

RIP PILOT PLANT FOR THE RECOVERY OF COPPER AND COBALT FROM TAILINGS

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ABSTRACT

Laboratory and mini plant test work were conducted to evaluate the recovery of Cu and Co from a tailings dump material. Results obtained during the test work using a commercially available RIP grade iminodiacetic acid resin are presented in this paper. The primary objective was the simultaneous recovery of Cu and Co, whilst minimising Cu losses due to precipitation. A more successful approach for the efficient recovery of Co was confirmed.

1. INTRODUCTION

Mintek has been developing resin-in-pulp (RIP) technology for base metal recovery from dense slurries since the late 1990s. This included evaluations of RIP technology for the in-pulp extraction of copper, zinc, nickel, cobalt, vanadium, manganese and gold cyanide [1, 2, 3] from different sources. These developments culminated, in collaboration with Bateman, in the development of the Metrix™ technology, and the design and construction of a demonstration unit [4].

The main application of RIP in the base metal industry would initially be scavenging of soluble metals from leach residues, precipitation residues and other solid wastes that have poor filterability or settling characteristics.

The major advantages of RIP are as follows [3]:

- It is effective for the recovery of soluble metals from pulps. The fact that the process operates in a pulp medium means that the upstream solid-liquid separation may be reduced or possibly eliminated. In addition, it may be possible to significantly reduce wash water requirements associated with conventional solid-liquid separation steps.
- RIP may be able to achieve lower discharge metal concentrations than those possible with the more conventional filtration or solid-liquid separation systems, hence reducing the potential for problems associated with the disposal of wastes containing soluble metals.

- The overall metal recoveries on the plant can be improved.
- Co-precipitated and adsorbed metal species can generally be recovered with the RIP process, which will further improve the overall metal recovery.

The target of the research presented in this paper was to recover soluble Cu and Co from a tailings material. These tailings contain acid-soluble Cu and Co that formed primarily due to localised co-precipitation during impurity removal stages and inefficiencies in the solid/liquid separation operations. They also contain some 'insoluble' Cu and Co associated with roast residues that could only be recovered by more aggressive leaching conditions.

Cu and Co could be efficiently recovered via RIP, but Cu could be recovered at a lower pH value than Co. Cu recovery can be done at pH 3-3.5 after ferric removal, while efficient cobalt recovery could be obtained at pH values > 4.0. Mintek previously evaluated the problem of simultaneous recovery of Cu, Ni and Co via RIP [5]. During that study it was concluded that a separate copper RIP plant followed by a Ni/Co RIP plant was economically unattractive at the metal prices used for the evaluation and the low Cu grade and hence annual tonnage (400 t/a). Hence, a combination of the two RIP circuits for the simultaneous recovery of Cu, Ni and Co in a single RIP plant was found to be the most economical.

The recovery of both Cu and Co in a single RIP plant would be expected to be problematic, as Cu precipitates out at the pH at which Co can be recovered most efficiently (4 or higher). However, although some Cu would precipitate at the pH of operation for recovery of Co, it could still be recovered. The efficiency of Cu recovery in this case would have to be confirmed for the specific application.

The current article presents results of the laboratory testwork followed by pilot-plant trials for Cu and Co recovery from the leached tailings. The following aspects of the RIP process were targeted:

- Simultaneous and efficient Cu and Co recovery operating a pH profile across the adsorption circuit;
- Evaluation of pulp and resin residence times on the overall performance of the plant.

2. PREPARATORY LABORATORY TEST WORK

In order to generate optimum parameters for the RIP pilot plant operation, the following laboratory test work should be done:

- Representative ore/tailings sample should be leached to produce pulp resulting in valuable metal concentrations suitable for RIP recovery (a maximum of 20% v/v resin concentration in the RIP adsorption tank);
- Appropriate resin selection should be done. Resin durability and price would be critical, as the upgrading ratio would generally be relatively low and the resin is quite expensive;
- Efficiency of valuable metal recovery at different pH values would determine the optimum pH for simultaneous extraction, whilst limiting valuable metal losses and minimising impurity co-extraction;
- An equilibrium adsorption isotherm should be done employing the selected pH, together with kinetics of adsorption to determine the pulp/resin flow rates to be applied on the plant. A McCabe-Thiele construction on the equilibrium isotherm generally provides a reasonable indication of the stage requirements. During this construction, preliminary stage efficiencies are assumed based on kinetics and previous experience.

2.1. Bulk leaching

Drill core sample of the tailings material was leached using sulphuric acid and the pulp produced had the composition presented in Table 1.

Table 1: Composition of PLS liquor

Element	Mg	Al	Si	Ca	Mn	Fe	Co	Ni	Cu	Zn
Concentration, mg/L	291	184	509	627	47	1720	422	<2	1350	24

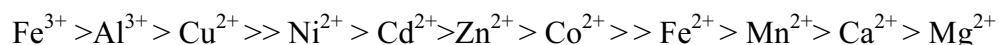
In order to eliminate complications that would have been caused by the presence of unleached solids in the pulp, leached slurry was filtered and the filtrate was used for the laboratory test work.

2.2. Resin selection

The resin selected for this investigation was a commercially-available imino-diacetic acid resin. The final resin choice was based on techno-economic comparisons of potential commercial RIP grade resins, which included resin loss predictions based on a number of durability test methods and the operating size of the resin. The selected resin had a d_{50} of

820 μm in the H^+ form. Reversible swelling from the H^+ to the Cu^{2+} form (fully loaded) and back was about 5 %. The resin was used in the H^+ form, as it would be recycled to adsorption from the acid elution circuit.

The resin selectivity order is given below [6]:



The selectivity order indicates that copper, nickel and cobalt should load in preference to ferrous, calcium and magnesium present in the feed. However, ferric loads preferentially to all these metals and could displace valuable metals from the resin during adsorption. Therefore, iron removal will be required prior to RIP where this resin is employed.

2.3. Cu/Co adsorption vs pH

S-curves (pH vs recovery) for Cu and Co recovery were generated in order to determine the optimum pH value for the recovery of both Cu and Co. Portions of PLS were pre-neutralized via lime addition to the targeted pH values prior to resin addition.

Precipitation of the main impurities and Cu during the pre-neutralization step to different pH values at ambient temperature are shown in Figure 1. Some Cu and Al precipitation was observed at $\text{pH} < 3.5$. It is believed that this was due to relatively fast addition of the neutralizing agent. More than 90 % of ferric and aluminum was precipitated at pH values ≥ 3 and 4.5 respectively.

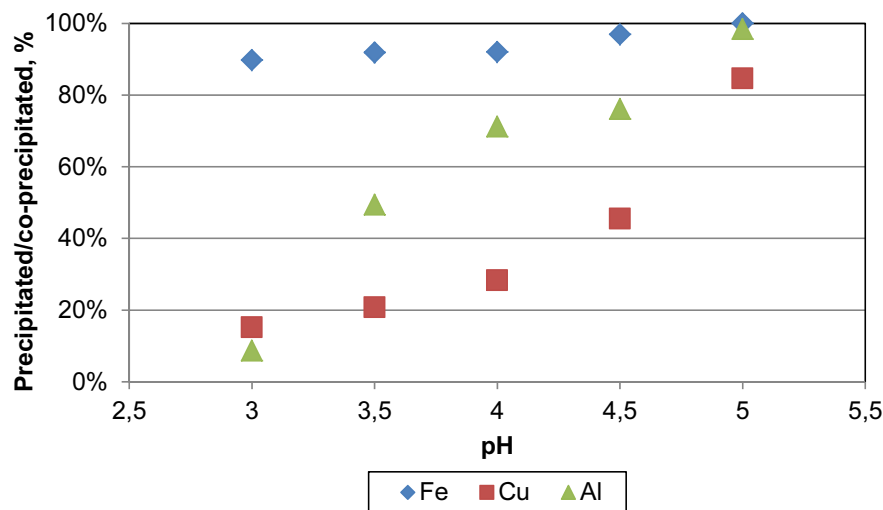


Figure 1: Metals precipitated/co-precipitated after neutralization prior to resin addition

Two S-curves were done at different initial PLS to the resin ratios, where “excess” (PLS:resin vol. ratio = 3.6) and “lack” (PLS/resin vol. ratio = 33.3) of resin relative to the total metals concentration were used. This would give an indication of the resin behavior in the front (“lack”) and back end (“excess”) of the adsorption circuit (see Figure 2).

When excess resin was added (PLS:resin vol. ratio = 3.6), less than 20 % of Fe and 80 % of aluminum present in the system (liquor and solids) reported to the resin at pH values higher than 4.5. Additional resin capacity provided by ‘excess’ resin caused more efficient redissolution of precipitated iron and aluminium hydroxides, and hence higher recoveries

were achieved especially at lower pH values. At higher pH values, the solubility of Fe and Al hydroxides are significantly lower, hence lower redissolution would occur. At a lower resin to PLS ratio (“lack of resin capacity” – PLS:resin vol. ratio = 33) less than 1% and 15 % of Fe and Al respectively reported to the resin at pH 4.5.

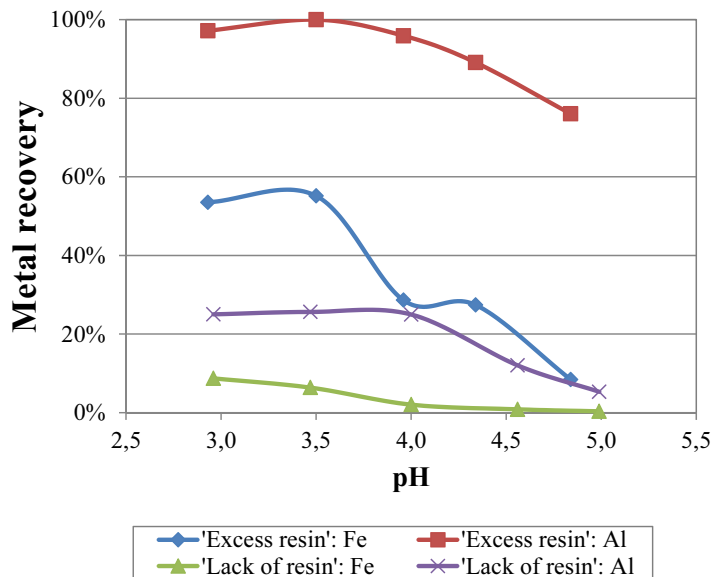


Figure 2: Recovery of impurities vs pH

The cobalt/copper behaviour vs pH for the two ratios tested is presented in Figure 3. Cobalt loading increased with pH, while copper loading remained almost constant, i.e. it would be a function of the equilibrium Cu concentration only. This clearly indicated that in order to maximize Co recovery whilst minimizing resin flowrate, an increased pH profile across the RIP adsorption circuit would be beneficial, as it would prevent Cu precipitation due to a too high pH in the initial stages, but it should maximize Co recovery at the back end of the plant where the Cu concentration would be low (<10 mg/L) and precipitation would be limited.

It was decided to perform the rest of the test work at pH 4.5 (1st RIP adsorption stage), as at this pH reasonable loadings of Cu and Co were achieved and co-loadings of impurities were acceptable, and the precipitation of Cu would be manageable (no copper loss was observed under ‘excess’ resin conditions). Operation at a pH value of 5 in the first 2 RIP stages, where the Cu concentration would still be relatively high, excessive Cu precipitation could occur.

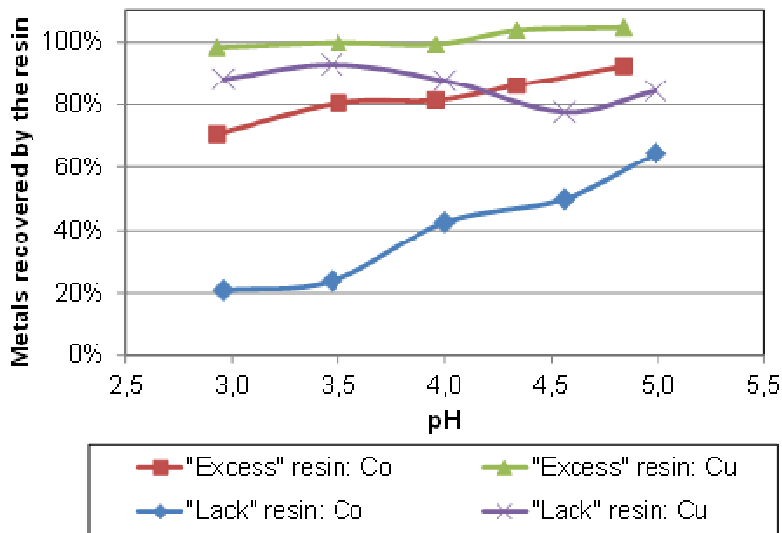


Figure 3: Cu and Co recovery vs pH

The final “loss” of copper to the residue (formed gypsum, Fe-hydroxides and Al-hydroxides) that exited from the resin adsorption test at different PLS:resin ratios is shown in Figure 4.

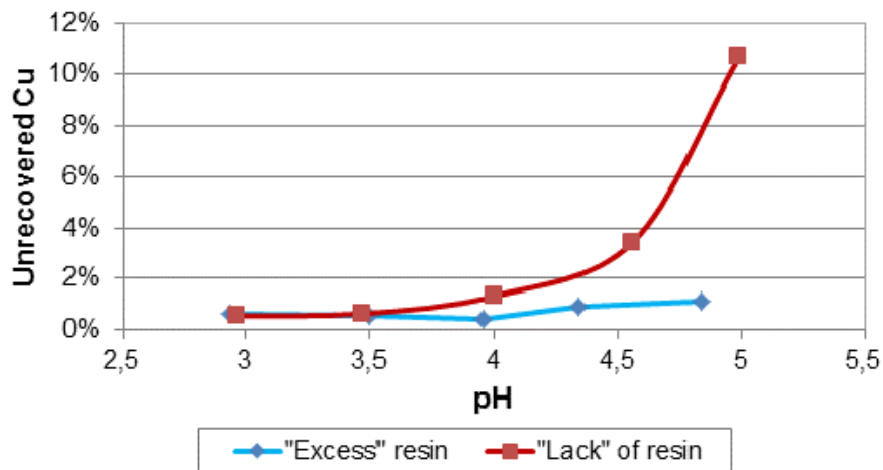


Figure 4: Copper ‘loss’ vs pH

A portion of the copper that co-precipitated with iron and gypsum was recovered by the resin, however, even at pH 3.5 some Cu losses (although low) were observed. This was probably due to encapsulation of Cu hydroxide by gypsum or Cu hydroxide precipitating on lumps of lime. The amount of cobalt reporting to the precipitate was relatively constant at the PLS:resin ratios tested (~2%). These losses were measured from batch tests and would most probably be excessive due to fast rate of neutralization/lime addition, whilst on a continuous circuit this could be minimized.

2.4. Equilibrium adsorption isotherm

Pregnant leach solution obtained after bulk leaching of a sample of the tailings and filtration was pre-neutralized to pH 4.5 via the addition of lime slurry. The portions of slurry obtained were contacted with the resin samples at different slurry:resin ratios for a period of 24 hrs under pH control.

The adsorption equilibrium profiles for the resin in the H⁺ form for cobalt and copper at a pH of 4.5 and ambient temperature (25°C) are presented in Figure 5. The metal concentrations in the feed and after neutralization are given in Table 2.

Table 2: Composition of pre-neutralized feed: equilibrium isotherm generation

Metal	Mg	Al	Mn	Fe	Co	Cu	Zn
	mg/L						
Feed	291	184	47	1720	422	1350	24
Feed after neutralization to pH 4.5 [#]	321	12	51	13	428	858	23
Precipitated/co-precipitated, %	0	93	0	99	0	36	0

[#] Concentrations of Mg, Mn, Co and Zn before and after neutralization indicated that very limited dilution occurred

Almost 40% of Cu was co-precipitated during neutralization. However, most of this copper was recovered in the adsorption equilibrium tests. In order to limit Cu precipitation prior to RIP, it is recommended that pre-neutralization of the pulp only be done to a pH of 3.5 outside the RIP circuit, as this would limit potential Cu losses.

The maximum copper loading that could be obtained from the leached pulp was > 55 g/L at a Cu concentration of higher than 500 mg/L in solution. However, when the Cu concentration in solution increased above 100 mg/L, the Co started to be displaced from the resin (Figure 5).

Behavior of the other impurities is given in Figure 6. Al, Ca, Mg, and Fe were displaced from the resin with an increase in Cu loading. The most noticeable “displacing behavior” of Cu was observed with Ca: with an increase in the copper loading, the calcium co-loadings decreased from 11 g/L (at around 5 g/L Cu) down to 2 g/L (at >50 g/L Cu on the resin).

Total ion exchange capacity of the resin in the H⁺ form, calculated from the sum of the total metal equivalents adsorbed by the resin, was ~ 2.1 eq/L (acid-form resin volume). Preliminary work on the resin indicated that the maximum Cu loading capacity (used as measure for theoretical operating capacity) was 2.2 eq/L. Hence, the metal loadings achieved at pH 4.5 were >95% of the maximum achievable.

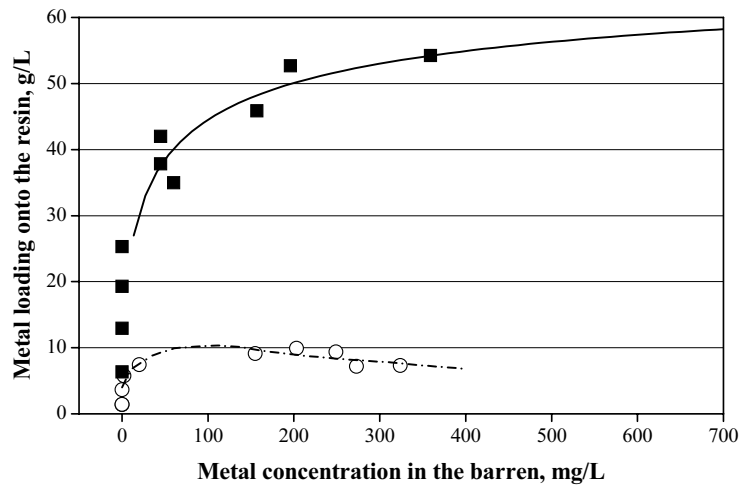


Figure 5: Cu and Co adsorption equilibrium isotherms

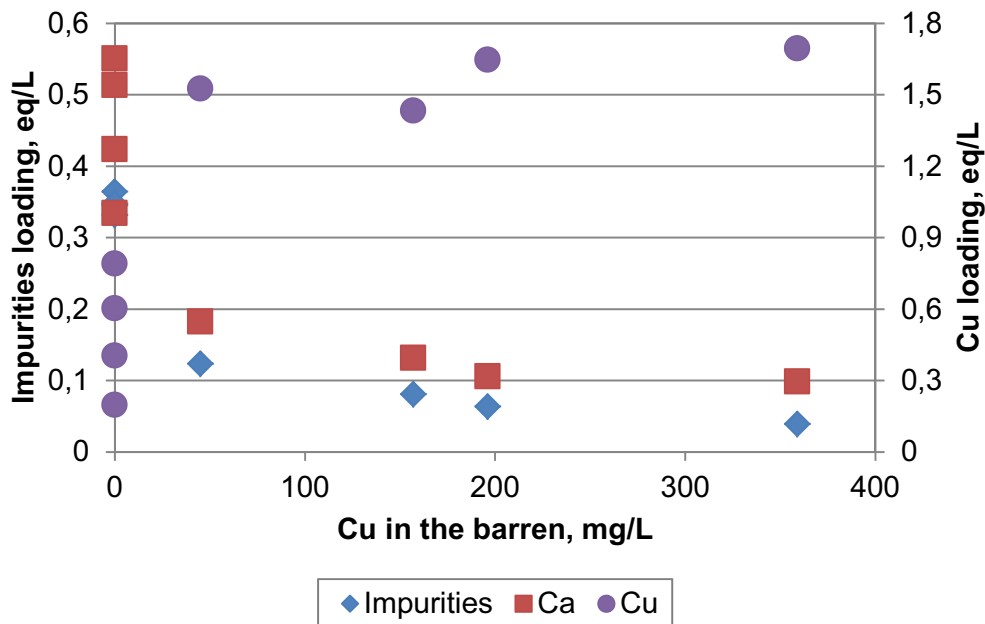


Figure 6: Displacement of impurities by Cu

2.5. Kinetics

Kinetic tests were performed by batch contacting a portion of the resin in the H⁺ form and freshly pre-neutralized PLS in a stirred reactor for a total period of 24 hrs at ambient temperature under constant pH control. Kinetics samples were collected at predetermined time periods. The resin addition was adequate to maximize Co recovery, without Cu starting to displace Co (Figure 5).

Kinetic tests were conducted to evaluate the effect of time on copper and cobalt loading from the pulp (gypsum and co-precipitated Cu and other impurities formed the pulp) at pH 4.5 and ambient temperature. The fractional uptake of Cu and Co versus time is presented in Figure 7.

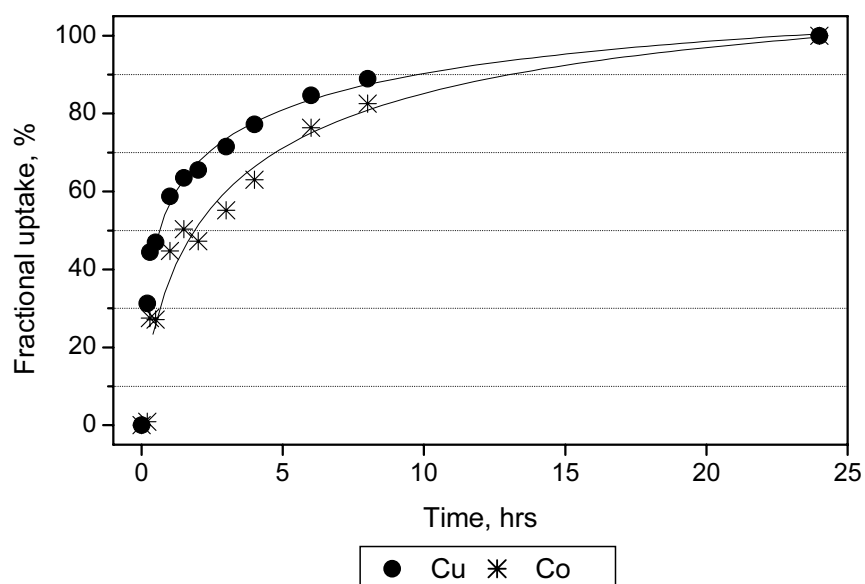


Figure 7: Kinetic of copper and cobalt adsorption

Fractional uptake of Cu/Co was calculated using the following equation:

$$\text{Uptake, \%} = \frac{Me_{\text{feed}} - Me_t}{Me_{\text{feed}} - Me_{\text{eq}}} \times 100 \quad \text{Equation 1}$$

where Me_{feed} – available metal concentration in the feed (excluding associated with solids), g/L;
 Me_{eq} – metal concentration in barren solution after 24 hours, g/L;
 Me_t – metal concentration in barren at time t, g/L.

Rate of Cu and Co adsorption was rather slow, possibly because of a complex mechanism of adsorption. The resin-in-pulp system consists of solids, solution and resin; hence, processes of re-dissolution/precipitation could also influence kinetics, complicate the mechanism of adsorption and in some cases become a process controlling step. Cu uptake

was faster than Co. However, this would have been a function of the gradient concentrations observed for Cu and Co at the pulp:resin ratio used: Cu concentration in the solution went down from 1.35 g/L to 0.045 g/L, while Co concentration decreased from 0.42 to 0.15 g/L.

The overall resin residence time at the start of this study was proposed to be 8 hrs (2 hrs/stage, 4 stages), which corresponded to ~90 and 80 % of Cu and Co overall fractional uptake respectively. However, a batch kinetic test is only an indication of the rate of loading, as the driving forces on the resin and in solution across the adsorption circuit are very different. The stage efficiency was assumed to increase from ~60 to 100 % as pulp flow from the 1st to the last reactor.

3. RIP PILOT PLANT

It was decided to run a pH profile across the RIP circuit to improve the Co recovery. The pH profile applied across the adsorption circuit was 4.5, 4.7, 5 and 5.2-6. Efficient recovery of Co would be required for any operation based on the value of Co, so Cu would probably be recovered across the first 2 stages and hence the Cu loading would be relatively depressed (<1 g/L in 1st stage). In the case of using the current leached tailings sample as feed, it most likely would cause increased impurity co-loadings on the resin exiting the adsorption circuit.

3.1. Pilot-plant preparations

3.1.1. McCabe-Thiele construction and calculations

Input parameters for the pilot plant were based on the copper adsorption equilibrium isotherm and kinetic test. The McCabe-Thiele construction produced is shown in Figure 8. Extraction efficiencies were chosen based on fractional uptake of Cu (Figure 7). Stage efficiency of the 1st adsorption stage (60 %) was taken based on 2 hr resin residence time/stage and this was used for the planning of the campaigns.

As the plant operated in a batch counter-current mode, the residual Cu and Co mass remaining in the Stage 1 pulp at transfer was estimated and taken into account for determination of the pulp:resin flow required. Co adsorption efficiency in the 1st stage was expected to be 0% or negative due to displacement of Co loaded at stage 2 with copper. Parameters tested, as well as resin loadings expected, are listed in Table 3.

Table 3: Parameters tested and expected outcomes from RIP campaigns

Cu in feed, g/L	1,35	Cu @ stage 1, g/L (predicted)	0,54
Co in the feed, g/L	0,4	Co @ stage 1, g/L (predicted)	0,4
Solids content, %	25	Cu/Co @ stage 4, g/L	0,0
Condition No	1	2	3
Pulp τ , hr	0,5	0,5	0,75
Resin τ , hr	2,0	3,0	3,0
Cu loading predicted, g/L	43,3	42,0	43,2
Co loading predicted, g/L	10,7	11,1	10,6
Pulp : resin flow	43	41	43
Liquor : resin flow	32	31	32

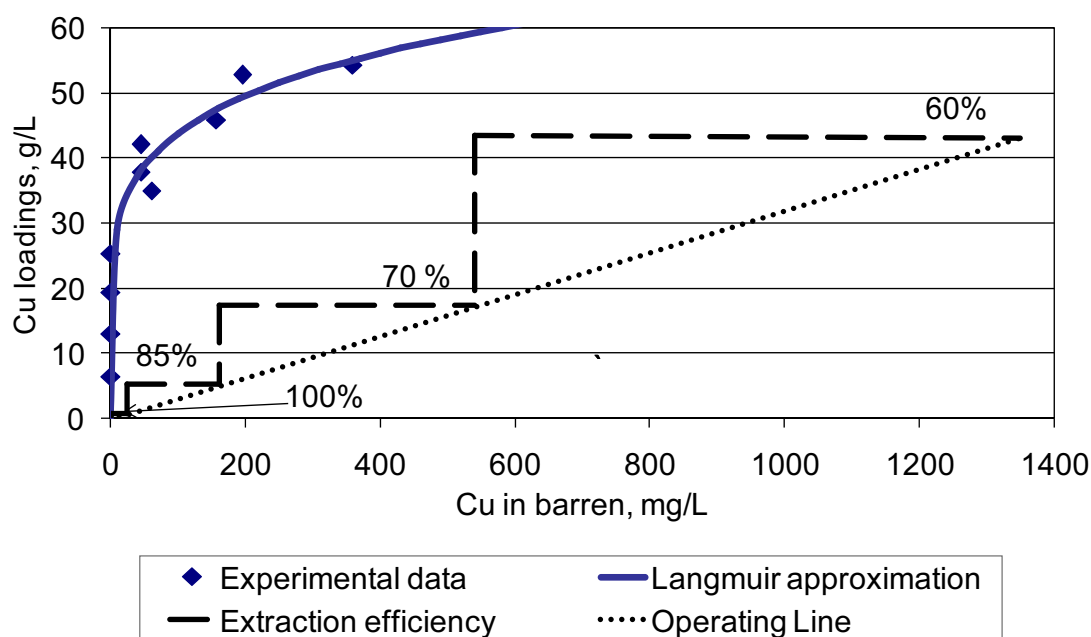


Figure 8: McCabe-Thiele construction for copper recovery

3.1.2. Bulk leaching

The “grab” tailings dump sample was leached with sulphuric acid at 25% solids (m/m) ratio in bulk. Leach behavior of the sample differed significantly from the one tested before and used for preparatory work. Final pH was lower (pH 1.3 against pH 1.8 observed previously) and Eh was 280 indicating that all the iron was present as ferrous. Cobalt concentration was lower than it was expected and, hence, the pulp was spiked with cobalt sulphate in order to end up with ~400 mg/L of Co.

3.2. Operation of RIP plant

A schematic diagram of the pilot-plant is shown in Figure 9.

3.2.1. Pre-neutralization of the feed leached pulp

Fe removal and pre-neutralization was done in two stages. Pulp was pumped through 5L reactors at 10 L/h flow rate (0.5 hr residence time/stage). The pH in the first reactor was 2.5 and in the second one – 3.5. Constant addition of H₂O₂ into the first reactor was done in order to keep the Eh at 570-580 (platinum probe) and convert all the iron into ferric. The pH was controlled automatically via lime addition (20% m/m Ca(OH)₂). These conditions were applied for the entire operating period, and were not specifically optimized.

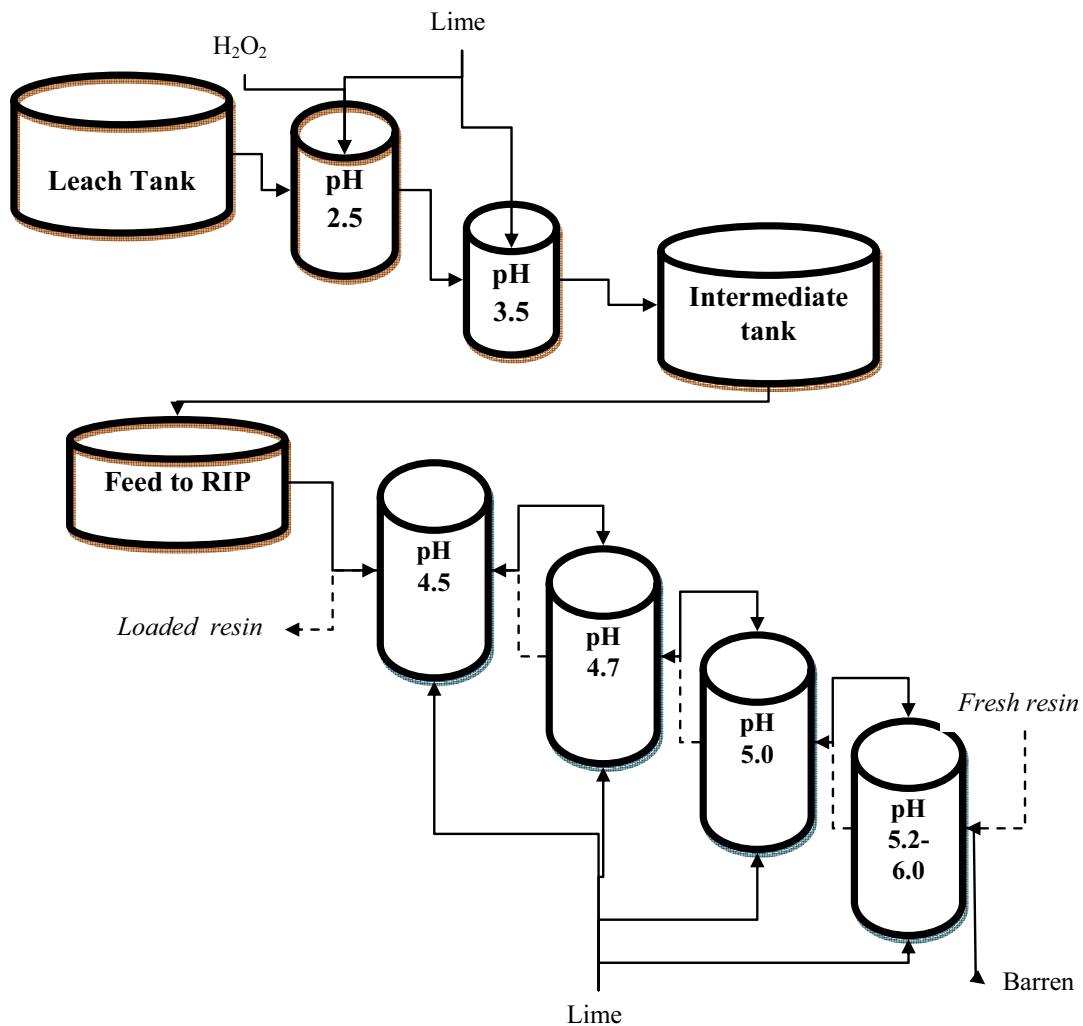


Figure 9: Cu and Co recovery pilot-plant

3.2.2. Adsorption

During adsorption, the feed pulp was contacted with resin in a batch counter-current mode of operation. The pulp was pumped into Stage 1 and it flowed by gravity through the plant, while resin was transferred manually batch-wise counter current to the pulp flow.

The plant consisted of four active adsorption stages, each with an operating volume of 5 L. Mixing of the resin and pulp was achieved with mechanical overhead stirrers. During adsorption, the pH was controlled in all the stages by automatic addition of 20% m/m Ca(OH)₂ slurry. Appropriate volumes of fresh resin in the H⁺ form were added into each reactor entering the adsorption circuit.

At transfer, the 1st reactor was taken off line, and each one of the other reactors moved one position up. A 5th adsorption tank containing the fresh resin was moved into the Stage 4 position. The resin and pulp from the reactor that exited the circuit were separated and removed from the circuit (Cu and Co masses associated with the solution were accounted for in the mass balance). A portion of the loaded resin was stripped to determine the metal loading, and the remainder was stored for further elution test work. At steady state, a small volume (10 mL) of loaded resin was removed from each stage at transfer in order to determine the metals loading profiles across the adsorption circuit. These resin volumes were replaced by fresh resin.

3.1. Comparison of results under different operating conditions

Applying an increased pH profile across the adsorption circuit proved beneficial, as the cobalt loadings were close to maximum expected.

3.1.1. Cu and Co recoveries

Results obtained during operation of the pilot-plant did not achieve the targeted Cu loading of 42-43, due to the fact that somewhat higher masses of Cu were removed from the 1st stage (in solution) at transfer than that estimated upfront (Table 3). Hence, the Cu throughput was somewhat lower than planned.

Comparison of the plant Cu and Co recovery efficiencies at the 3 different operating conditions tested is shown in Figure 10 and Figure 11 respectively. Actual operating lines varied due to the different Cu throughputs as mentioned above. Variation in Co operating lines was less significant for Conditions 2 and 3 due to the fact that the cobalt concentration in the 1st stage exiting the plant was close to that predicted.

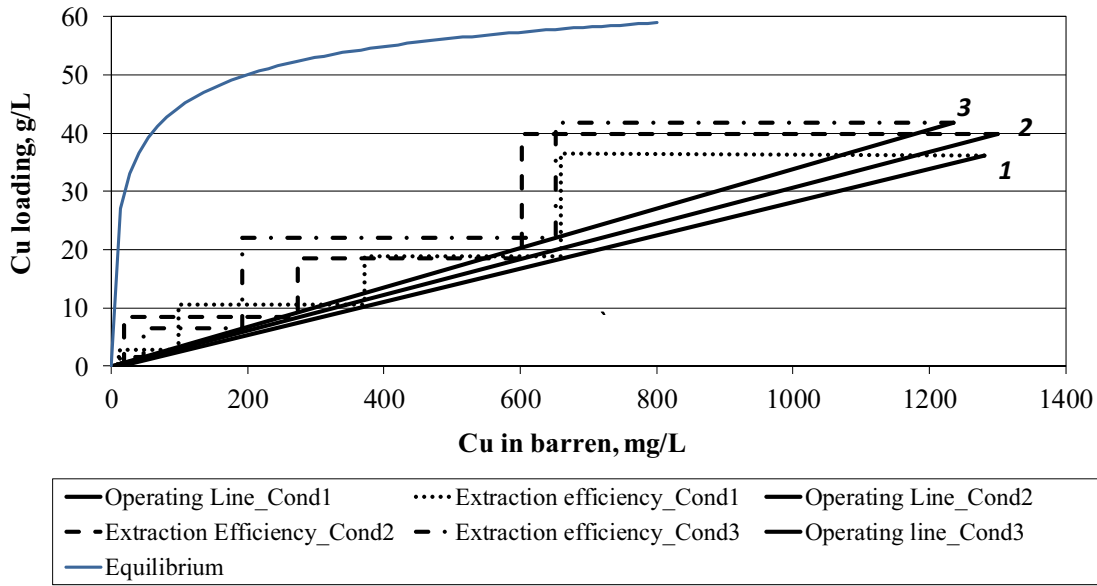


Figure 10: Copper behavior under different operating conditions

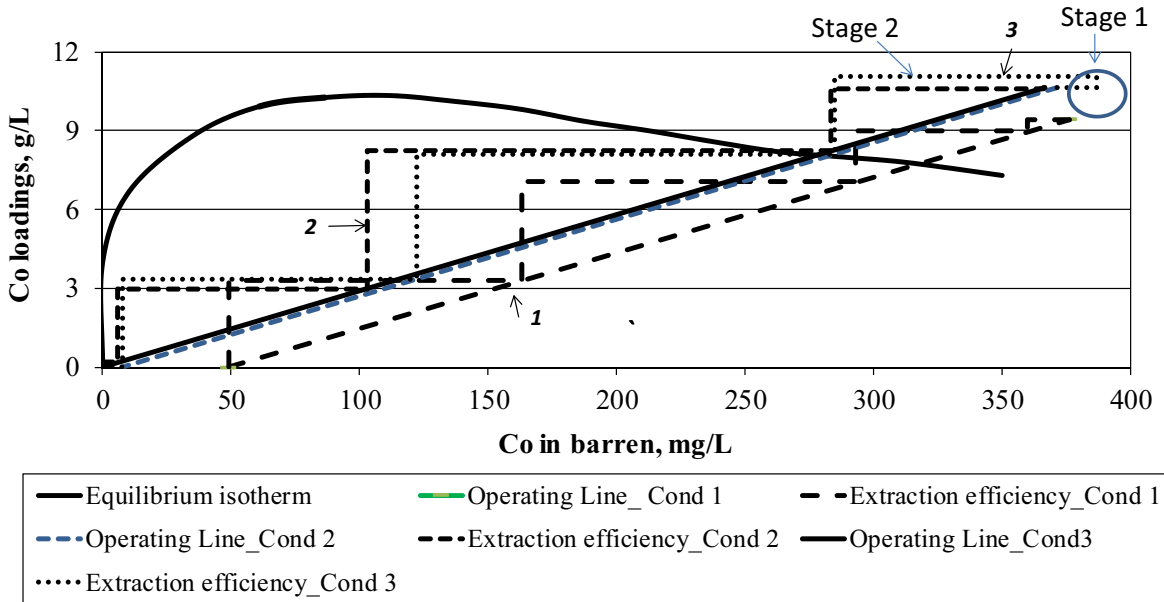


Figure 11: Cobalt behavior under different operating conditions

A resin residence time of 2 hrs and a pulp residence time of 0.5 hrs per stage were inadequate for efficient recovery of Cu and Co at the pulp:resin ratio tested, the number of stages, and the pH profile across the adsorption circuit. Implementation of such conditions resulted in leakage of >10 mg/L Cu and >30 mg/L Co.

Increasing the resin residence time to 3 hrs improved the overall recoveries of copper and cobalt significantly, resulting in <2 mg/L of Cu and <10 mg/L of Co reporting to the barren. The longer resin residence time would increase the resin inventory by around 50% in the adsorption circuit, which might only be an overall resin inventory increase of about 16% (elution time of 8 hrs).

Increase in the pulp residence time to 0.75 hrs per stage did not seem to improve recoveries of Cu and Co significantly, as there was still some leakage of Co. However, under this operating condition, some Co was displaced from the resin by Cu in the 1st adsorption stage (Co solution concentration higher than that in the feed) as was expected during planning of the campaigns. As this was the final campaign, any Co build up in solution (due to displacement from the resin) within the circuit would have been closer to actual steady state conditions than during the first two campaigns.

3.1.2. pH profile and Co behaviour

The barren solution exiting Stage 4 still contained >5 and <10 mg/L Co for Condition 2 and 3. The pH was significantly increased (5.2 to 6) in the last stage in order to eliminate soluble Co leakage. Results obtained during testing of Condition 2 and 3 are shown in Figure 12.

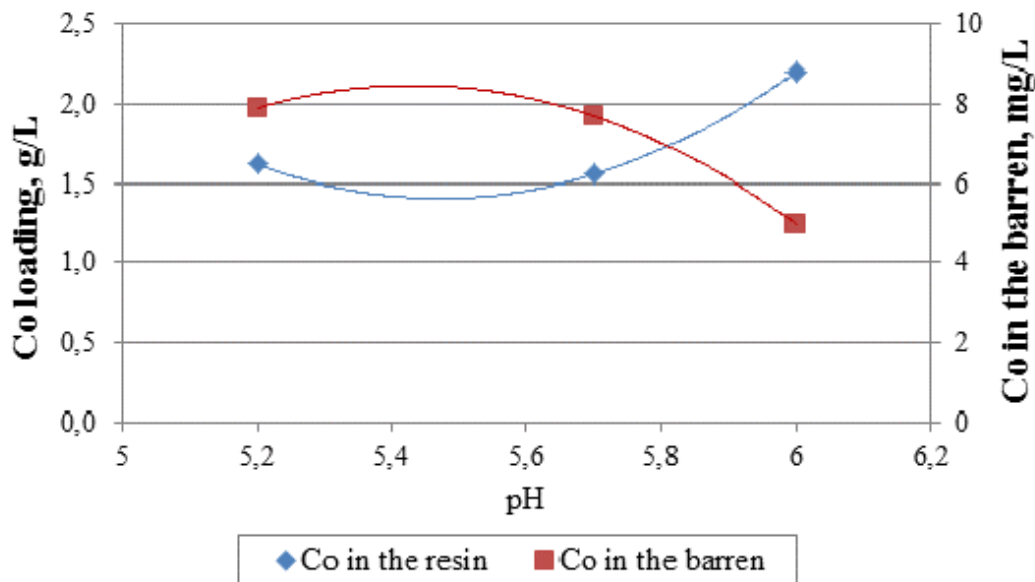


Figure 12: Cobalt loading/uptake vs pH in the final stage

It is realised that the pulp residence time was higher for Condition 3, but it is believed that the change in loading onto the resin was primarily a function of pH and not residence time. However, to further decrease soluble Co losses, a slightly lower pulp: resin flow or an additional adsorption stage would be required.

3.1.3. Impurities deportment to loaded resin

Mg, Ca and Zn had similar adsorption behaviors: they were loaded in the last two adsorption stages together with Co at pH 5-6 and later displaced to an extent by Cu in the first and second stage at pH 4.5-4.7. Their concentration in the resin exiting the plant did not vary significantly at conditions tested. The final Ca, Mg and Zn loadings were 3.3, 0.32 and 0 g/L respectively. Some displacement of Mn was observed in the first stage, with its maximum loading being achieved in the second stage at pH 4.8. Some loading of Al occurred in spite of the fact it should be precipitated at pH>4.5. Ni loading trend was similar to that of Co, resulting in a maximum loading of ~0.3 g/L on the resin exiting Stage 1.

Generally, the final deportment of impurities such as Mg, Ca, Zn and Mn to the loaded resin exiting Stage 1 was relatively insensitive to the three operating conditions tested. However, the increase in pulp and resin residence times appeared to have influenced the impurity loadings in Stages 2-4, probably due to the displacements and the internal solutions achieving steady state.

Iron deportment to the loaded resin exiting Stage 1 varied slightly, but the averaged loading obtained was 0.05 g/L.

4. CONCLUSIONS

Preparatory test work for the pilot-plant indicated that:

- More than 90% of iron and aluminium precipitated at pH>3 and 4.5 respectively. However, some iron and aluminium still reported to the resin due to some re-dissolution of the precipitates formed if excess resin was available.
- Cobalt loading increased with pH, while copper loading remained almost constant. Optimum pH value for simultaneous Cu and Co recovery was found to be 4.5, as operation of the first stage at pH > 4.5 could cause excessive Cu precipitation and, hence increase the potential soluble Cu loss.
- The equilibrium adsorption isotherm indicated that with an increase of pulp:resin ratio Cu started to displace Co from the resin. It was impossible to achieve Cu loadings higher than 45 g/L without a loss in Co loading (based on equilibrium with specific pulp composition at pH 4.5). Employing a pH profile across the adsorption circuit would allow more efficient recovery of Co, whilst minimising Cu losses. This actually causes an increase in Co loading above that expected from the equilibrium isotherm (due to internal Co solution concentrations varying).

During operation of the pilot-plant it was established that:

- It was impossible to achieve <2 mg/L Cu and Co in the liquor going out of the plant at τ_{resin} of 2 hrs and τ_{pulp} of 0.5 hrs at resin:pulp ratio tested within 4 adsorption stages. Decrease in the resin:pulp flow (and hence impurities throughput) or introduction of an additional adsorption stage would help in improving Cu and Co recoveries.
- Increase in τ_{resin} from 2 to 3 hrs improved Cu and Co overall recoveries significantly, resulting in <2 mg/L of Cu and <10 mg/L of Co reporting to the barren. Stage efficiencies for Cu recovery increased with an increased τ_{resin} , while cobalt recovery in the 1st stage tended to be zero.
- Increase in τ_{pulp} from 0.5 to 0.75 hrs per stage at constant τ_{resin} of 3 hrs did not improve recoveries of Cu and Co significantly, with around 5-8 mg/L of Co reporting to the barren. Co concentration in the 1st stage was slightly higher than in the feed, which was not seen during the first 2 campaigns. This was attributed to the total length of the overall pilot campaigns and, hence, the solution composition was closer to actual steady state.
- Applying an increased pH profile across the adsorption circuit proved beneficial, as the cobalt loadings were close to maximum expected based on the Co mass flow and simultaneous recoveries of $> 99\%$ for both Cu and Co in one RIP plant.
- The final deportment of impurities such as Mg, Ca, Zn and Mn to the loaded resin was relatively insensitive to the conditions tested.

5. ACKNOWLEDGMENTS

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