

Unlocking Rustenburg Base Metals Refiner's Sulfur Removal Section

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The Anglo American Platinum Rustenburg Base Metal Refiners (RBMR) operation is the largest producer of nickel cathode in South Africa. It treats slow-cooled matte permitting the recovery of a platinum-group metal-containing magnetic fraction for downstream processing and a non-magnetic fraction to produce nickel and copper cathode, cobalt sulfate and sodium sulfate. The refinery was recently expanded from 21 to 33 ktpa nickel. During the ramp-up phase, the sulfur removal section was found to be a constraint to throughput. Nickel hydroxide is precipitated from nickel spent electrolyte by addition of sodium hydroxide, and filtration of the nickel precipitate allows sulfur to be removed from the circuit as aqueous sodium sulfate in the filtrate. It was determined that the filtration fluxes were limiting the circuit's capacity. An obvious solution would have been to install additional filtration capacity at significant capital cost and longer lead times. RBMR chose first rather to initiate a collaborative project with Anglo American Technical Solutions to better understand the impact of process conditions on filtration characteristics and to evaluate whether the filtration flux could be increased by improving the quality of the nickel precipitate.

A deeper understanding of nickel hydroxide precipitation chemistry and kinetics in the context of high nickel and sodium hydroxide concentrations was developed, guided by precipitation theory and confirmed by laboratory tests and plant observations. It was found possible to significantly improve the filtration rate by modifying the precipitate characteristics. A new precipitation process was engineered and retrofitted into the existing plant reactor. Since start-up of the new precipitation system, the available capacity of the sulfur removal section has more than doubled, and control of the process has improved with the better agitation. This paper describes the theory and results used to design the new agitation system as well as the impact of the change on capital investment, operability, and process efficiency.

INTRODUCTION

The Anglo American Platinum Rustenburg Base Metal Refiners (RBMR) operation forms part of the company's smelting and refining complex in Rustenburg aimed at separating and recovering both platinum-group metals (PGMs) as well as base metals. The hydrometallurgical route for treating converter matte was adopted by Anglo American Platinum in 1966. Since its inception the refinery has undergone a number of expansions including the commissioning of a new refinery in 1988 and the expansion to 33 ktpa in 2011. The process has evolved to increase throughput, increase recovery and improve safety, health and environment aspects of the operation. The 2011 brownfields expansion was achieved through changes to the process technology using the existing assets and installing some additional equipment, including a new tankhouse for nickel electrowinning. The changes are

described in detail by Bryson et al. (2008). The expansion project proceeded in a capital-constrained environment following the 2008 financial crisis. The project itself was deferred for a year in 2009 and the plant only started plating cathode from the new tankhouse in 2011. Technical difficulties in integrating certain of the new processes and equipment prolonged the ramp-up phase for the plant (Anglo American Platinum Limited Annual Report, 2012). The sulfur removal section in the BMR was found to be a constraint to throughput in 2013, prohibiting the plant from reaching its name-plate capacity.

Nickel hydroxide is precipitated from nickel spent electrolyte by addition of sodium hydroxide, and filtration of the nickel precipitate allows sulfur to be removed from the circuit as aqueous sodium sulfate in the filtrate. The hydroxide is retained within the circuit by redissolution using additional nickel spent electrolyte. This section was identified as a bottleneck by the frequent build-up of solution ahead of the processing step to a point where the plant needs to turn down or stop. The rate of flow is dictated by the flux achievable during the filtration of the hydroxide precipitate. The nominal throughput capacity of the section was estimated to be an equivalent of 21 ktpa (the original plant capacity even though additional filtration capacity had been installed as part of the expansion project) and needed to be increased by more than 50% to reach 33 ktpa.

The obvious solution to increase throughput is to install additional filtration capacity at significant additional capital expenditure. The capital requirement for expanding the filtration capacity was estimated to be in excess of ZAR 60 million with a lead time of 9 months for the first filter. RBMR chose first rather to initiate a collaborative project with Anglo American Technical Solutions (ATS) to better understand the impact of process conditions on filtration characteristics and to evaluate whether the filtration flux could be increased by improving the quality of the nickel precipitate. Underpinning the project is the fundamentals of precipitation (nucleation and growth mechanisms) which are constrained by the desired chemistry of the overall circuit. The scope of the investigation included generating a deeper understanding of the processes involved during nickel hydroxide precipitation in the context of high nickel and sodium hydroxide concentrations; quantifying the extent to which filtration flux can be improved by manipulating the precipitation conditions; and engineering of the process equipment capable of delivering the desired precipitate.

SULFUR DEPARTMENT

The flowsheet can be simplified as shown in Figure 1 for purposes of discussing the sulfur balance. The slow-cooled matte, better known as Waterfall Converter Matte (WCM), is received by RBMR where it undergoes a number of milling and magnetic separation steps to produce a non-magnetic nickel-copper matte (NCM) and a magnetic-enriched concentrate (MEC). The sulfur associated with the matte accounts for 70% of the total sulfur input into the refinery. The MEC is subjected to a number of oxidative pressure leaching and atmospheric leaching steps targeting primarily the sulfide phases to produce PGM-rich residue for processing at PMR. Sulfuric acid is used as lixiviant in these leaching steps, accounting for additional sulfur input.

The NCM undergoes a series of metathetic and oxidative leaching steps in the Leach and Purification section to solubilise and separate nickel from copper. Sulfur is solubilised by the oxidation of sulfides to sulfate. The liquors undergo further purification, including the recovery of cobalt as well as the removal of selenium, tellurium, iron, lead and zinc. The leaching and purification steps require additional sulfuric acid and other sulphur-based reagents. These reagent additions account for the remaining sulfur input into the refinery.

Not all of the sulfur in the matte is solubilised and the remaining sulfur is rejected in the copper pressure leach residue (CPLR). The amount of sulfur rejected with the residue depends on the extent of sulfur oxidation during copper leaching. The solubilised sulfur passes through the copper electrowinning tankhouse to produce copper spent electrolyte. It is used to replace fresh acid addition

into the Leach and Purification section and migrates through the circuit to form part of the feed stream to nickel electrowinning. Some sulfur is rejected from the circuit during the various purification steps, while the remaining bulk of sulfur passes through the nickel electrowinning tankhouse to be rejected in the sulfur removal section. Nickel is returned to the leach and purification section as nickel dissolution solution (NDS). The sodium sulfate produced from the sulfur removal section is transferred to the sodium sulfate crystallisation plant where it is crystallised. The net result is the estimated removal of 60 ktpa sodium sulfate through the sulfur removal section at an equivalent production of rate of 33 ktpa nickel cathode.

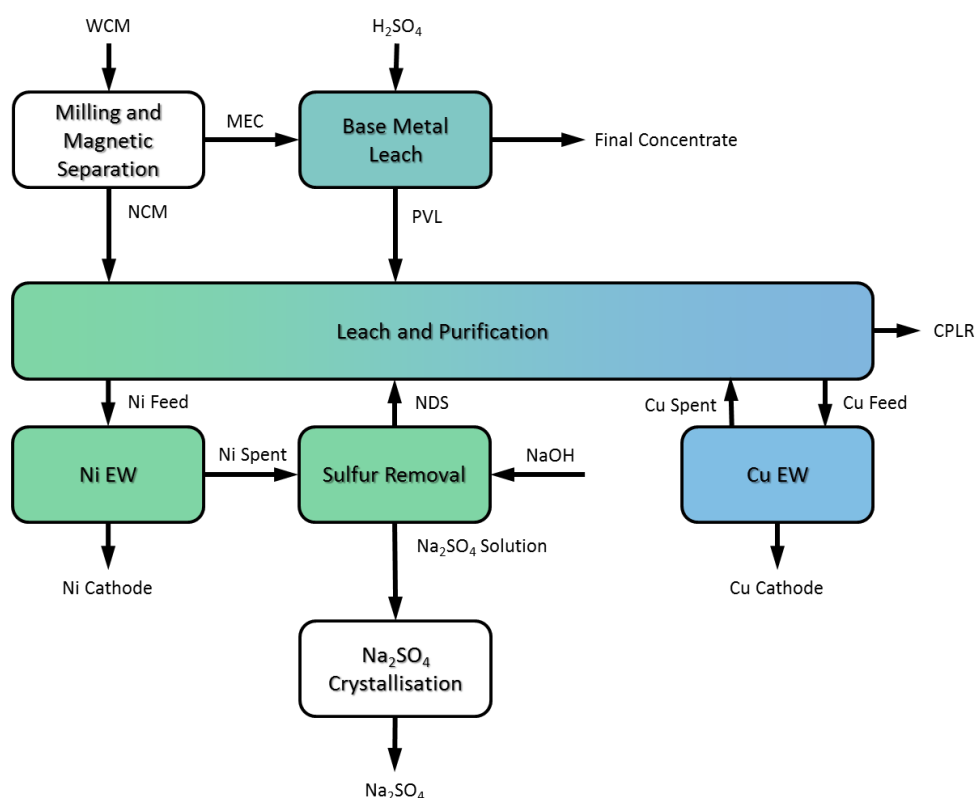


Figure 1. Simplified block flow diagram illustrating sulfur deportment through RBMR.

SULFUR REMOVAL

Soluble sulfur is rejected by the filtration of nickel hydroxide from neutralised nickel spent electrolyte. The sulfur removal section (Figures 2 and 3) draws spent electrolyte from storage into the leading reactor, neutralising free acid using sodium hydroxide. The nickel hydroxide is precipitated in the second reactor by raising the pH, again using sodium hydroxide. The reactors operate at 80 °C. The volume (22 m³) of the reactors results in a mean residence time of approximately 30 minutes per reactor. The precipitated slurry is transferred through a pumpbox to a bank of six rotary vacuum drum filters (Eimco filters). The resultant filtrate passes through a polishing filtration stage raising the pH value to ensure the nickel concentration is below 10 mg/L before it is transferred to the crystallisation plant. The hydroxide cake is re-pulped and transferred to the dissolution reactors where it is dissolved using additional spent electrolyte. The net result of precipitation and dissolution is the neutralisation of free acid produced during electrowinning, resulting in the sodium hydroxide consumption reflecting nickel production. The dissolution solution is finally returned to the Leach and Purification section.

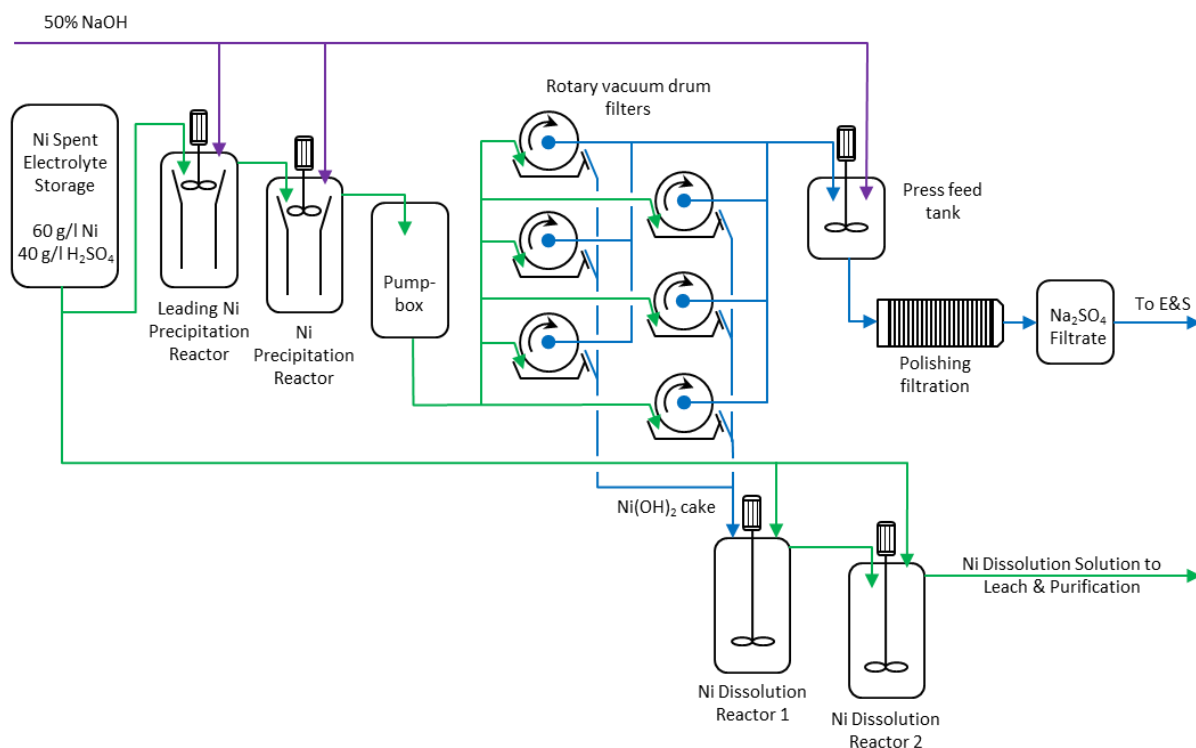


Figure 2. Process flow diagram of the sulfur removal section.

The titration curve of nickel spent electrolyte (Figure 4) indicates the initial neutralisation of free acid followed by the precipitation of nickel hydroxide. The prominent pH increase between acid neutralisation and nickel precipitation allows for the two reactions to be segregated into the leading and precipitation reactors, respectively. The ratio of sodium hydroxide used for the two reaction steps depends on tankhouse operation (advance electrolyte tenor and bite) and is reflected in the split of spent electrolyte to precipitation and dissolution. This split ratio is not explicitly set during the operation of the plant. The operator sets the flow to precipitation and allows the controller to draw additional spent to dissolution to maintain the final pH value of the dissolution solution. The reactions are therefore inherently balanced.

Ideally, the operator would control the flow to sulfur removal to balance nickel spent volume. This is, however, not possible in a system that is constrained by filtration capacity. The plant assumes the characteristics of a “pull” system. The rotary vacuum drum filters operate at maximum capacity and the operator adjusts the throughput in discrete increments to the number of filters on-line. The pumpbox acts as a small buffer, allowing the operator to balance the flows. The flow is subject to fluctuations causing instability in pH control which will be shown later in the paper to be severely detrimental to the filtration characteristics of the precipitate produced. This further aggravates the problem, causing the throughput to drop below that required to balance the spent electrolyte volume, causing the storage tanks to fill. The last resort available to the operator is to drain the boots of the filters to the dissolution reactors, sustaining flow through the plant at the cost of retaining sodium sulfate solution in the circuit. This causes the sodium sulfate concentration to rise and solution storage in other parts of the plant to fill. Inevitably, the plant needs to be turned down or even stopped to allow the sulfur removal section to recover. The obvious solution to this problem is to elevate the filtration capacity of the section through further capital investment. A more challenging solution involves reevaluating the fundamental filtration characteristics of the nickel hydroxide precipitate, however unlikely this may seem since the existing circuit had operated for more than 30 years.

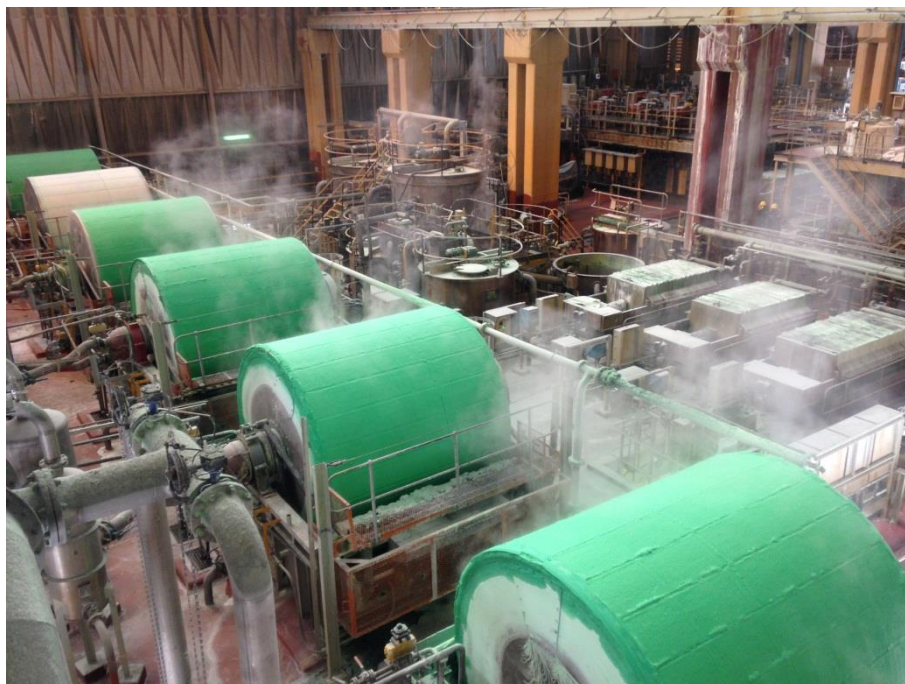


Figure 3. Photograph of the sulfur removal section showing the rotary vacuum drum filters in the foreground and the polishing filters, precipitation and dissolution reactors in the background.

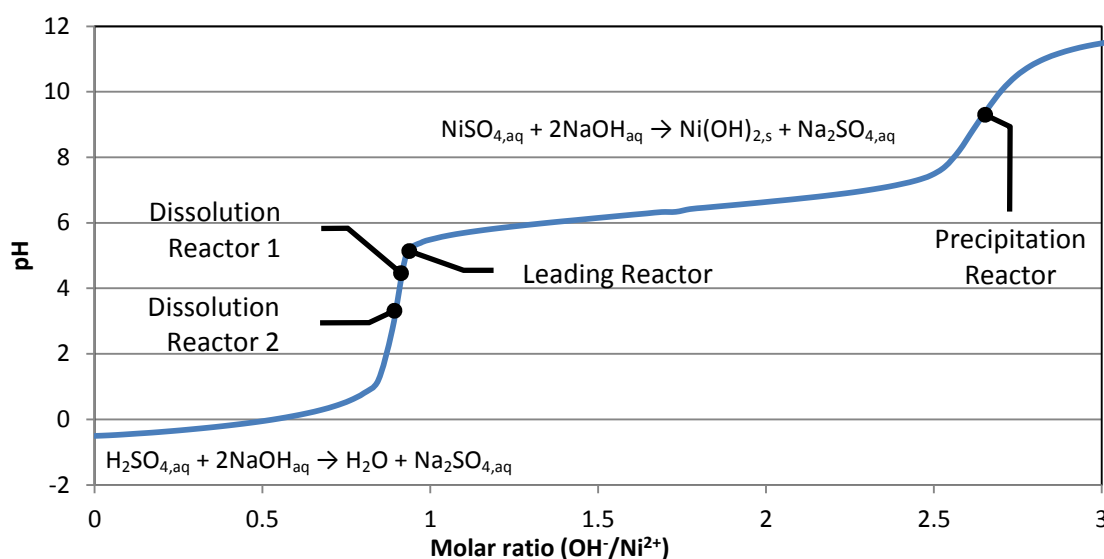


Figure 4. Titration curve of nickel spent electrolyte using sodium hydroxide.

METHODS OF IMPROVING PRECIPITATE QUALITY

Supersaturation is the extent by which a compound exceeds its own solubility in solution and impacts the properties of solids produced. As an example, refer to the textbook by Söhnel and Garside (1992) for a discussion on the relationship between supersaturation and precipitation. The control of supersaturation is not easy, given the low solubility levels and fast reaction kinetics involved during precipitation. The kinetics can exceed the rate of mixing, causing the precipitation reaction to be localised within the reactor volume. Concentration of the reactants and method of mixing play pivotal roles in the level of supersaturation generated and hence the quality of the precipitate produced.

Demopoulos (2009), in his review paper on precipitation in hydrometallurgy, suggests the following methods for controlling supersaturation:

- pH control,
- metal complexation and dissociation,
- dilution,
- redox reactions, and
- dissolution reactions.

The latter two options are not applicable to the large-scale production of nickel hydroxide. The redox method can only be used in multivalent systems such as $\text{Fe}^{2+}/\text{Fe}^{3+}$, while the dissolution method is complicated by the requirement to form a precursor compound from which the final precipitate is produced.

The method of supersaturation control via pH control was studied by Sist and Demopoulos (2003) on synthetic nickel laterite leach solutions (6 g/L Ni). It entails a semi-batch step-wise precipitation procedure, keeping the pH value below the point where the onset of homogeneous nucleation is observed. The method requires seed material to promote heterogeneous nucleation. According to the authors, the method can be translated to a continuous process where each step represents a reactor in series and the final product is recycled as seed. Application of this method at RBMR would require more than the suggested four stages, given the higher nickel concentration (60 g/L Ni).

The control of supersaturation via complexation during nickel hydroxide precipitation is achieved using ammonia, as described by Mubarak and Lieberto (2013) and E et al. (2015). The ammonia complex $(\text{Ni}(\text{NH}_3)_6)^{2+}$ lowers the concentration of free nickel, limiting the supersaturation level. A molar ratio $\text{NH}_4^+:\text{Ni}^{2+}$ in excess of three is required for this method to work. The high ammonia concentration required excludes it from being applied to RBMR.

Dilution of the reagent streams using barren solutions is a guaranteed method of reducing supersaturation but it may not always be practical to apply. The extent of dilution achieved by addition of water or recycling of filtrate within the constraints of the process would be insufficient to alter the dominating precipitation mechanism. It is however possible to internally dilute the feed streams using the reactor's content, depending on the reactor's operating parameters and mixing. This proved to be the optimum solution for RBMR and will be discussed in more detail.

INVESTIGATION

A plant survey was conducted in September 2013 to characterise the circuit. The filtration flux rates (flow of filtrate per unit filtration area) of samples from different points in the circuit were measured using a standardised filtration test (Figure 5). The filtration test was designed to simulate the rotary vacuum drum filters, targeting a similar cake thickness at the same vacuum pressure. Some solids were observed in the leading reactor and, in spite of the low solids loading, presented a high filtration resistance. The highest flux rates were observed at the precipitation reactor, decreasing to half of that value in the pumpbox and ending at a third of the original value at the boot of the filters. This was interpreted to be indicative of a fragile precipitate and seems to have been considered in the design of the original plant. The reactors are fitted with low intensity agitators ($<0.07 \text{ kW/m}^3$) with draft tubes and no agitation in the pumpbox. At the time of the survey, the initial laboratory results indicated that an improved precipitate could be produced under high intensity agitation ($\sim 0.7 \text{ kW/m}^3$) leading to better dispersion of the reactants. The immediate action taken was to lower the NaOH addition point from the surface to a point close to the impeller in the precipitation reactor. This resulted in an increase in nominal throughput capacity from an estimated equivalent of 21 to 24 ktpa nickel.

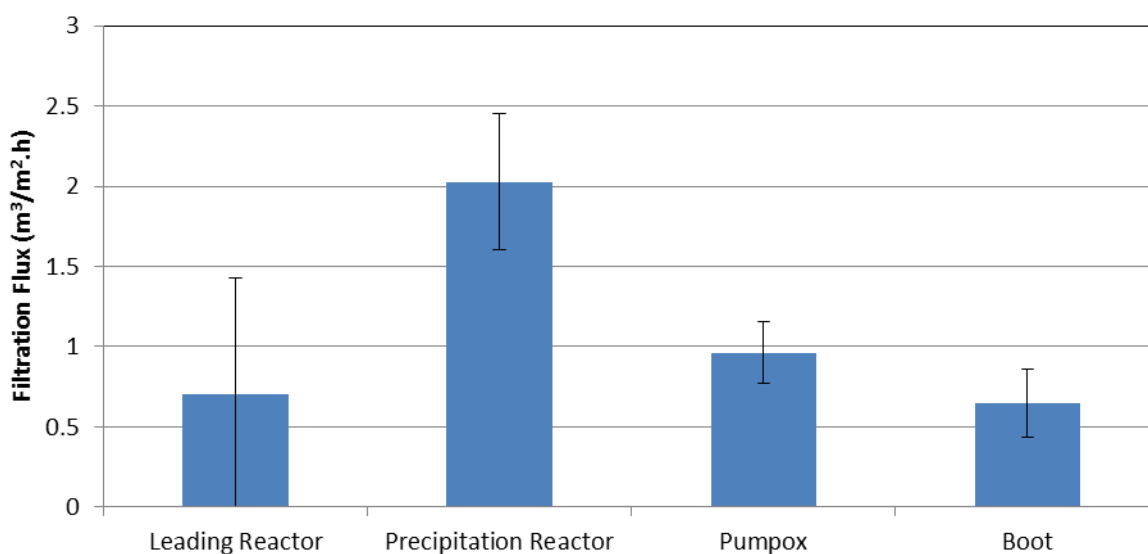


Figure 5. Filtration flux rates measured during plant survey.

Investigation of the process continued at the hydrometallurgy facilities of ATS. A laboratory reactor (Figure 6) was operated as a Mixed Suspension Mixed Product Removal (MSMPR) reactor by continuously feeding plant solution and sodium hydroxide, and overflowing the nickel hydroxide product slurry. The reactor was baffled and agitated using a variable-speed overhead stirrer fitted with a double pitch-blade turbine impeller. The reactor was heated using thermal fluid to control the contents at 80 ± 0.1 °C. All feed and product vessels were placed on load cell platforms for mass balance purposes. A pressurised pH probe was used and the pH value was maintained within 0.05 pH units of the set-point by the controlled addition of 45% sodium hydroxide. All the equipment (pumps, stirrer, heating circulator, pH transmitter and weight transmitters) were connected to a process controller for data logging and control purposes. The majority of the testwork was conducted in a 2 L laboratory reactor. The effect of scale was evaluated in a 7.5 L laboratory reactor and a 50 L pilot plant reactor train. Scale-up was constrained by available equipment and mixing conditions could not be replicated in full.

The initial programme evaluated reaction conditions (agitation intensity, pH set-point and residence time), reactor configuration (pre-neutralisation, reagent dilution, reagent addition point and seed recycle) and scale (2 L, 7.5 L and 50 L). All the tests yield precipitates characterised by filtration flux rates more than double what was achieved under stable plant conditions. The results showed the reaction condition had the most profound effect on the filtration characteristics of the precipitate. Little benefit was derived from changing the reactor configuration and the different scales evaluated showed the results to be scalable.

The improved results from the tests were explained as follows. The low solubility of nickel hydroxide and high concentrations of reactants (60 g/L Ni in spent electrolyte and 45–50% NaOH) can easily cause high supersaturation levels to be generated, pushing the system deep into the homogeneous nucleation region. Mixing in a continuous precipitation reactor involves contacting three fluids, consisting of the two reactant streams and the bulk solution. The combined concentration of the reactants (Ni and OH) in the bulk is at its lowest value by controlling the reactor close to the stoichiometric ratio. Supersaturation levels are then limited when mixing the feed streams with the bulk, effectively diluting the reactants prior to the reaction. This is easily achieved in a laboratory-scale reactor with high circulation rates promoting mixing of the inlet streams with the bulk.

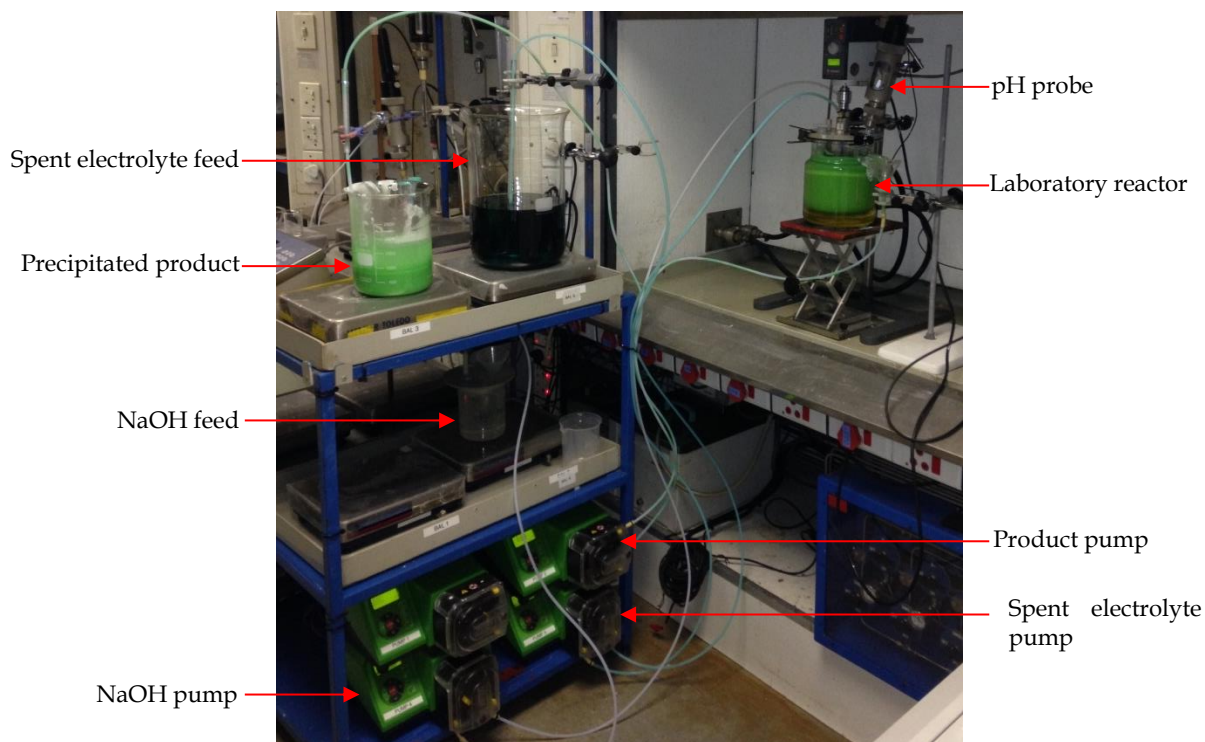


Figure 6. Continuous testwork set-up for nickel hydroxide precipitation.

The mechanism can be further explained using Figure 7. The black diagonal lines represent increasing levels of supersaturation starting from solubility ($S = 1$). The blue and red lines represent the mixing lines of spent electrolyte and sodium hydroxide with the bulk solution, respectively. The solid mixing lines represent the optimum pH value (~ 8.5) with equivalent supersaturation levels generated while mixing the two reactants. With a lower bulk pH value (7.0, dotted lines) the supersaturation level decreases while mixing the spent electrolyte, but increases while mixing sodium hydroxide. The opposite is true with a higher bulk pH value (10, dashed lines). The mixing line with the highest supersaturation level dominates, suggesting deteriorating filtration characteristics on both sides of the optimum.

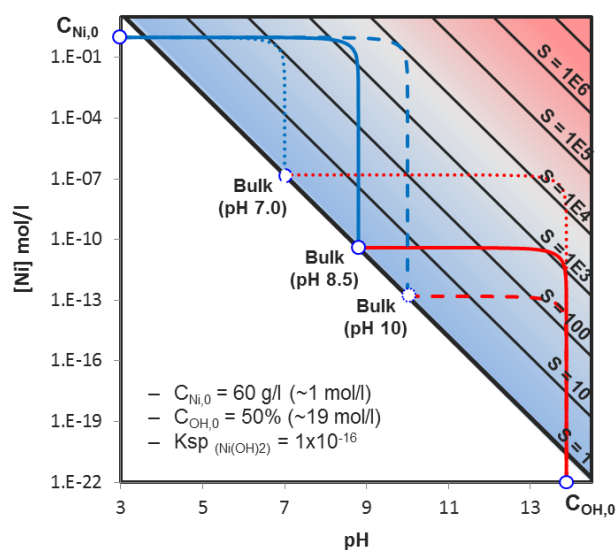


Figure 7. Precipitation diagram for single-stage continuous precipitation of nickel hydroxide. Diagonal line represents increasing levels of supersaturation.

The explanation was tested against an expanded set of runs evaluating the pH set-point (Figure 8). The extents of precipitation in all of the tests were greater than 98%. The highest flux rate was achieved at the point of lowest combined reactant concentration. At pH set-points below this optimum point, the nickel concentration rises in the bulk solution, likely causing high supersaturation levels around the sodium hydroxide inlet. Again, at pH set-points above this optimum point, the hydroxide concentration rises in the bulk solution, likely causing high supersaturation levels around the nickel spent electrolyte inlet. The explanation held up to the extended testwork and was used to develop the design intent of a new reactor.

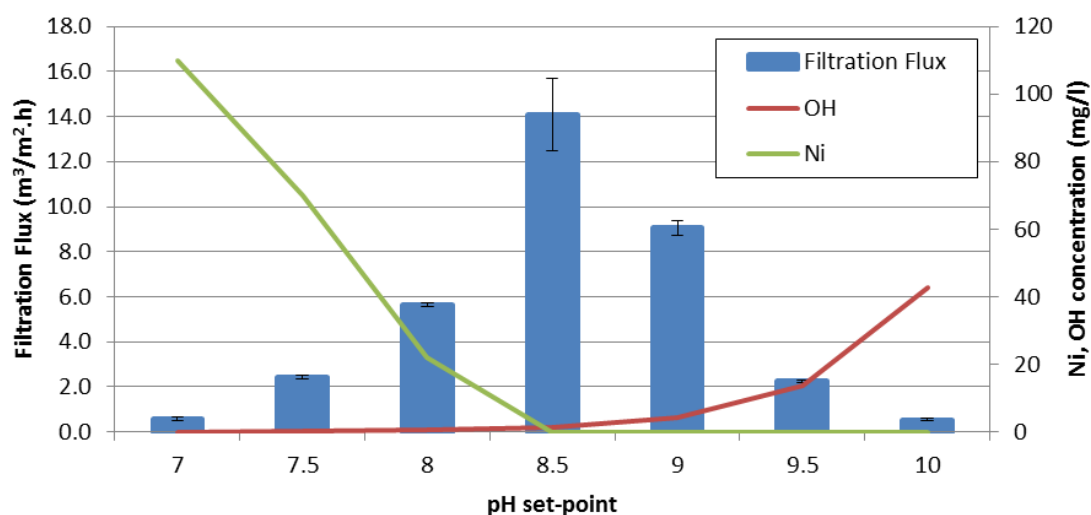


Figure 8. Extended tests evaluating the filtration flux as a function of pH set-point. Nickel concentrations were measured using inductively coupled plasma optical emission spectroscopy (ICP-OES) and hydroxide concentrations were calculated from pH measurements and the water dissociation constant at 80 °C.

Field-emission gun scanning electron microscope (FEG-SEM) micrographs indicate a change in the habit of the precipitate formed (Figure 9). The shape of the precipitates produced at both high and low pH set-point tends to be highly irregular, forming loose agglomerates with some fine particles being present. The precipitate produced at the optimum pH set-point is spherical in shape and more regular in size. The particle size distributions of the precipitates were measured using a laser diffraction instrument (Malvern Mastersizer). The Sauter mean diameters, d_{32} , of the particles produced at both high (pH 9.5, 6.45 μm) and low (pH 7.5, 9.14 μm) set-points were lower than the optimum set-point (pH 8.5, 20.2 μm). Flux through a packed bed is proportional to the Sauter mean diameter of the particles, corroborating the results from the filtration tests.

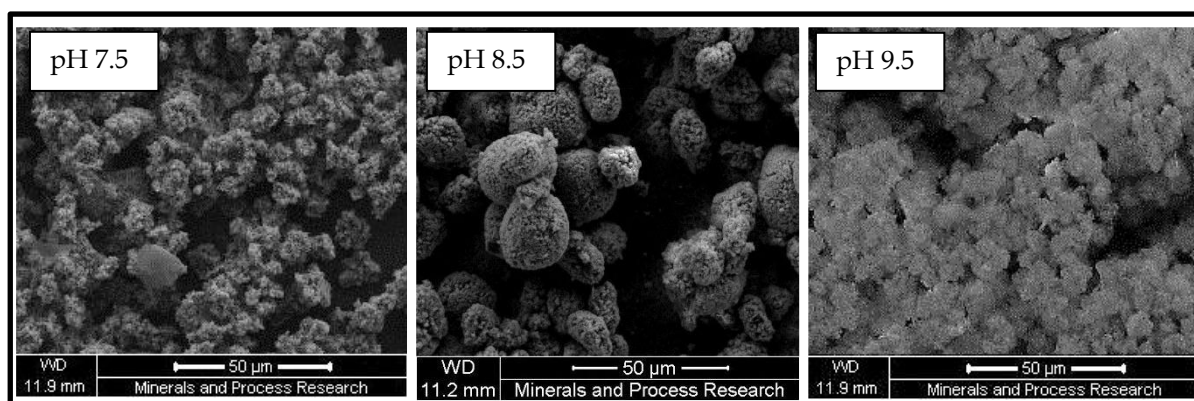


Figure 9. FEG-SEM micrographs of precipitate produced at various pH set-points.

IMPLEMENTATION

A new agitator was engineered and installed into the standby reactor in the precipitation train. The following design intent was used:

- Single-stage precipitation of nickel hydroxide at optimum pH set-point.
- Promote mixing of feed streams with bulk solution.
- Increase circulation of the bulk to promote homogeneity.

The resulting design was a quad-impeller agitator with the feed streams added to the top and bottom of the reactor. Bulk mixing was realised through the multi-impeller arrangement while mixing of the feeds was realised at the individual top and bottom impellers. Off-take from the reactor is from the middle, which is also the measurement point of the pH value. The installed cost of the new agitation system was less than ZAR 1 million.

The agitator was commissioned in June 2014. Mixing in the reactor was characterised prior to hot commissioning to confirm it met the design intent. At the time of installation, there was still some uncertainty of the optimum agitation speed and it was therefore fitted with a variable-speed drive. As part of commissioning, the circulation time of the reactor was measured at varying agitation speeds using the methods described by Bourne (1997). The agitation speed was selected to yield the design circulation time (6 seconds). Performance of the section has since been satisfactory, negating the need for further optimisation. The measured residence time distribution approximated an ideal CSTR (continuous stirred tank reactor) while the circulation time was significantly lower compared to the old configuration (66 seconds).

PLANT OPERATION

One of the immediate effects observed during commissioning was lower boot levels in the rotary vacuum drum filters. The filtration characteristics of the precipitate improved to such an extent that the filtration rate exceeded the feed rate of the individual filters. Spot filtration tests suggest an improved, robust precipitate being produced, with filtration flux rates measured up to 12.4 m³/m².h at the reactor and decreasing by only 20% at the drum filter (67% historic). FEG-SEM micrographs (Figure 10) indicate that the habit of the precipitate had changed to match that observed in the optimum laboratory tests.

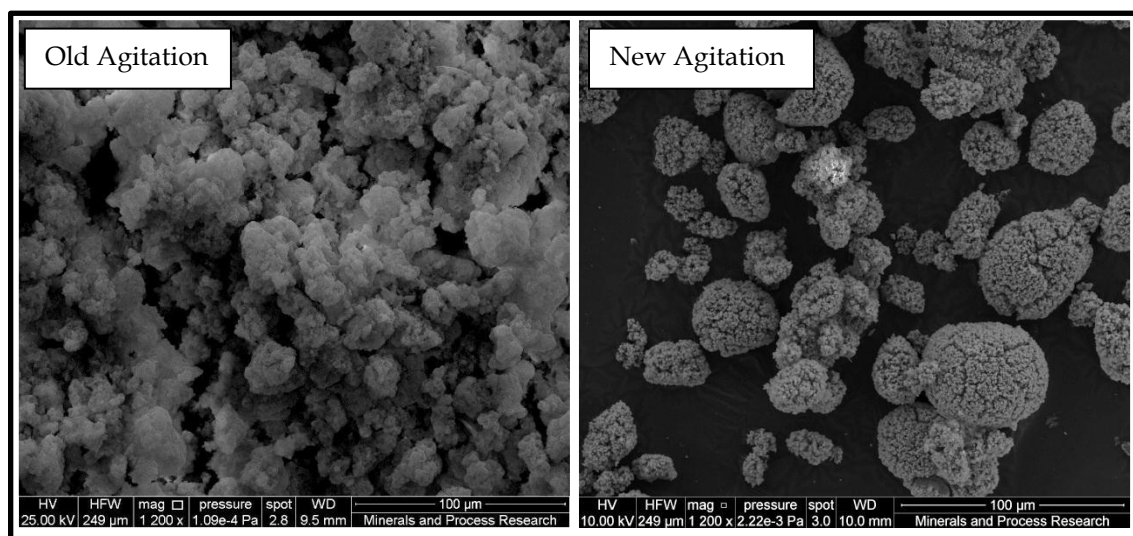


Figure 10. FEG-SEM micrographs of the precipitate produced from the plant before and after installing the new agitation system.

Operability and efficiency of the plant were assessed using process data for the period prior to the investigation, after the change in the addition point of NaOH and after the installation of the new agitation system. pH control in the reactor benefited from the improved dispersion of NaOH, resulting in a faster responding signal. An advanced process controller was deployed with the new agitation system to help control pH. The overall result was a decrease in the standard deviation in the pH signal from 0.86 to 0.63 with the changed feed point and finally to 0.48 with the new agitation system. The improved pH control is essential to consistently produce a good quality precipitate, as was shown during the investigation.

Operability of the sulfur removal section is reflected in the storage levels of the spent electrolyte. The capacity of the storage is 670 m³ and the plant is turned down when the levels approach 600 m³. The frequency of spent storage exceeding 600 m³ has decreased from 9.1% to 4.8% with changed feed point to 1.1% with the new agitation system at continually increasing throughputs. The typical number of filters on-line decreased from five to four with the new agitation system, while the mean boot levels dropped from 85% to 45%. The requirement to drain the filter boots has fallen away, resulting in the average sodium sulfate concentration of the nickel electrolyte decreasing from 170 g/L to 150 g/L. The sulfur removal section is no longer considered to be a constraint to throughput.

The sulfur removal section has consistently been able to match the plant production rate since the installation of the new agitation system. Two capacity runs were conducted to establish the new maximum capacity of the section. This was done by building spent electrolyte volume in storage tanks and then operating the plant with a reduced number of filters. The flow of spent electrolyte was systematically increased to the point where the pumpbox stabilised, indicating the filtration limit. The plant was maintained at this point until the spent volume was depleted. The equivalent nickel production rate was then calculated from the caustic consumption and extrapolated to an equivalent of five filters being on-line (five on-line, one standby). The two runs, first with one filter and the second with two filters, indicated an equivalent capacity of 49 and 55 ktpa, respectively. The section has sufficient filtration capacity to reach 33 ktpa, provided the precipitate's filtration characteristics can be maintained during ramp-up.

CONCLUSIONS

The Anglo American Platinum Rustenburg Base Metal Refiners operation was recently expanded to 33 ktpa nickel cathode capacity and is in the process of ramping up. The sulfur removal section was found to be a constraint to throughput, limiting plant capacity. Through a collaborative effort with Anglo American Technical Solutions the sulfur removal section was unlocked from an equivalent capacity of 21 ktpa Ni to initially 24 ktpa Ni by moving the caustic addition point and finally to a capacity well in excess of 33 ktpa Ni by installing a new agitation system. The new agitation system changed the habit of the precipitate to match what was observed in the laboratory trials. The project was delivered from initialisation through investigation and design to implementation in 12 months. Cost of installing the new agitation system was less than ZAR 1 million. This obviated the need of the fall-back option, which would have required increasing filtration capacity by installing three additional rotary vacuum drum filters at an estimated cost of ZAR 60 million and with a lead time of 9 months for the first filter.

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