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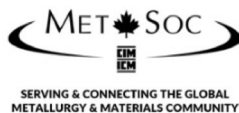
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ReAK- Reduction of arsenic in copper concentrates

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Abstract

ReAK (Reduction of arsenic in copper concentrates) is a binational project between Chilean and German partners. The overall objective of the research is to expand the existing processes of copper production in such a way that the increasing arsenic concentrations in the upcoming ores can be processed in a cost-effective and environmentally friendly manner, such that the excess in arsenic can be immobilized and disposed safely.

The project deals with different process steps along the copper production chain. For example, research is being focused on arsenic-selective flotation so that an arsenic-poor and an arsenic-rich fraction are produced right at the start of the process chain. The same goal is being pursued in the development of a sensor-supported separation of arsenic-rich ore fractions. This arsenic-rich fractions are now to be further processed by advanced roasting processes, as well as by leaching processes (sulfidic or microbiological).

In currently existing copper processes, the arsenic is loaded as arsenite in a gas scrubber. The project is researching new ways to oxidize that arsenite to arsenate, which can be precipitated as a less soluble compound and would therefore be more environmentally friendly to landfill.

Finally, possible landfill routes will be researched and the immobilization and mobilization processes of the landfilled arsenic will be investigated.

1. Introduction

Arsenic oxide is one of the most famous poisons of the Middle Ages and the Renaissance. Napoleon, for example, was poisoned with arsenic, which could only be proven by modern analysis using a hair analysis. (Süss-Fink, 2012)

Due to its extremely toxic properties, arsenic is nowadays becoming more and more of a problem for the copper industry (Gilsbach & Dorner, 2020). Copper has always been an essential part of almost all technological progress and is therefore an indispensable part of the industrial and everyday surroundings.

The arsenic already enters the copper extraction process with the ore. In many mines, including those in Chile, the mining process leads to deeper ore layers, which contain more sulphide minerals than nearer-surface layers of ore. These sulfidic ores are rich in Copper but also have high contents of arsenic, which then has to be separated in the course of the process chain and subsequently deposited. (Gilsbach & Dorner, 2020)

The ReAK project deals in its entirety with different process steps in copper production and focuses on the most environmentally friendly ways of separating and depositing the arsenic residues. In the course of arsenic separation from the copper concentrate, the arsenic enters a sulphuric acid solution as arsenite via the gas phase. The state of the art is now to oxidise the trivalent arsenic to arsenate (pentavalent) by means of hydrogen peroxide and to precipitate it as a calcium compound. Alternatively, due to the high cost of hydrogen peroxide, the arsenite is precipitated as calcium arsenite without an oxidation step. The latter form in particular is comparatively soluble in water, which is why the arsenic can be flushed out of the landfills and thus enter the environment. (Nazari, Radzinski, & Ghahreman, 2016)

2. Project Overview

Following the copper production chain, the project investigates new ideas of arsenic detection, separation and treatment to find economically feasible and environmentally friendly solutions for a sustainable copper production in Chile. Figure 1 gives an overview about the work packages of the project.

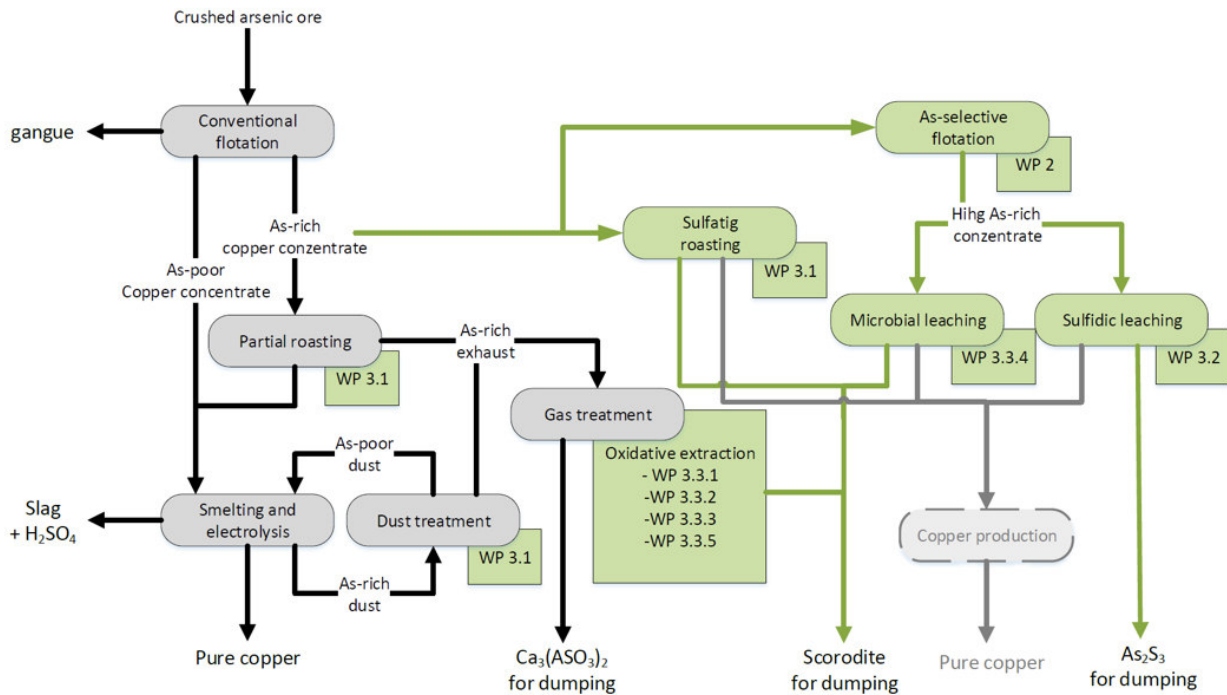


Figure 1: Schematic representation of the process steps investigated within the project ReAK (green) following the current process chain of copper production.

WP 1 - Systematic characterization

Following this task, work package 1 (WP1) is dealing with a systematic characterization of different arsenic containing Chilean copper ores. The mineral chemistry of all arsenic and recyclable material carriers is investigated, taking into account the particle size distribution and the degree of adhesion. Two objectives are pursued: One goal is to develop strategies for a possible pre-separation of arsenic-containing minerals through detailed knowledge of mineral characteristics. Particular attention must be paid to the particle size distribution and degree of adhesion of the ores and concentrates.

Another goal of the work package is to investigate to which extent the ores can be mapped online on the conveyor belt with different sensors like XRF-, LIBS-, and Hyperspectral sensors. The idea is to test which of these methods, or a combination of them could give a meaningful picture of the composition of the incoming ore. This information can be very valuable for the following processing and could lead to higher efficiency not only in the treatment of the arsenic but also in the copper yield.

To follow the arsenic through the different treatment steps of the project, WP1 is also responsible for the characterization of the in- and output materials of the following WPs.

WP 2 - Selective flotation

Separation of copper arsenic sulfides (e.g., enargite ($\text{Cu}_2\text{CuAsS}_4$) and tennantite ($\text{Cu}_{12}[\text{S}(\text{AsS}_3)_4]$) and other copper sulfides (such as chalcopyrite (CuFeS_2), chalcocite (Cu_2S), and bornite (Cu_5FeS_4)) is particularly difficult because they have similar surface properties (Fornasiero, 2001) (Long, 2014) (Plackowski, 2012). However, the oxidation rate of minerals varies and this suggests that surfaces with different chemical properties can be produced by controlling the degree of oxidation (Fornasiero, 2001) (Long, 2012) (Plackowski, 2014). Even knowing that arsenic is a penalty element requiring special treatment in copper production, there are only few descriptions in the literature of processes separating copper sulfides into arsenic-rich and arsenic-poor products.

The project will explore how control of the electrochemical environment during the flotation process can improve the separation of arsenic-containing copper sulfides from copper- and iron-containing sulfides in complex copper ores. This will involve careful control of both the solution redox potential (achieved by chemical reagents) and the degree of galvanic interactions (possibly achieved by a selection of grinding media) to investigate whether this provides improved flotation selectivity. Therefore, a series of biopolymer pressers (provided by Borregaard Germany) that show selective depression of pyrite in the separation of chalcopyrite and pyrite (Mu, 2018) will be tested. This aspect of biopolymer depressants adds an additional variable to control selectivity in the separation sought in this project.

Another variable could be the testing of different pretreatments: For example, electrohydraulic fragmentation (EHF) will be used to selectively separate the copper-rich components in the form of chalcopyrite (CuFeS_2) and enargite (Cu_3AsS_4) from the siliceous residual rock at the phase transitions in order to make the subsequent flotation more efficient.

WP 3 - Treatment of arsenic-rich copper concentrates and dusts

Roasting

If the produced copper concentrate contains 2-3 wt.% arsenic, which is quite typical for Chile (Charitos, 2016) (Hedström, 2016), this leads to problems when importing it into other countries such as Japan or China, which have introduced limits of between 0.1 wt.% and 0.5 wt.% As. A similar situation is still expected for other countries (WoodMac, 2013) (Ecometales, 2015) (Beisheim, 2017). So far, the problem of arsenic-rich copper concentrates has been solved in a makeshift way by mixing arsenic-rich and arsenic-poor concentrates. However, this is becoming increasingly difficult today (Ecometales, 2015). A solution already applied to remove arsenic from the concentrate is the so-called roasting. This term generally stands for three pyrometallurgical technologies that, in combination with gas purification and wastewater treatment steps, offer complete solutions to remove arsenic from the concentrates and convert it into a stable form without generating significant As emissions (Charitos, 2017a) (Charitos, 2017b) (van der Meer, 2014). These roasting technologies are partial roasting for arsenic-rich copper concentrates (Charitos, 2016), (Hedström, 2016), roasting of

arsenic-rich copper smelter dusts (Beisheim, 2017) (Güntner, 2016), and sulfating roasting (Güntner, 2011) for arsenic-rich copper concentrates (in combination with existing oxide ore-processing copper smelters) (Chambers, 2012). However, these three technologies are at different technology readiness levels.

In the field of partial roasting, the work of the project partner Metso:Outotec focuses on the investigation of reaction kinetics. The aim is to design optimal reactor operating windows through a better understanding of the influence of temperature and sintering potential in order to push the residual arsenic content in the purified concentrate to below 0.2 wt.%. The behavior of antimony (Sb) is also of particular interest since Sb is an undesirable contaminant in copper smelters.

In the area of roasting of arsenic-rich copper smelter dusts, Metso:Outotec will conduct experiments with As-containing smelter dusts. This issue is similar to that of partial roasting, i.e. the conditions that allow maximum arsenic removal will be determined experimentally. Since here the arsenic is mainly present as arsenate, in contrast to partial roasting of arsenic-rich copper concentrates, it is of particular interest to investigate the type and amount of a reducing agent (e.g. coal) for arsenic removal from the dust.

Metso:Outotec will also conduct tests with arsenic-rich concentrates in the field of sulfating roasting. In this branch of the process, it is of particular interest to experimentally determine the percentage of arsenic that is bound in the roasting product. This will allow modeling of the process as well as design of subsequent gas cleaning and leaching. Since it is particularly important to understand the dependence of copper recovery during leaching on the experimental conditions of roasting, Metso:Outotec will also perform leaching tests on this.

The obtained results will be used to produce new/optimized block diagrams and process flowsheets, which will also include the main mass flows. In the end, a complete picture of the function, economics and potential implementation of this technology including the resulting arsenic balance of a conventional copper smelter will be generated.

Sulfidic leaching

Another approach to remove arsenic from arsenic-rich concentrates, in which it is usually also present in sulfidic form, is by means of sulfidic leaching. A subsequent stabilization in the form of arsenic sulfide (As_2S_3 , auripigment) is quite possible, as this is the most stable naturally occurring mineral of arsenic, which would make safe landfilling feasible.

Preliminary investigations have shown that it is quite possible to remove 50 - 70 % of the arsenic from the concentrate per run. In subsequent steps, the arsenic is precipitated in the form of As_2S_3 , which is ready for landfill after washing and drying. The leaching solution can then be partially recirculated after regeneration, thus saving important resources and minimizing pollution in the water sector. Since the planned process does

not involve oxidizing processes, other components in the concentrate are not changed either, which may well have a considerable influence on the further processing.

Within the project, the influence of the mineralogical composition, the bonding form and the grain size of the concentrate on the leachability will be investigated. This will be done first with synthetic arsenic compounds and later with real concentrates. The degree of leaching of bismuth and antimony will also be investigated, since these are partially co-leached. In the leaching tests on real concentrates, the maximum possible leaching rate will be determined and the influence of secondary components, such as sulfide-forming metals, will be investigated. Resulting polysulfides can best be determined photometrically due to the intense and specific coloration. In addition to the composition of the concentrates, the leaching conditions (pH, temperature, concentrations and leaching duration) also play a decisive role and must therefore be optimized.

After precipitation, the arsenic sulfide is washed and subjected to initial leaching tests, both on fresh and aged samples. To increase stability, thermal post-treatment of the precipitated material will be tested.

Oxidative Extraction

The transformation of arsenic(III) to arsenic(V) with subsequent precipitation as scorodite is called oxidative extraction. Different processes can be used for this purpose. In the ReAK project, so-called advanced oxidation processes (AOP) are investigated, including UV-assisted oxidation, ozone-based oxidation and electrochemical oxidation. Alternatively, microbial oxidation is also being investigated. The idea here is that it should at least partially replace the current, but very cost-intensive process in which hydrogen peroxide is used as an oxidant.

One way of minimizing the amount of hydrogen peroxide required for the oxidation of arsenite is **UV-assisted** processes. Irradiation with UV light can split hydrogen peroxide molecules. Highly reactive hydroxyl radicals (redox potential of 2.73 V) are formed from the comparatively weakly oxidizing hydrogen peroxide (redox potential of 1.77 V). This should reduce the amount of hydrogen peroxide required. The extent to which this can reduce treatment costs of copper mining will be investigated as part of this project.

Various UV-assisted processes are being tested in laboratory-scale trials. The efficiency of irradiating the hydrogen peroxide with conventional low- and medium-pressure mercury lamps as well as with mercury and excimer lamps emitting in the vacuum UV range will be tested. With these light sources emitting in the vacuum UV range, it is expected that the use of hydrogen peroxide is no longer needed, since the hydroxyl radicals can be formed from the splitting of water.

Similar to UV-assisted oxidations, hydroxyl radicals can be formed by various process combinations **with ozone**. Ozone itself can trigger a radical chain reaction in the alkaline pH range, which leads to the formation of these radicals. A combination of ozone with ultrasound (sonolysis) or with hydrogen peroxide is also possible. These three process combinations will be investigated with regard to their suitability for the treatment of arsenic-containing wastewater after roasting. Again, several series of experiments will be carried out on each process, varying the reaction conditions (pH, ozone dose, ozone input system, reaction time). The efficiency of the treatment will also be investigated again. Here, the project partner Enviro Chemie will carry out the first tests with synthetic wash water. The test results will subsequently be validated with real water samples. Since the introduction of ozone into the water plays a significant role in the efficiency of the oxidation, various aggregates for the introduction of ozone will also be tested. In addition to conventional air injection elements such as aeration bodies or injectors, a novel injection system will be tested, which is able to inject gases into the water in an energy-efficient and pressureless manner by means of rotational movement.

To obtain further information about the results of the work package “**electrochemical Oxidation**” please check the Paper of Anna-Lisa Bachmann “ReAK – Reduction of arsenic in copper concentrates - Electrochemical oxidation”, which is also published within the scope of this conference.

At the Institute of Biosciences (IBW) of the TU Bergakademie Freiberg there is a mixed culture of bacteria leaching cobalt arsenide (safflorite, CoAs_2) at pH 2 (“BiCoNi4”), which will be tested to see whether it also **oxidizes arsenite to arsenate microbial**. For this purpose, secondary mineral formation will first be investigated. In addition, metagenome sequencing is to be performed to provide information on the complexity of the composition and adaptations of the mixed culture to the arsenic-containing environment.

Pure cultures are to be isolated from the mixed culture and tested for their leaching behavior and tolerance against arsenite. Genome analysis of the most promising strains will reveal possible adaptations of the strains to high arsenic concentrations. Comparative transcriptome analyses with an isolated strain of the mixed culture in the presence and absence of cobalt arsenide and arsenite, respectively, will be used to investigate which genes are induced in the presence of arsenic and lead to its tolerance (or possibly to the conversion of arsenite to arsenate).

If an arsenite oxidation gene is found in the BiCoNi4 mixed culture and not in the pure cultures, then the isolation of the pure cultures should be adjusted in a way that, if possible, the necessary gene can be detected in new isolates by means of genome or transcriptome analysis.

In addition to the BiCoNi4 mixed culture, other acidophilic arsenite oxidizers should be searched for. For this purpose, samples should be obtained from acidic sites, preferably

containing arsenic, e.g., tailings with arsenic-bearing minerals, mine waters, or even from the Chilean Altiplano. The strains isolated from the new mixed cultures are to be physiologically characterized and examined for their genome sequence, always with respect to arsenite oxidation genes.

Looking for scorodite, a secondary mineral investigation should be performed after arsenite oxidation with the pure cultures obtained and, if necessary, with moderately thermophilic sulfobacilli.

With the strain (either from the BiCoNi4 culture, with a Sufobacillus or an isolate from further samples) that seems most promising with regard to scorodite formation at the lowest possible temperatures, a continuous system for arsenite oxidation in the bioreactor is to be established on a laboratory scale. A stable continuous process is mandatory for a biological process for arsenite oxidation in order to be considered for an industrial application.

Microbial leaching

So far, there have been practically only moderately successful experiments for leaching enargite with *Acidithiobacillus ferrooxidans* at 25°C - 35°C and more successful ones with *Sulfolobus* or *Acidianus* strains at 70°C or 80°C. Since archaea are considered difficult to handle, because reaching and maintaining such high temperatures can be laborious, and since scorodite can be formed as a product even at 45°C, the strategy is to attempt enargite leaching primarily with bacteria such as sulfobacilli or leptospirilli in the range between 40° and 60°C.

In addition to the experiments with already known strains, the project will also isolate new strains, whose enrichment under different Fe²⁺ concentrations and with as pure enargite as possible, at temperatures of 40-60°C will be investigated. Inoculation material will be taken from running bioleaching plants (if possible from areas where the stockpile has warmed up) from very arsenic acidic sites in Chile. Among other things, pure enargite surfaces will be exposed to such waters and the bacteria adhering to enargite will be transferred to culture media after a few days. Isolates will be obtained from the enrichment cultures and it will be verified at different parameters whether the new isolates actually leach enargite.

WP 4 - Investigation of immobilization and mobilization processes of arsenic

In this work package, the mobility of arsenic in the different residues will be investigated by the project partner Wismut GmbH. The arsenic elution of the residues will be investigated by means of reference methods to determine the classification criteria applicable in Germany according to the Landfill Ordinance (LAGA, 2012).

The tests are then evaluated according to (EPA, 1995) and (Düwel, 1991). Mechanical stability indices and limiting abrasion velocities are determined. The classification of

the heavy precipitation erosion tendency is carried out by determining additional soil-specific input parameters (water content, bedding density, roughness, erosion resistance and sink factor) from EROSION 3D (Schmidt, 1996) and comparing them using the parameter catalog (Michael, 1996).

The residues will be installed in percolation columns and examined for their elution behavior under site conditions following the test procedures CEN TS 14405 (method for percolation testing of granular waste not validated according to LAGA), DIN 19528 (column test certified according to LAGA for inorganic substance fractions) and EPA method 1314 (determination of the solid/liquid distribution of inorganic substances in granulated material as a function of the solid/liquid ratio under percolation conditions). The experimental conditions are modified to represent best- and worst-case scenarios.

To estimate wind erosivity under site conditions, data on wind speeds are researched and used together with the determined solids characteristics using (EPA, 1993) and (Düwel, 1991) to calculate removal rates. Because of their influence on the wind-exposed surfaces, different shapes of the depositional body are also considered. The calculations are performed in parallel for deposit sites of Wismut GmbH, where a comparison with actual dust measurements can be made, and for artificially aged residues, which are altered in the same way as for the percolation tests and examined for grain size distribution and compressive strength.

The calculation of the heavy precipitation erosion is carried out with the erosion model EROSION 3D with additional use of heavy precipitation and terrain model data (consideration of the shape of the deposit body) and a correspondingly high model resolution. In addition to erosion, the deposition of the material is also of interest, since the deposition surfaces should be designed in such a way that the deposition can take place within the secured area.

Furthermore, possibilities of residue stabilization are also worked out and shown by the partner Wismut GmbH.

Finally, an evaluation of the test results for the different investigated (stabilized) precipitation products will indicate expected elution - and erosion material flows for best- and worst-case scenarios. According to the expected emissions, technical designs of possible deposition sites are shown in each case, which are suitable for minimizing the emissions. In each case, the classification of the deposition scenarios according to German/European guidelines is also given.

To upscale the obtained results, an on-site pilot test site will be designed to test the monitoring of long-term residue storage on a pilot scale. The technical design and operation of the pilot site will be conceived in such a way that the best possible emission reduction, loading operations as well as the metrological recording of rainwater surface runoff, leachate runoff and dust emission can be realized.

WP 5 - Disposal concept

In this work package, the current status of the landfilling of arsenic-rich residues from the copper industry in Chile and the regulations to be expected in the future from the Chilean authorities, will be discussed. Furthermore, the current status of environmental regulations, landfilling and the best available technologies for the landfilling of arsenic-containing residues in Europe (Germany) will be determined. From these findings and the results of this project, it can then be deduced to what extent the available technologies in Europe (Germany) are transferable to Chile and whether these are sufficient to comply with future limitations. With the previously determined data, a disposal concept with possible recommendations for handling the resulting waste will be derived.

This work package will be carried out jointly by the Fraunhofer IWKS and the PUC by awarding a suitable student project.

WP 6 – Life Cycle Assessment (LCA)/ Live Cycle Costing (LCC)

The development and optimization of new industrial processes - in this case processes for handling arsenic-rich copper ores and their concentrates - is usually associated with undesirable side effects that can impair and damage the environment through emissions and resource consumption. In this context, investment and operating costs are decisive for the competitiveness of the process. For this reason, a summarized ecological and economic assessment has to be made, analyzing the opportunities and risks of the investigated process with regard to competing processes. Strategies for future processes in dealing with arsenic-rich copper ores are to be derived from the findings obtained, and a roadmap for next steps is to be developed.

The aim of this work package is to create a basis for decision-making for significant players within the copper industry by recording the existing material flows and using the system analytical methods LCA and LCC, and to close crucial gaps in life cycle analysis. The process simulations will capture the ecological and economic quantification of all relevant processes along the entire value chain, from flotation (WP 2) to disposal (WP 5).

At the beginning of the project, the ecological and economic potentials of the processes will be estimated based on literature data and challenges will be identified at an early stage. Subsequently, the processes will be investigated with primary data collected in the project and evaluated according to ILCD Handbook (ILCD, 2010). For the collection of real data in the field, the Pontificia Universidad Católica de Chile (PUC) will coordinate and support the work package. These data will be modeled in openLCA software. Results will be evaluated and critically discussed with all partners. Due to the early and inconsistent development stages of the different process chains, a comparison among them would be misleading. This is therefore explicitly not the aim of this work package.

3. Conclusions

The here outlined Project “ReAK- Reduction of arsenic in copper concentrates” has the aim to find new ways for the save, clean and economically feasible treatment of arsenic from arsenic-rich copper ores. Copper is and will be one of the most needed metals on earth for several applications. Hence the global demand will definitely further increase. Even if the problem seems to be localized in Chile, it is quite sure that other copper concentrate producing countries will sooner or later have to face the same challenges. In this context, this project can help Chile becoming a pioneer in the development of technologies for an ongoing save and clean global copper production.

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