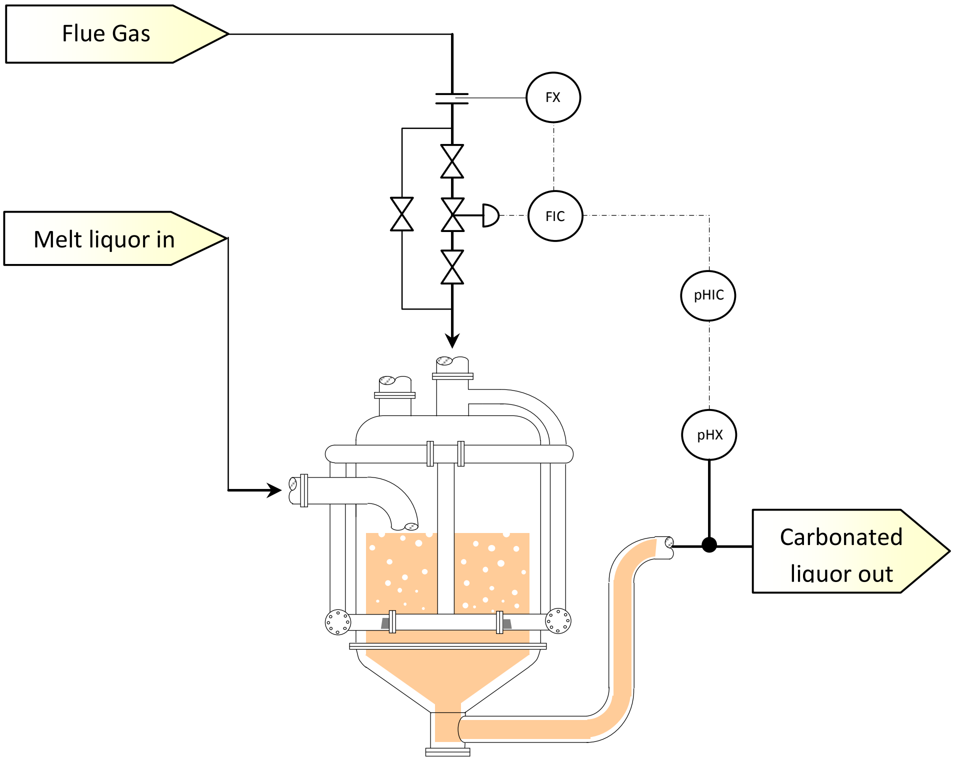

Figure 4. Control of saturator pH.

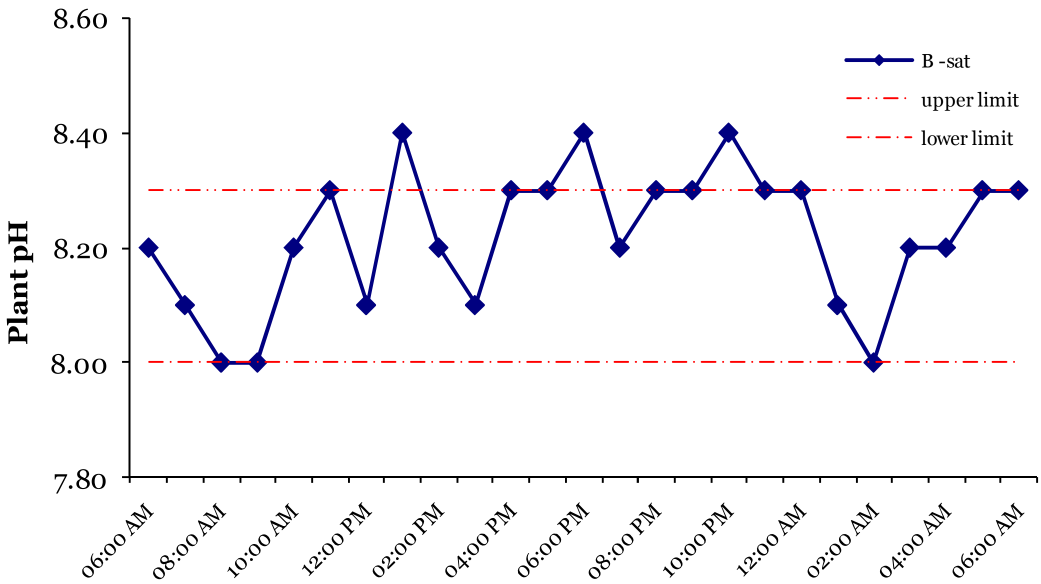

Figure 5. B-saturator plant pH trend under steady state conditions.

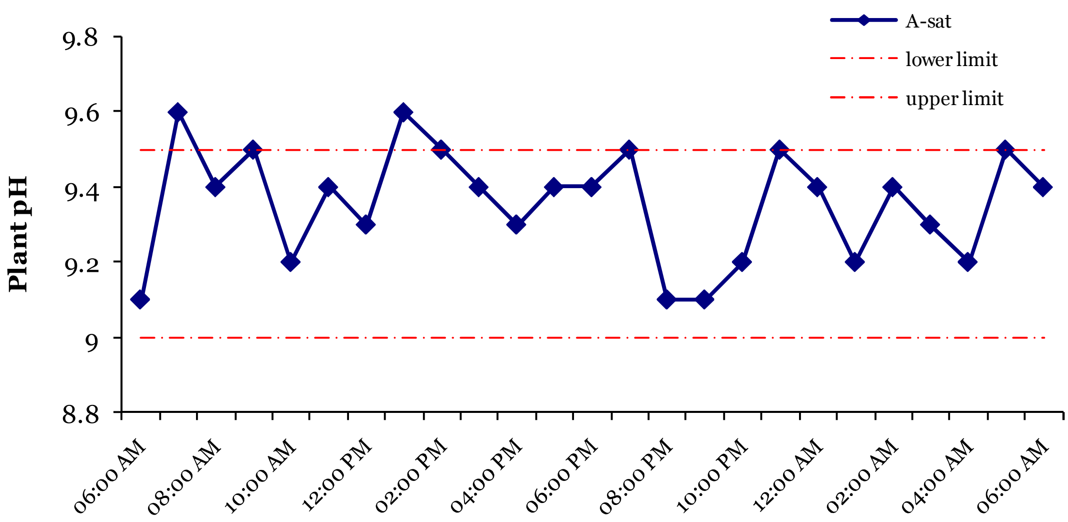

Figure 6. A2-saturator plant pH trend under steady state conditions.

Lime and liquor mixing system

The slaked lime is introduced into melt liquor using a flow ratio variable speed lime wheel and turbulent mixing is achieved through a static in-line mixer consisting of several notched baffles in series. The overall residence time across this system is in the region of 10 seconds, based on a melt volumetric flow of 120 m^3/h. The static in-line mixer (Figure 7), with its low residence time and effective mixing, is a huge step forward when compared to the previous system comprised of lime and liquor mixing via a stirred tank. Studies conducted by Bervoets and Cox (1992) on ash gain across carbonatation revealed that lactic acid formation across the lime liquor-mixing tank was contributing significantly to the dissolution of calcium carbonate precipitate, thus elevating the final ash in the carbonated liquor. The major factors contributing to lactic acid formation where identified as:

- Retention time in the lime liquor-mixing tank.
- Initial reducing sugar levels in melt liquor.
- High temperatures.
- Lime dosage concentration on melts solids.

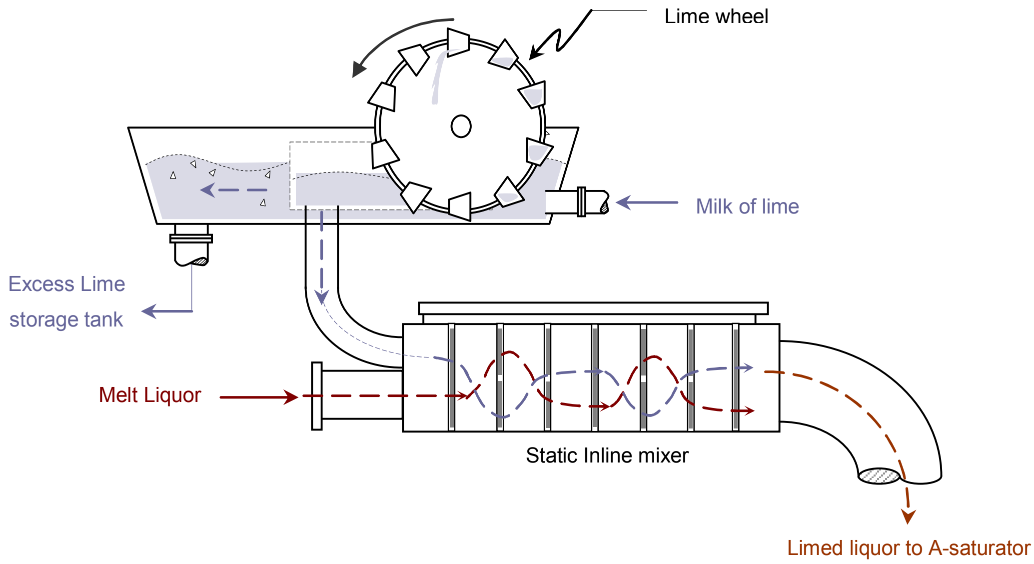

Figure 7. Lime wheel with static in-line mixer.

Filtration equipment and throughput control philosophy

To remove the carbonate slurry from the liquor, Hulref operates 13 Smiths rotary auto filters in parallel, with one always off-line for regeneration (Figure 8). Each filter consists of 56 leaves enveloped with a 100% polypropylene cloth. The resulting filtrate is collected in a brown liquor tank for further decolourisation in the resin plant. A cascade control system, which uses the level outputs from the brown liquor tank and C-saturator (auto-filter supply tank), is used to determine a common flow set point of liquor through each filter. Throughputs in the station range from 100 to 135 m^3/h, enabling the refinery to melt up to 95 ton/h of VHP. At the start of each filter cycle, the initial liquor is turbid and is recycled to the C-saturator for re-filtering.

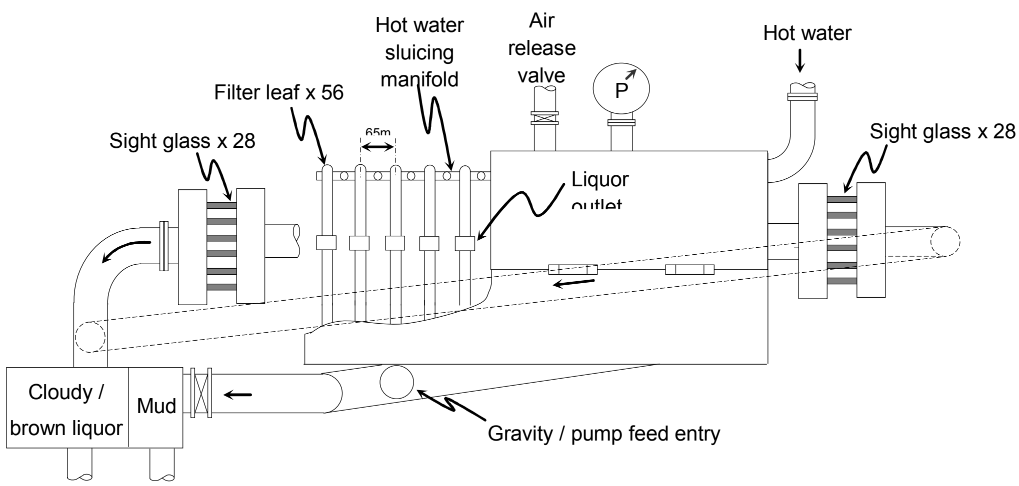

Figure 8. Cross-section of auto-filter.

To improve the quality of the liquor, during July 2002 the refinery process staff changed the type of filter cloths from PY7 to PY26. This change in cloth type resulted in an increased throughput, but at the expense of brown liquor clarity. Unpublished work by the refinery during 2005 revealed that PY26, although having a tighter weave, produced three times more suspended matter when compared with the PY7 under standard laboratory filtration tests (Table 1). Magnified images of both cloths revealed that the fibres of PY7 are much smaller and closely woven (Figures 9 and 10), thus explaining the marked reduction in liquor clarity when using the PY26. The refinery has since standardised on PY7 as the preferred choice for filtration.

Table 1. Comparison of the specifications of PY7 and PY26 filter cloths.

Specification	PY7	PY26
Material	100% polypropylene	100% polypropylene
Weave	2/1 Twill	2/2 Twill
Weight (g/m^2)	220-320	400-460
Thickness (mm)	0.52	0.99
Air flow ($mL/cm^2/sec$ @100 Pa)	3.0-6.0	1.5-3.0

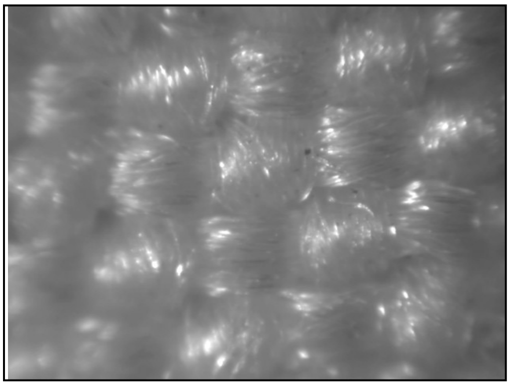

Figure 9. PY7 filter cloth.

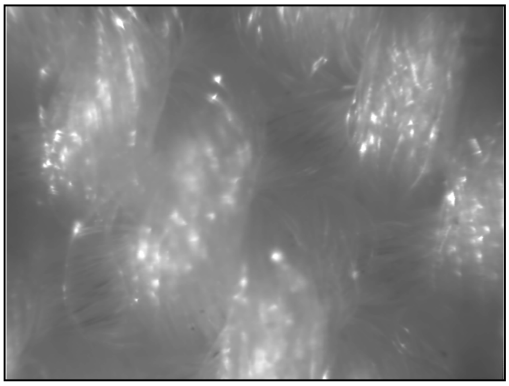

Figure 10. PY26 filter cloth.

Filter station operations

The two key deliverables of the filter station are to maintain suitable throughputs while keeping the lime carry-over as low as possible. The most challenging aspect of the filter station management is the filter wash cycles (*in situ* regeneration frequency) and cloth replacement frequency, which entails stripping down and replacing the cloth envelopes per leaf. This operation generally takes eight hours, is labour intensive and has a significant bearing on the cost of operations.

The *in situ* regeneration of the filter will proceed when the throughput in the filter has been significantly reduced (<4 m^3/h). The liquor content in the filter is drained to a recycle tank while the mud is sluiced off the filter leaves using hot water. The liquor is collected, pressed and desweetened in plate and frame filters. The filter is then gravity fed with liquor from the C-saturator, and the initial liquor, which is turbid, is recycled to either the C or A-saturator. The clarity of this liquor is visually monitored through catch samples that are then subjected to a vacuum filtration test using 8 μm filter paper, and put forward once the quality has improved. The time taken to regenerate and reinstate a filter into service is known as the wash cycle (typical Hulref wash time = 30-40 minutes).

Hulref filter station challenges

One of the key challenges faced by Hulref was reduced filter station performance. A key measurement used by the refinery to determine the performance of the filter station is the downtime associated when the auto-filter supply tank (C-sat) is full, resulting in a complete raw sugar melt stop. Losses associated with this reduced performance for 2008 amounted to 8000 tons of lost production. Investigations revealed that the filters were in poor condition, from both a scaling and mechanical point of view. A filter refurbishment plan completed in July 2009 saw a 20% increase in throughputs; however, the refinery was still plagued with downtime. The area that remained scientifically unexplored was the operation of the auto-filter station by process staff. The key functions required by operations staff to maintain throughputs are:

- Rate of filter regeneration – expressed and measured as 'filter washes per hour'.
- Cloth replacement – measured as 'cloth changes/week'.

To further understand the impacts of the abovementioned on quality and throughputs, a small project team was initiated to collect technical information detailing the station's throughputs and quality against filter cycle times. The purpose of the information gathering was to generate a simple mathematical model to establish the following:

- *In situ* regeneration frequency producing optimum throughputs.
- Filter cloth replacement frequency that produces optimum throughput and quality.
- Process control targets for standardisation of regeneration rates for process operators.

The station was modelled using throughput and quality data from several filters that were tracked per cycle over a period of 200 cycles. The throughput data was obtained using a flow meter installed on the liquor inlet line to the filter. Insoluble matter measurements from composite samples of the liquor over the cycle time were used as a quality measurement. The results from cycles 138 to 141 are shown in Figure 11.

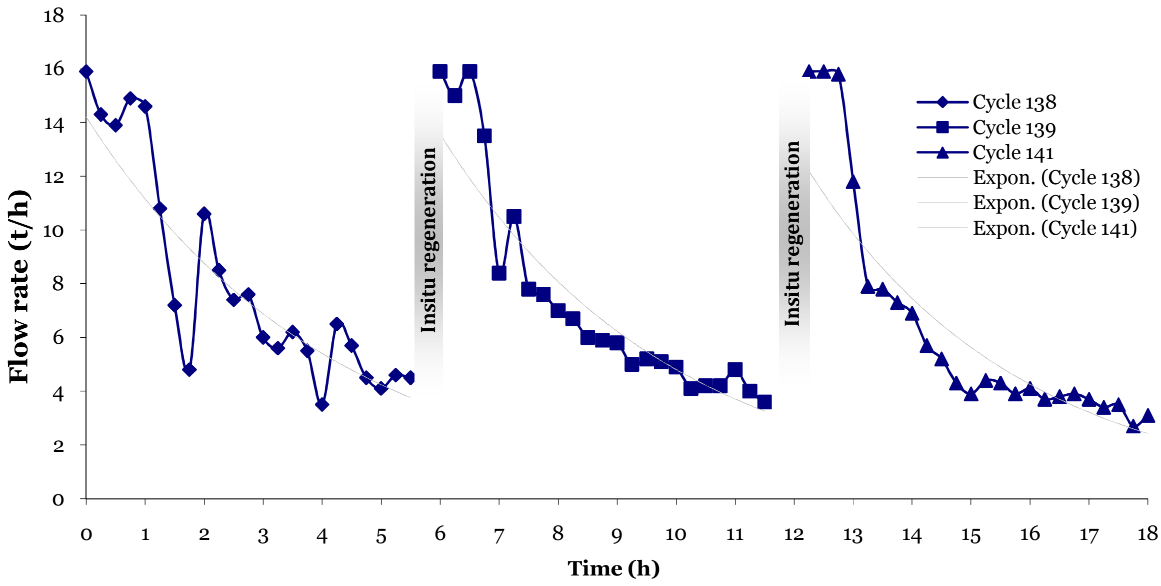

Figure 11. Typical trend throughputs over filter cycles.

Using the information captured over 200 auto-filter cycles, the station was modelled in terms of total station throughputs and quality. The results of the model reveal that two auto-filter regenerations per hour produce optimum results in terms of throughputs. A frequency above this results in too much filter dead time (time taken to regenerate filter exceeding operating time), which has the effect of depressing the station's overall throughput. This effect can be seen in Figure 12, where regenerations exceeding 48 per day results in throughputs below the optimum.

The plot of average cycle throughputs and quality (Figure 13) reveal that the filter cloths must be overhauled before 150 cycles to ensure that lime carry-over remains less than 10 ppm, thus minimising resin fouling. It is interesting to note that the quality of the brown liquor starts to decrease from 12 ppm at the start of the cycle, reaches a minimum around 100 cycles, and then deteriorates thereafter.

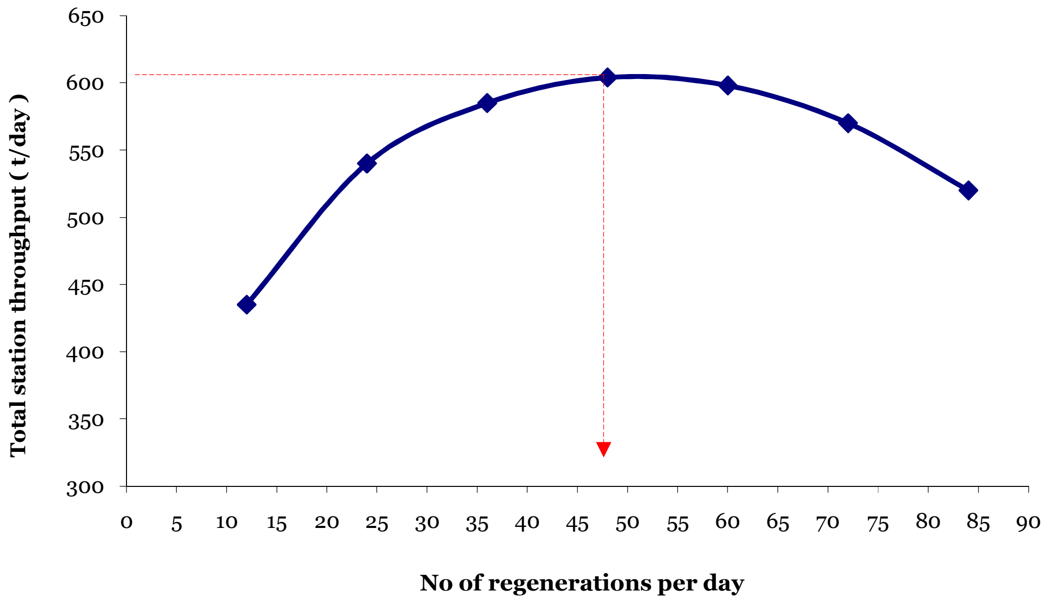

Figure 12. Regeneration frequency versus total filter station throughput.

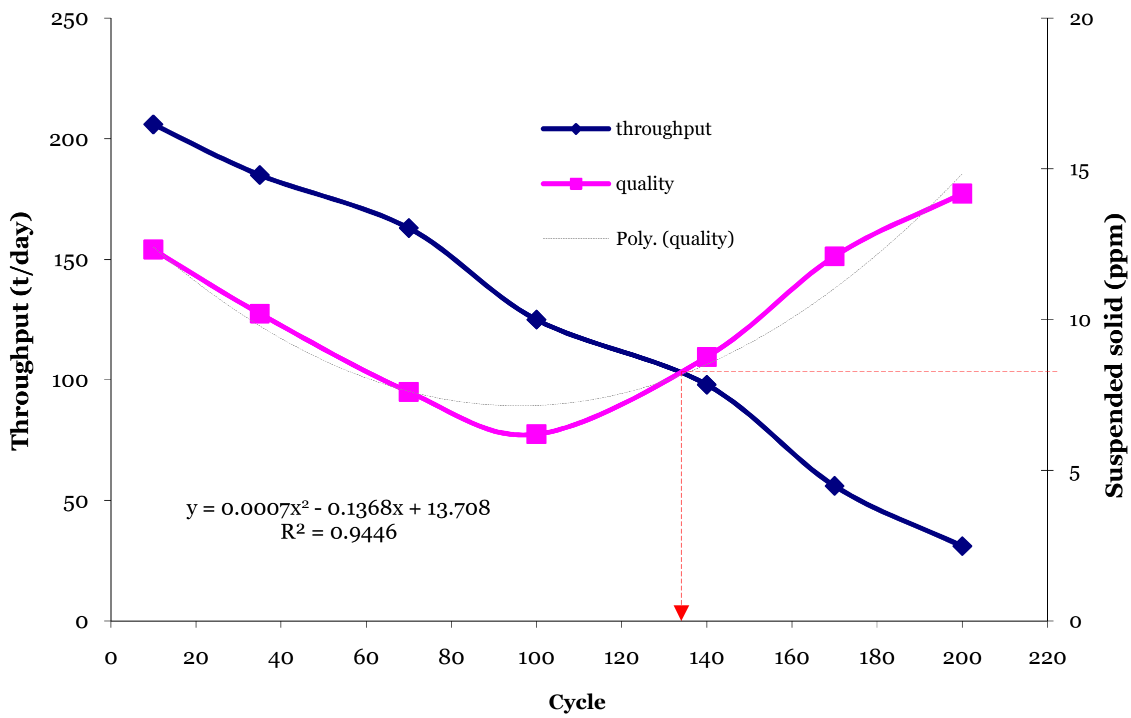

Figure 13. Throughput of auto filter versus suspended solids in brown liquor quality.

The information acquired from the modelling of the filter station provides good insight into the desired regeneration and filter cloth overhauling frequencies. This key information has since been standardised across the operations staff and entrenched as part of the station's Key Performance Areas (Figure 14). It has also provided useful information when troubleshooting during periods of poor filtration.

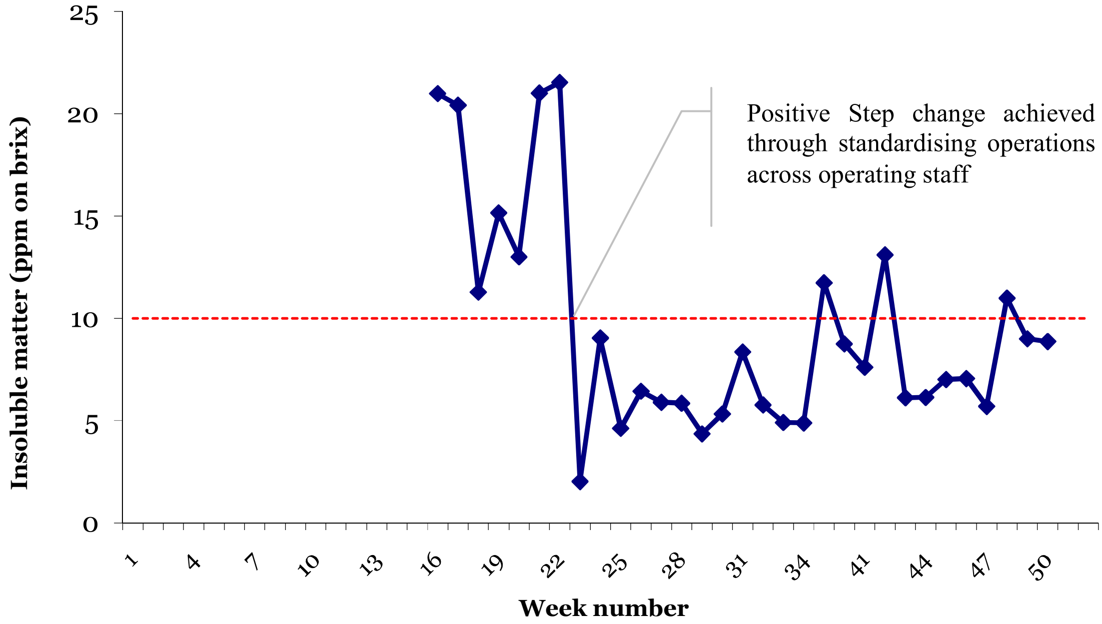

Figure 14. Weekly trends of insoluble matter in brown liquor.

Saturator maintenance challenges

Another challenge that faced Hulref was lime fouling of the saturator sparge pipes, which has a destabilising effect on the pH control. The frequency measured for sparge pipe fouling in 2009 was measured as 89 days (three months). At this point, the saturator is taken offline, and the sparge pipes are stripped, cleaned and installed over a 3-day period during which the refinery is constrained to a production rate of 80-85 ton/h. The techniques used to better manage this rate of fouling included the following:

- Introduction of exhaust steam through the sparge pipes to dislodge scale build-up.
- Introduction of grit blasting to remove lime scale.

High pressure cleaning, grit blasting and mechanical wire brushing of fouled sparge pipes were evaluated as cleaning techniques during the 2008/09 season (Figure 15). It was found that the average operating time of each saturator increased from 89 to 144 days when grit blasting was employed.

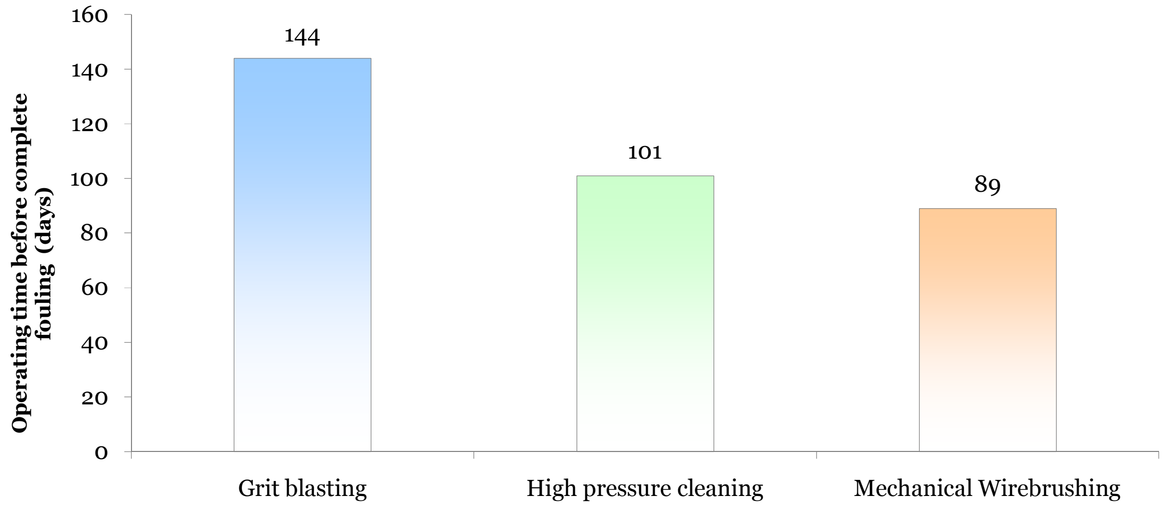

Figure 15. Comparison of sparge pipe cleaning methods.

The grit blasting technique proved to be a significant step forward compared to the traditional mechanical cleaning method using wire brushes.

The way forward

A review of the carbonatation station decolourisation performance revealed a steady improvement since 2000 climaxing at 53% decolourisation in 2008. This is attributed to changes to equipment, operations management and team efforts on the technical front. However, there are a few areas that still require optimisation, including:

- Installation of a reliable method to measure lime dosages on melt using the Distributed Control System (optimisation of lime utilisation).
- Flue gas entering the saturators requires temperature conditioning to further improve the carbonatation reaction.
- The auto-filters require automation with turbidity meters to provide a tighter control on lime carry-over to the resin plant.
- A buffer supply tank situated after the melt station is required to smooth out the erratic flow into the saturators, thus improving the liming efficiency.

Conclusions

The continuous improvement philosophy adopted at the carbonatation station has resulted in consistently good colour and suspended solids removal. The continuous improvement approach is a careful blend of effective operations management and engineering solutions.

The use of a scientific approach to operations management has proved to be very time consuming but very effective in standardising operating methods against given targets, and eliminating the plant variability that arises from making the wrong decisions.

Acknowledgements

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