Mitigating Effects of Silica in Copper Solvent Extraction

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Copper–cobalt ores in the Democratic Republic of Congo are characterised by high silica contents. These minerals partially dissolve during acidic sulphate leaching of the ores and significant amounts of silica report to the pregnant leach solution in various forms, including dissolved, colloidal, or gel-type silica. The presence of silica in the feed to the downstream solvent-extraction circuit gives rise to considerable operating problems, including severe crud formation, difficulties in maintaining organic phase continuity, high aqueous-in-organic entrainment, and transfer of impurities to the electrowinning circuit. An inexpensive inorganic reagent has been identified that successfully mitigates the effects of silica in copper solvent extraction through chemical means. It has no adverse effects on the chemical properties of the extractant system or the downstream electrowinning, but exhibits a remarkable effect in improving the physical properties of the solvent-extraction system. Results are presented for both synthetic solutions and real leach liquors from an operation that suffers from severe silica contamination.

INTRODUCTION

The Central African Copperbelt, which stretches from Zambia through to the Democratic Republic of Congo (DRC), is estimated to possess over one-third of the global reserves for cobalt and one-tenth for copper (Crundwell et al., 2011). The main minerals of the oxide zone, which is the focus of most current operations, are malachite (CuCO₃·Cu(OH)_2), chrysocolla (Cu, Al)_2H₂Si₂O₅(OH)₄·nH₂O) and heterogenite (CoO(OH)), with siliceous dolomite and quartz (SiO₂) as the main gangue minerals (Crundwell et al., 2011). Copper production is via a hydrometallurgical process that comprises leaching of the ore in sulphuric acid, solution purification using solvent extraction (SX) and metal recovery by electrowinning (EW) (Schlesinger et al., 2011).

The high silica contents of these copper-bearing ores and their host rocks result in high levels of dissolved and colloidal silica in the pregnant leach solutions (PLS). On reporting to the SX circuit, this silica causes a multitude of physical problems, many of which are not typically experienced in other regions of the world. The most well-known of these is severe and excessive crud formation (Cytec, 2006; Megaw & Burelli, 2015; Sole & Tinkler, 2016). Other effects include retardation of phase separation times, flipping of phase continuity—even under organic-to-aqueous phase ratios (O:A) exceeding 3:1, and high organic-in-aqueous entrainment losses to the raffinate and associated increase in extractant and diluent consumptions. In particularly severe cases, silica can transfer to the EW circuit, where it polymerises due to the high acidity and may deposit on the lead anodes, which can lead to shorter anode lifespan.
In recent years, several approaches to mitigating the effects of silica in these circuits have been attempted (Gillaspie et al., 2010). The ideal process would provide a way to remove supersaturated dissolved silica from the PLS without impacting on overall metal recovery or purity. The use of silica-specific coagulants has had only limited success (Brown et al., 2017; 911 Metallurgist, 2018): in many cases, the surface-active nature of the coagulant itself interferes with the interfacial SX reactions and can sometimes make the problem worse (Bender et al., 2018). Practically, physical approaches have had some success. These include control of the O:A ratio (Kashala et al., 2018) and use of a diluent wash (Alexander et al., 2018); however, their application is constrained by existing equipment limitations. To date, there is no fully acceptable and affordable approach to dealing with this problem.

In this work, the use of an inexpensive inorganic reagent to chemically mitigate the effects of silica in copper SX circuits is proposed. The reagent, a proprietary chemical with the tradename SilStop, was tested on both synthetic solutions and real leach solutions obtained from a Copperbelt operation that suffers extreme effects of silica. It is demonstrated that the reagent is able to counteract the physical problems caused in SX by the presence of silica, but has no adverse effect on the chemical properties of the SX system. It also has no adverse effect on the morphology and purity of the cathode product, should it be carried over to the EW circuit.

**CHEMISTRY OF SILICA IN SOLUTION**

The leaching of silicon-bearing copper ores or silicates associated with the host rock takes place according to Eq. [1], as exemplified by the mineral chrysocolla (Readett & Miller, 1995; Schlesinger et al., 2011):

\[
\text{CuSiO}_3 + 2\text{H}_2\text{O} + \text{H}_2\text{SO}_4 \rightarrow 2\text{CuSO}_4 + \text{Si(OH)}_4 + \text{H}_2\text{O}. \quad [1]
\]

Silica can also be present in the PLS as suspended particles that have not been adequately removed by upstream clarification processes. Dissolved silica is in the form of monomeric silicic acid, Si(OH)\(_4\) (Cooper, 1998). This remains in solution until its solubility limit is reached under particular conditions of temperature, pH or aging. Supersaturation in deionised water typically occurs when the concentration of SiO\(_2\) exceeds 120–150 mg/L (Zuhl & Amjad, 2013). When there is no suitable surface for the monomer to settle onto, polymerisation will take place to form colloidal silica. The polymerisation reactions that lead to the growth of colloidal particles are given by:

\[
\begin{align*}
2\text{Si(OH)}_4 &\rightarrow (\text{H}_2\text{SiO}_4)_4; \quad [2a] \\
\text{Si(OH)}_4 &+ (\text{H}_2\text{SiO}_4)_2 \rightarrow (\text{H}_2\text{SiO}_4)_3; \quad [2b] \\
\text{Si(OH)}_4 &+ (\text{H}_2\text{SiO}_4)_{n-1} \rightarrow (\text{H}_2\text{SiO}_4)_n. \quad [2c]
\end{align*}
\]

The polymeric units have a large surface area, but are small enough (1–100 nm) not to settle under gravity (Bergna et al., 1994; Hearn et al., 2016). Polymerisation of dissolved silica to colloidal silica is irreversible and, depending on the conditions, can proceed further to form either amorphous silica gel or crystalline coagulates. Gelatinous silica is of low density, difficult to separate from the aqueous phase, absorbs the organic and does not compact easily (Littlejohn, 2007).

Factors that influence the form of the silica (whether colloids, gels or precipitates) include the extent of silica supersaturation, temperature, ionic strength and pH (Queneau & Berthold, 1986; Readett & Miller, 1995; Cooper, 1998). Reactivity of silica depends on the zeta potential, defined as the potential at the surface of shear between the electrolyte solution and charged solid. If the solution is acidic (low pH), the silica particles have a low zeta potential and collisions occur rapidly, leading to formation of gel and aggregates. The silica system is at its greatest stability at about pH 2, where it passes through a point of zero zeta charge and there is no driving force for rapid agglomeration (Readett & Miller, 1995; Cooper, 1998). Below pH 2, the rate of polymerization is proportional to the concentration of hydrogen ions (H\(^+\)); above pH 2, it is proportional to the concentration of hydroxide ions (OH\(^-\)). This
characteristic of inverse pH solubility implies that any carryover of silica from the SX extraction circuit to the strip circuit can result in silica precipitation (Miller et al., 1997).

The presence of silica, in all forms, has detrimental effects on the SX process. Colloidal silica can cause slower filtration, poor solution clarity, and excessive flocculent consumption in the upstream circuits. Although the total concentration of silica in the PLS has little detrimental effect, the size distribution of colloidal silica is a critical parameter because emulsion stability increases with decreasing particle size (Readett & Miller, 1995). This influences phase continuity because silica particles are wetted by the aqueous phase—not the organic phase: phase separation is promoted if the solids are wetted by the dispersed phase, but the emulsion is stabilised if solids are wetted by the continuous phase (Readett & Miller, 1995). When colloidal and suspended silica particles are entrained in an emulsion, they stabilise the system and contribute to poor phase disengagement, ‘flipping’ of the phase continuity, carry-over of impurities, high organic-in-aqueous entrainment losses and crud formation (Miller et al., 1997; Musadaidzwa & Tshiningayamwe, 2009; Cole et al., 2016). Agglomeration of colloidal silica is a major contributor to crud formation (Cytec, 2006) and may lead to impurities being deposited and thereby contaminating the cathode during EW.

EXPERIMENTAL

Solvent Extraction Testwork
Initial laboratory studies were carried out using a synthetic PLS comprising 20 g/L Cu (added as CuSO₄·5H₂O) at pH 2 and an organic phase comprising 35 vol.% ACORGA M5774M (Solvay) in BP Mining Solvent. Silica was added as sodium silicate. The silica-mitigating reagent, SilStop, was added as an inorganic salt. All testwork was carried out within 24 h of making up the synthetic electrolyte to ensure that similar aging conditions were employed. Subsequent confirmatory testwork was carried out using PLS provided by Mutanda Mining, DRC. Organic phases comprising 35 vol.% ACORGA 5774M and LIX 984N (BASF) were tested.

The chemical performances of the various systems were evaluated by determining their extraction isotherms. Different relative volumes of PLS and the organic phase were contacted by magnetic stirring for 10 min to achieve equilibrium. The phases were then separated and the copper content of the aqueous phase was determined by complexometric titration using ethylenediamine tetraacetic acid (EDTA) (Bassett et al., 1978). Organic-phase copper concentrations were determined by difference.

The physical performance was evaluated by measuring the rate of phase disengagement under standard conditions (BASF, 2016). Equal volumes (300 mL) of the PLS and organic phase were contacted in a baffled beaker using an overhead stirrer rotating at 1400 rpm for 3 min. The stirrer was then switched off and the rate of phase separation measured using a video recorder.

Surface tension measurements of synthetic and plant aqueous phases in the presence and absence of the silica-mitigation reagent were measured using a Krüss K6 du Nooy platinum-ring tensiometer. Measurements were carried out in triplicate and the average value is reported.

Electrowinning Testwork
The effect of carryover of the silica-mitigation reagent to EW was investigated by comparing the cathode produced under standard laboratory conditions in the absence and presence of the reagent. The synthetic electrolyte contained 38 g/L Cu (as CuSO₄·5H₂O) and 175 g/L H₂SO₄. A 5-L polypropylene cell was employed and the electrolyte was recirculated using a 35-L reservoir. Electrowinning was carried out for a plating cycle of 24 or 48 h at 50°C, using a stainless steel cathode (0.0117 m² plating area) and lead–tin–calcium anode, using a current density of 300 A/m² and electrolyte flowrate through the cell of 2.0 L/m²/min. The deposits were examined by scanning electron microscopy (SEM) in secondary-electron mode to observe the surface morphology. Cross-sections of the deposits were epoxy mounted and analysed for surface roughness.
RESULTS

Effect of Silica and its Mitigating Agent on Copper Extraction Efficiency

Figure 1 shows the effect of increasing silica concentration on the extraction of copper from a 20 g/L Cu synthetic PLS at pH 2 by 35 vol.% ACORGA M5774 in BP Mining Solvent. Initial testwork was carried out using ACORGA M5774 because this modified aldoxime formulation has a high concentration of 2,2,4-trimethyl-1,3-pentanediol diisobutyrate (TXIB) modifier. Such modification is reported to have an adverse effect on physical behaviour when silica is present in solution, so this system was considered to represent a more extreme condition. Although lower concentrations of silica (up to 125 mg/L) depressed extraction slightly, higher concentrations (up to 1000 mg/L) had little effect on the extraction isotherm. It is likely that higher silica concentrations promoted polymerisation to larger particles that are less likely to interfere with the interfacial reaction.

Figure 2 shows the effect of addition of the mitigation agent on the extraction isotherms. A slight depression of the initial portions of the isotherms was observed, but maximum extractant loading remained unchanged, within the experimental error of the measurements.

Experiments were then carried out using an actual plant PLS obtained from a DRC operation that suffers from the effects of silica in the PLS. This contained approximately 15 g/L Cu and 600 mg/L Si. In addition to Acorga M5774, extraction was also carried out using LIX 984N, which is the extractant currently employed on this plant. The extraction isotherms obtained for the PLS as-received and in the presence of 13.3 mmol/L silica-mitigating reagent (a molar ratio of SilStop:Si = 0.6) are shown in Figure 3. The presence of SilStop had no effect on the extraction efficiency of either extractant.
Effect of Silica on Phase-Disengagement Time

Experiments were carried out using both aqueous- and organic-phase continuity. It has become well known on the Copperbelt that the wetting properties of silica are such that a relatively large O:A ratio (2 to 3.5) may be required in the mixer to ensure that the system remains in the preferred organic continuity. Under the standard phase-disengagement test conditions, the presence of silica in the PLS caused the system to continually flip to aqueous continuity: results are reported under these conditions.

Figure 4 shows the effect of silica on the rate of phase separation under standard laboratory test conditions when using the synthetic PLS. It is evident that the rate of phase separation deteriorated with increasing silica content. This effect is in agreement with evidence from plant behaviour.

Using 550 mg/L Si, which is a typical value for DRC PLS concentrations, Figure 5 shows the effect of adding progressively increasing amounts of the mitigation reagent on the phase separation. Addition of SilStop at a SilStop:Si molar ratio of ~1.1 completely mitigated the detrimental effect of silica on the rate of phase disengagement.
Phase disengagement tests were also carried out using real PLS with SilStop added at different molar ratios to the Si present in this solution. The results for LIX 984N are shown in Figure 6: similar results were found for Acorga M5774. A molar ratio of Si:SilStop of 1–2 mitigated the detrimental effect of the silica. Higher Silstop concentrations had no further benefit.

The effect of contact time of the reagent (added as the inorganic salt) with the real PLS was investigated for times ranging from 5 to 60 min. A mixing time of 5 min was found to be adequate for the required effect to be achieved.

**Effect of Silica and Silica-Mitigating Agent on Surface and Interfacial Tension**

SX is an interfacial process, with extraction of copper from the PLS taking place at the aqueous–organic interface. It is well known that the presence of surfactants or other surface-active contaminants in the PLS can affect the kinetics of extraction, as well as retard the rate of phase disengagement. Silica is believed to act in a similar manner in exerting a negative effect on the separation of the two phases. To gain some understanding of the mechanism of action of the silica-mitigating agent, the effects of silica and the reagent on the surface tension of synthetic and plant solutions were evaluated. The results are presented in Table I.
Table I: Effect of silica and silica-mitigating agent on surface tension of PLS.

<table>
<thead>
<tr>
<th>PLS</th>
<th>Additive</th>
<th>Surface tension (mN/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Synthetic PLS</td>
<td>Zero Si</td>
<td>72.9</td>
</tr>
<tr>
<td></td>
<td>250 mg/L Si</td>
<td>72.8</td>
</tr>
<tr>
<td></td>
<td>500 mg/L Si</td>
<td>71.8</td>
</tr>
<tr>
<td></td>
<td>500 mg/L Si + 0.45 mol/mol SilStop:Si</td>
<td>55.7</td>
</tr>
<tr>
<td></td>
<td>500 mg/L Si + 0.90 mol/mol SilStop:Si</td>
<td>61.9</td>
</tr>
<tr>
<td>Plant PLS</td>
<td>As received (~600 mg/L Si)</td>
<td>74.5</td>
</tr>
<tr>
<td></td>
<td>+ 0.38 mol/mol SilStop:Si</td>
<td>55.2</td>
</tr>
</tbody>
</table>

While it is evident that the silica-mitigating reagent acted to modify the surface tension of the aqueous phase, such that deterioration in phase-separation time and other physical characteristics were avoided, further assessment of the mechanism of action remains necessary.

Effect of Silica-Mitigating Agent on Downstream Electrowinning Process

It is anticipated that a concentrated SilStop solution would be added to the PLS stream where it would interact with silica present and counteract its negative effects on the physical characteristics of the SX circuit. Any residual reagent is expected to report to the raffinate; however, it is possible that some reagent could be transferred to the downstream EW circuit so its effects on the morphology of the copper cathode deposit were evaluated.

Using the experimental conditions described above, copper was electrowon from a standard electrolyte composition in the presence of large excesses of the inorganic reagent. Figure 7 shows SEM secondary-electron images of the deposits after plating for 24 and 48 h. There was no detrimental effect of the presence of the silica-mitigating reagent on the deposits; in fact, its presence appeared to offer a grain-refining effect, noted particularly for the 48-h deposits. Figure 8 shows higher magnification views of the 48 h deposits without SilStop and with the higher concentration, confirming the apparent smoothing of the deposit surface in the presence of this reagent.

Surface roughness was quantified for the 48-h specimens by measuring the relative peak height across a defined cross-section of the deposit. Roughness was measured in terms of $Ra$, defined as the arithmetical mean roughness measured from all profile values. The results are shown in Figure 9 and Table II. The significant improvement in surface smoothness by the grain-refining action of the additive is evident.

Table II: Effect of SilStop on surface roughness of copper deposited for 48 h at 300 A/m².

<table>
<thead>
<tr>
<th>Electrolyte additive</th>
<th>Surface roughness $Ra$ (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zero SilStop</td>
<td>61.4</td>
</tr>
<tr>
<td>0.024 mol/L SilStop</td>
<td>45.2</td>
</tr>
<tr>
<td>0.048 mol/L SilStop</td>
<td>46.1</td>
</tr>
</tbody>
</table>

These results indicate that any accidental contamination of the silica-mitigating reagent in the copper EW circuit will not have any detrimental effect on the quality or morphology of the cathode product, and may, in fact, actually improve the smoothness of the deposit.
Figure 7: Scanning electron microscopy secondary electron images of copper deposited for (a)–(c) 24 h and (d)–(f) 48 h in the presence of (a), (d) zero SilStop, (b), (e) 0.024 mol/L SilStop, and (c), (f) 0.048 mol/L SilStop measured at a magnification of 100×.
Figure 8: Scanning electron microscopy secondary electron images of copper deposited for 48 h in the presence of (a) zero SilStop and (b) 0.048 mol/L SilStop measured at a magnification of 250×.

Figure 9: Surface roughness analysis of cross-sections of copper deposited for 48 h in the presence of (a) zero SilStop, (b) 0.024 mol/L SilStop, and (c) 0.048 mol/L SilStop.

DISCUSSION

The results above show that the detrimental effects of silica on phase disengagement in copper SX can be mitigated by addition of an inexpensive inorganic reagent, SilStop, added at a molar ratio of Si:SilStop of approximately 1. The reagent appears to have no negative impact on the extraction chemistry or loading capacity of the extractant; furthermore, there is no detrimental effect on the downstream EW process.

The role of silica in crud formation is well known. It is, however, very difficult to assess crud formation under batch laboratory conditions, particularly when solution volumes are small. The effect of SilStop on crud formation in continuous operation is still required to be evaluated.

The mechanism of action is proposed to be the formation of a soluble complex between silica in solution and the reagent. The results of Table 1 indicate that the surface tension is reduced by the addition of the reagent in the presence of silica, therefore effectively neutralising its effect. This proposal remains conjecture at this stage and further work is required to fully understand the
behaviour of the reagent in these systems. Silica effects are also very dependent on aging and solution composition, so the optimum dosage may need to be determined for individual operations.

**Indicative Cost of Implementation**

It is proposed that SilStop will be added in the form of a solution (to avoid contamination of the PLS by solids) directly into the PLS from a make-up tank.

The cost for a fully managed, maintained and operated dilution and dosage plant capable of treating a PLS containing 15 g/L Cu, 600 mg/L Si for a DRC copper operation is estimated at USD 12-20/t Cu.

The benefits of SilStop addition accrue from mitigation of the deleterious effects of silica in the circuit. Directly quantifiable benefits are the reductions in extractant and diluent consumptions owing to reduced entrainment losses of organic phase to the raffinate and reduced crud formation. These savings are estimated at about USD 26/t Cu. Other benefits include improved operability of the SX circuit, the ability to operate at lower mixer O:A ratios in the SX extraction stages, reduced tendency for the flipping of phases and reduced downtime from events like silica coating of anodes and crud cleanout of settlers.

**CONCLUSIONS AND RECOMMENDATIONS**

Laboratory experiments using both synthetic and actual plant leach liquors have shown that the presence of silica is detrimental to phase separation in copper SX circuits. This physical effect can be chemically mitigated by the addition of an inexpensive inorganic salt to the PLS. The dosage required is approximately a 1:1 molar ratio relative to the Si in the aqueous phase. This reagent can restore the phase separation behaviour close to that observed in the absence of silica. The reagent has no adverse effect on the morphology of copper deposited in the downstream EW circuit.

Because of the time-dependent characteristics of silica in aqueous solution and its dependence on acidity, temperature and other factors with respect to the nature of its species, it is necessary to repeat this work on live plant solutions. Opportunities are sought for a plant trial at an operation that suffers from the effects of high levels of silica in their leach solutions.

**ACKNOWLEDGEMENTS**

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**REFERENCES**


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The papers in this volume have been for the most part prepared from Word documents supplied by the authors, with additional typesetting and formatting by The Southern African Institute of Mining and Metallurgy.
Foreword

The African Copperbelt has experienced an enormous resurgence of activity since 2008, following decades of political and economic instability. Copper production in Democratic Republic of Congo (DRC), Africa’s top producer and sixth-largest in the world, rose 6.9% in 2017 to 1.09 million tonnes, while output of cobalt surged by 15.5% to 73 940 tonnes. Zambia, as the seventh-largest global producer, produced some 800 000 tonnes of copper during the same period. Today, global interest in cobalt, particularly for use in lithium-ion batteries that are an essential component of the exploding electric vehicle market, as well as in consumer electronics and solar energy applications, is firmly focused on DRC, which produces almost 60% of the world’s supply. The Copperbelt region currently commands an impressive proportion of capital spending, project development, operational expansions, and metal value production in the Southern African mining industry, particularly with respect to cobalt initiatives, in both DRC and Zambia.

The geology and mineralogy of the ores are significantly different from those in other major copper-producing regions of the world. Both mining and metallurgy present some unique difficulties, not only technically, but also with respect to logistics and supply chain issues, human capital, community engagement, geopolitics and legislation. This conference provides a forum for discussion of all these topics, spanning the value chain from exploration and projects, through mining to processing.

Interest in and support for this conference has been strong, consolidating the perception that this region is increasingly recognised as a global leader in both primary copper and cobalt production and technologies. The papers in these Proceedings represent authors from six continents and sixteen countries: many more nationalities are represented on the delegate list. This conference offers an ideal opportunity to gain in-depth knowledge of and exposure to the Southern African copper and cobalt industry, and to understand better the various facets of mining and processing in this part of the world. We are encouraged by the participation of many young and emerging professionals from the Copperbelt region, who are the future custodians of this industry.

On behalf of the Organising Committee, I extend thanks to all authors and presenters for their contributions, to the members of the Technical Review Panel, who gave so generously of their time and expertise in ensuring that the published papers meet a high standard, and to the Session Chairs, who make everything happen seamlessly on the day. We are also grateful for the tremendous support of our sponsors, without whom this event could not happen. I also wish to convey a special word of appreciation to Camielah Jardine and her excellent team at SAIMM for their endless patience, good humour and tireless efforts in organizing this conference.

We trust that these few days will be stimulating and enjoyable, allowing you to make new acquaintances and renew old friendships, while improving your understanding of new and existing technologies, as well as the business concerns and imperatives driving this industry in these challenging and exciting times.

On behalf of the Organising Committee, I have great pleasure in welcoming you to the Second Copper Cobalt Africa Conference at this spectacular venue in the heart of Africa.

Kathryn C. Sole (FEng, PhD, FSAIMM)
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