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Removal of Sb Impurities in Copper Electrolyte and Evaluation of As and Fe Species in an Electrorefining Plant

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Abstract: Antimony and arsenic concentrations and their oxidation states (Sb(III), Sb(V), As(III) and As(V)) in copper electrorefining electrolyte can affect copper cathode quality through the formation of floating slimes. A laboratory-scale pilot plant was operated to remove Sb from commercial electrolyte. The pilot plant consisted of a pre-treatment process with copper shavings followed by ion exchange. The results indicated that Sb(III) was removed from copper electrolyte completely, while Sb(V) was partially eliminated. The concentrations of As(III) and As(V) were not affected, and the poisoning of the ion exchange resin by Fe(III) was avoided by pre-reduction to Fe(II) by copper shavings. The operation configuration of the pilot plant was applied to the design of an industrial plant for Sb/Bi removal at the Atlantic Copper Refinery in Huelva, Spain. The evolution of Sb, Fe and As species in the commercial electrolyte was monitored prior to and after the installation of the Sb/Bi removal plant. The results show a ca. 45% decrease in total Sb content (from 0.29 g L^{-1} to 0.16 g L^{-1}) in the electrolyte. This reduction is more noticeable for Sb(III), whose concentration decreased from 0.18 g L^{-1} to 0.09 g L^{-1}, whereas Sb(V) concentration diminished from 0.11 g L^{-1} to 0.07 g L^{-1}. The resin also retained ca. 75% of the Bi content (0.15–0.22 g L^{-1}). The total As increased during the study period (from 7.7 to 9.0 g L^{-1}) due to changes in plant inputs. Arsenic was predominantly As(V) (ca. 93–95%). The total Fe concentration experienced little variation (0.9–1.1 g L^{-1}) with Fe(II) being the main species (ca. 94–96%).

Keywords: copper electrolyte; antimony; arsenic; iron; removal; species; ion exchange

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1. Introduction

During the copper electrorefining process, anode impurities, most frequently As, Bi, Fe and Sb, are dissolved with copper from the anode into the electrolyte [1,2]. These impurities can accumulate in the electrolyte or form different types of anode slimes which can adhere to the anode, deposit on the bottom of the electrolytic cell or float within the electrolyte [3–6].

The redox species of arsenic and antimony have a great influence on the formation of the insoluble precipitates which could affect copper cathode quality. As(V) and Sb(V) can form a series of arsenato antimonic acids (AAAc), which can further react with As(III), Sb(III), and Bi(III) to form arsenato antimonates [1,7]. As(V), Sb(III) and Bi(III) can form arsenates [8] and Sb(V) plays a substantial role in the formation of floating slimes [9–11], which are amorphous and chemically undefined compounds that may contain Sb(III), Sb(V), Bi(III), As(V) and As(III) [12,13]. Floating slimes are commonly avoided by controlling the total antimony concentration in the electrolyte below 0.5 g L^{-1} [13] by maintaining the concentration of arsenic in the electrolyte above 6–7 g L^{-1} and an As/(Sb+Bi) molar ratio above 1.5–2 in the anodes [14].

Other techniques have been proposed to control antimony concentrations in copper electrolytes, including precipitation [15], adsorption [16,17] and ion exchange [18]. Ion exchange resins are used in several copper refineries [19,20] to remove Sb and Bi and maintain their concentrations below 0.50 g L^{-1} in commercial electrolytes [21,22], although the concentrations of these elements can increase due to the high impurities content in copper anodes [23]. The resins employed are usually chelating, a subgroup of ion exchange resins with coordination compounds that can establish selective covalent-like bonds between the central ion of the functional group of the resin and donor ions in solution [24].

In the literature, there are several studies on the removal of Sb from copper electrolyte [25,26], but there has been no discussion about the elimination and distribution of Sb by oxidation state. According to Riveros, 2008 [18], the difficult elution of Sb from ion exchange resins is achieved with concentrated HCl, where Sb(III) was easily eluted and Sb(V) elution was extremely slow resulting in Sb build-up and a decrease in resin life.

In addition to the Sb species, it is important to know the oxidation states of Fe during the ion exchange treatment of the electrolyte. This is of main importance for Fe(III), as some studies carried out with synthetic copper electrolytes have shown that the presence of Fe(III) (but not Fe(II)) affects the retention capacities of Sb and Bi species on aminophosphonic resins. These studies refer to the "poisoning" of sulphonated phosphonic and aminophosphonic resins with the Fe(III) ion [27,28].

The objective of this paper is to document the oxidation states of As, Sb and Fe in copper electrorefining electrolyte during a laboratory-scale pilot plant demonstration of a Sb/Bi removal process by ion exchange using an aminophosphonic resin, and after the process was implemented on an industrial scale. In addition to Sb, Fe and As species were monitored to evaluate the pre-treatment of the electrolyte prior to ion exchange.

2. Materials and Methods

2.1. Laboratory-Scale and Industrial Sb/Bi Removal Plants

The previous laboratory-scale operation to remove Sb and Bi from the copper electrolyte was described by F. Arroyo-Torralvo et al. (2017) [23]. The experimental configuration is a two-step process in which the electrolyte is pumped through two columns in series. The first column was filled with copper shavings. The copper shavings were used to promote the reduction of Fe(III) to Fe(II), and thus avoid the possible poisoning of the ion exchange resin with Fe(III). The second column was filled with an ion exchange resin with chelating aminomethylphosphonic acid groups (Lanxess Lewatit MonoPlus TP 260, Lenntech, Delft, Holland), that removes Sb and Bi from the electrolyte. The volume of the resin bed used in the tests was 100 mL which occupied a column height of 35 cm. The operating configuration involves the conditioning of the resin with H_2SO_4, loading of the electrolyte, electrolyte displacement and backwash with H_2SO_4, regeneration of the resin with HCl + thiourea, displacement of HCl with H_2SO_4 at different pH (2, 4 and 6) and conditioning of the resin with recirculated H_2SO_4 from a previous stage.

The laboratory-scale plant allowed the calculation of ion exchange resin capacity and the evaluation of operating protocol (load-elution-regeneration) [23]. It was used as a model for the design of the industrial Sb/Bi removal plant. The Sb/Bi removal plant was installed and connected to the electrorefining circuit within the Atlantic Copper Metallurgical Complex (Huelva, Spain) in 2018. The principal chemical components of the copper electrolyte were 45 g L^{-1} Cu, 12 g L^{-1} Ni and 180 g L^{-1} H_2SO_4. The main impurities present at high concentrations are ca. 9 g L^{-1} As, 1 g L^{-1} Fe, 0.30 g L^{-1} Sb. The concentration of Bi during the operation of the laboratory-scale plant was 0.15–0.18 g L^{-1} Bi, and 0.22 g L^{-1} during the operation of the industrial Sb/Bi removal plant.

2.2. Analytical Techniques

The Sb and As redox species were determined by high-performance liquid chromatography (HPLC) coupled to hydride generation-atomic fluorescence spectrometry (HG-AFS). The instrumentation consisted of a quaternary pump (JASCO PU-2080 Plus, Hachioji, Tokyo, Japan) fitted with an ion-exchange column (Hamilton, Reno, NV, USA) coupled to an AFS spectrometer (PS Analytical Millennium Excalibur 10.055, Orpington, UK) equipped with boosted discharge hollow cathode lamps for Sb or As (Photron, Victoria, Australia). A detailed summary of the analytical parameters for the determination of As(III), As(V), Sb(III) and Sb(V) in copper electrolyte has been previously described [29].

For determination of Fe(II), the absorbance of the red-orange Fe-phenanthroline complex formed was measured using a SPECTRONIC 200 spectrophotometer (Thermo Scientific, Waltham, MA, USA). The wavelength of 510 nm was selected, which corresponds to the maximum absorption of the complex spectrum. The total Fe content (sum of Fe(II) + Fe(III)) was calculated by adding hydroxylamine hydrochloride to the samples, to reduce Fe(III) to Fe(II), before the addition of o-phenanthroline. Fe(III) was determined indirectly by the difference between the total Fe content and the Fe(II) content [30].

3. Results and Discussion

3.1. Removal of Sb Species from Copper Electrolyte by Ion Exchange Resin

3.1.1. Laboratory-Scale Plant

The oxidation states of Sb and Fe in copper electrolytes were determined during the operation of the laboratory scale pilot plant. The treatment consisted of pumping the electrolyte through a column with copper shavings and then through a second column with Lewatit TP 260 ion exchange resin. The overall time of the process (loading of the electrolyte and posterior regeneration stage of the resin) was adjusted to a working day (8–12 h). Additionally, the removal efficiency of bismuth from the electrolyte with this resin has been estimated at 75% [23]. Table 1 shows the average concentration of antimony and iron species in the initial electrolyte, the electrolyte treated only with copper shavings, and the electrolyte treated both with copper shavings and the ion exchange resin.

Table 1. Average concentrations (g L^{-1}) of Sb and Fe species in samples of initial electrolyte, treated electrolyte with Cu shavings and treated electrolyte with Cu shavings and ion exchange resin in the laboratory-scale plant. The results of the concentrations are expressed as mean ± standard deviation ($n = 14$).

	Initial Electrolyte	**Electrolyte + Cu Shavings**	**Electrolyte + Cu Shavings + Ion Exchange Resin**
	mean ± sd	mean ± sd	mean ± sd
Sb(V)	0.12 ± 0.02	0.13 ± 0.02	0.07 ± 0.01
Sb(III)	0.20 ± 0.04	0.19 ± 0.07	0.01 ± 0.01
Total Sb	0.32 ± 0.06	0.32 ± 0.08	0.08 ± 0.03
Fe(II)	0.90 ± 0.07	0.92 ± 0.07	0.93 ± 0.07
Fe(III)	0.08 ± 0.04	0.05 ± 0.03	0.05 ± 0.04
Total Fe	0.98 ± 0.06	0.97 ± 0.08	0.98 ± 0.08

The electrolyte initially had an average concentration of 0.32 g L^{-1} of Sb with Sb(III) being the predominant species (0.20 g L^{-1}) and Sb(V) as the minor species (0.12 g L^{-1}). When the electrolyte circulated through the column with copper shavings, neither the total antimony content nor the distribution of the antimony species was affected. The elution of the electrolyte through the ion exchange resin resulted in a decrease in total Sb from 0.32 to 0.08 g L^{-1} (ca. 74% extraction). The absorption rate was different for the Sb species. The resin removed most of the Sb(III) contained in the electrolyte, ca. 95% extraction, as its concentration decreased from 0.20 to 0.01 g L^{-1}. This high extraction efficiency of Sb(III) removal with Lewatit TP 260 obtained for industrial copper electrolyte is consistent with previous works that have also employed aminophosphonic resins, such as Doulite-467 and Amberlite IRC747 using synthetic copper electrolyte [18,31]. There is little information about the chemical mechanisms involved in the absorption of Sb(III) likely as SbO^+. The absorption implies a chelation reaction with the amine and/or the phosphate groups of the resin, despite the fact that Sb(III) has little affinity for amine or phosphate compounds. The exact composition of the possible chelate is unknown [32,33].

On the other hand, Sb(V) was only partially removed from the electrolyte by the Lewatit TP 260 resin, as its concentration decreased from 0.12 to 0.07 g L^{-1}, a reduction of ca. 42%. The low affinity of Sb(V) in comparison to Sb(III) for amine or phosphate groups under very acidic conditions has been reported [32]. The partial retention of Sb(V) on a resin with aminophosphonic groups is not expected, as Sb(V) should exist in H_2SO_4 media as $Sb_3O_9{}^{3-}$. Therefore, initial studies indicated that it was not clear if the retention of Sb(V) was due to an ion-exchange mechanism or precipitation in the resin pores [18]. Further studies using scanning electron microscopy by these authors have revealed that Sb(V) is, at least initially, adsorbed by a true ion-exchange mechanism [33].

The average concentration of Fe in the electrolyte is ca. 1 g L^{-1}, the reduced species Fe(II) is the main species (0.90 g L^{-1}), while Fe(III) is minor (0.08 g L^{-1}). When the electrolyte crossed through the column with copper shavings and then through the ion exchange resin, the average total Fe concentration did not change significantly. The reduction of Fe(III) to Fe(II) was promoted, as the average Fe(III) concentration decreased from 0.08 to 0.05 g L^{-1}, with a corresponding increase in Fe(II) concentration from 0.90 to 0.93 g L^{-1}. This indicates that the poisoning of the column due to Fe(III) contamination was prevented by the pre-treatment with Cu shavings [23]. The reduction of Fe(III) to Fe(II) due to the oxidation of Cu shaving to Cu^{2+} ions which are transferred into the electrolyte, can be explained considering the higher redox potential of Fe^{3+}/Fe^{2+} (E^0 = 0.77 V) compared to the redox potential of Cu/Cu^{2+} (E^0 = 0.34 V) [34].

3.1.2. Industrial Sb/Bi Removal Plant

The Sb/Bi removal plant using Lewatit TP 260 ion exchange was installed to treat industrial electrolytes in June 2018. The determination of the species of Sb and Fe in the initial electrolyte, electrolyte pre-treated with copper shavings, and electrolyte with shavings and circulated through the ion exchange resin was evaluated similarly to the pilot plant.

When the industrial plant was started, the electrolyte contained 0.21 g L^{-1} of total Sb with 0.12 g L^{-1} Sb(III) and 0.09 g L^{-1} Sb(V). The total Sb content in electrolytes had decreased from the laboratory-scale pilot plant test in 2016 (0.32 g L^{-1}) due to changes in the chemical composition of copper anodes refined from 2016 to 2018.

When the electrolyte was passed through copper shavings, the total Sb content and species distribution did not change significantly (Table 2), in accordance with previously published studies, which indicate that metallic Cu does not reduce Sb(V) [18]. The circulation of the electrolyte through the second column with ion exchange resin resulted in 100% removal of Sb(III), while Sb(V) was only partially removed. The Sb(V) concentration decreased from 0.09 to 0.04 g L^{-1} indicating a proper functioning of the resin since it eliminated ca. 55% of initial Sb(V) content. Overall, the decrease in Sb total content was from 0.21 to 0.04 g L^{-1} representing ca. 80% of the initial content, a similar value to that obtained from the laboratory scale tests.

Table 2. Average concentrations (g L^{-1}) of Sb and Fe species in samples of initial electrolyte, treated electrolyte with Cu shavings and treated electrolyte with Cu shavings and ion exchange resin in the industrial Sb/Bi removal plant. The results of the concentrations are expressed as mean ± standard deviation (n = 8).

	Initial Electrolyte	**Electrolyte + Cu Shavings**	**Electrolyte + Cu Shavings + Ion Exchange Resin**
	mean ± sd	mean ± sd	mean ± sd
Sb(V)	0.09 ± 0.01	0.07 ± 0.01	0.04 ± 0.00
Sb(III)	0.12 ± 0.01	0.14 ± 0.04	0.00 ± 0.00
Total Sb	0.21 ± 0.01	0.21 ± 0.03	0.04 ± 0.00
Fe(II)	0.85 ± 0.03	0.86 ± 0.02	0.86 ± 0.05
Fe(III)	0.06 ± 0.02	0.05 ± 0.02	0.05 ± 0.01
Total Fe	0.91 ± 0.03	0.91 ± 0.03	0.91 ± 0.04

The total Fe content of the electrolyte was 0.91 g L^{-1}, in the same range as the Fe concentration of the electrolyte employed for the previous experiences performed in the laboratory-scale plant (0.98 g L^{-1}). Fe(II) was again the major species (0.85 g L^{-1}) as compared to Fe(III) (0.06 g L^{-1}), as shown in Table 2. The use of the first column with copper shavings successfully favored the reduction of the content of Fe(III) in the electrolyte, which remained at 0.05 g L^{-1} after the elution through the second column filled with ion exchange resin. Fe(III) represents only 5.5% of the total Fe content of the electrolyte. In this sense, the poisoning of the ion exchange resin due to a high concentration of Fe(III) was prevented. These results agree with previous experiments performed at laboratory scale with the treated industrial electrolyte from the Atlantic Copper Refinery, which indicated that the concentration of Fe(III) ranged 0.03–0.07 g L^{-1} or 2.7–6.5% of the total Fe. Additionally, a similar laboratory experiment performed with industrial electrolyte doped with Fe(III), indicated that Cu shavings have the capability to reduce most of the added amount of Fe(III), resulting in a final concentration of Fe(III) in the electrolyte of 0.03–0.15 g L^{-1}, representing 1–8% of the total Fe [23]. The loading of Fe(III) onto aminophosphonic resins is an ion exchange process. For a sulfate-based electrolyte it can be described by Equation (1), where R represents the resin not involved in the ion exchange [28]:

$$Fe_2(SO_4)_3 + 6(HR) \rightarrow 2(FeR_3) + 3H_2SO_4 \tag{1}$$

The elution and regeneration of the resin have been accomplished by washing with HCl solutions, followed by H_2SO_4 conditioning [23].

The effect of the operation of the Sb/Bi industrial removal plant on the total As content and the distribution of As species in the electrolyte was examined. Electrolyte samples were analyzed on a periodic basis prior to the installation of the Sb/Bi removal plant (February-May 2018) and after its installation (July-December 2018). Figure 1 shows that the total As content was not affected with mean As concentrations of 9.11 g L^{-1} and 9.17 g L^{-1} in the two periods, respectively. This confirms that As is not retained by the aminomethylphosphonic resin, as described by Dreisinger et al., 1993 [35]. At the low pH of the electrolyte, both As(V) and As(III) are present mainly in the form of neutral species (H_3AsO_4 and $HAsO_2$, respectively) according to Eh-pH diagrams [4]. Therefore, their retention onto this resin due to an ion exchange resin mechanism is not favored.

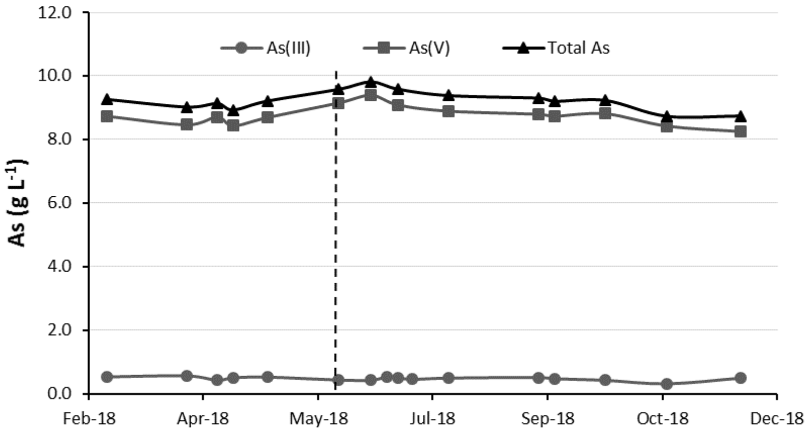


Figure 1. Trend of As species and total As concentration in copper electrolyte for 2018. Dashed line indicates the start of the industrial Sb/Bi removal plant.

No significant oxidation or reduction of As species in the electrolyte was found. Prior to the IX (ion exchange) plant installation, the mean concentrations of As(III) and As(V) were 0.51 g L^{-1} and 8.60 g L^{-1}, respectively. After installation, the mean concentrations were 0.46 g L^{-1} and 8.71 g L^{-1}, respectively. This indicates that in both periods As(V) represent ca. 94% of the total As content in the electrolyte. Braun et al., 1976 [13] indicated that the control of antimony concentration in the electrolyte can be by precipitation of

$SbAsO_4$. The total antimony is controlled by the electrolyte As(V) concentration which promotes precipitation of Sb(III) in the slimes layer as $SbAsO_4$. As(III) from the anode appears to oxidize to As(V) and may inhibit Sb(III) oxidization to Sb(V).

3.2. Evolution of Sb, As and Fe in the Copper Electrolyte

The trends of the total Sb, As and Fe concentration, as well as their redox species, were studied during the 2015–2019 period to further understand the ramifications of the new Sb/Bi removal plant in 2018. The results summarized in Figure 2 indicate that both the total content of Sb and the concentrations of the species decreased over the studied period. The total Sb concentration decreased from 0.29 g L^{-1} to 0.16 g L^{-1} with a more pronounced decline after 2018. The ion exchange plant as expected successfully removed more Sb(III) (0.18 g L^{-1} to 0.09 g L^{-1}) than Sb(V) (0.11 to 0.07 g L^{-1}). This represents a reduction of ca. 50% for Sb(III) and 36% for Sb(V) which reflected Sb(III) preferred absorption on the Lewatit TP 260 ion exchange resin.

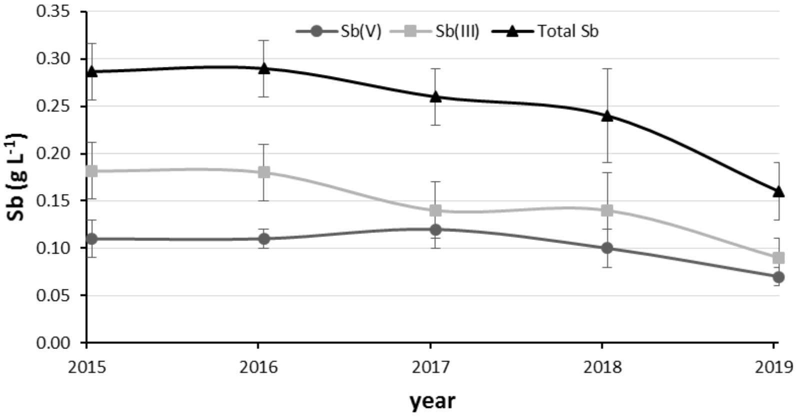


Figure 2. Annual trend of Sb(III), Sb(V) and total Sb concentrations in copper electrolyte from 2015 to 2019.

The annual trends in Figure 2 reveal that in 2017 the distribution of Sb as Sb(III) and Sb(V) was different than the other years. The anomalous behavior was the result of periods where Sb(V) was the predominant antimony species. This is shown in Figure 3 where the percentages of Sb(III) and Sb(V) from 2015 to 2019 are displayed. The electrolyte initially (2015 and 2016) contains 60–70% Sb(III) and 30–40% Sb(V). The proportion of both Sb species became more similar after the start of operation of the Sb/Bi removal plant with ca. 54% Sb(III) and 46% Sb(V).

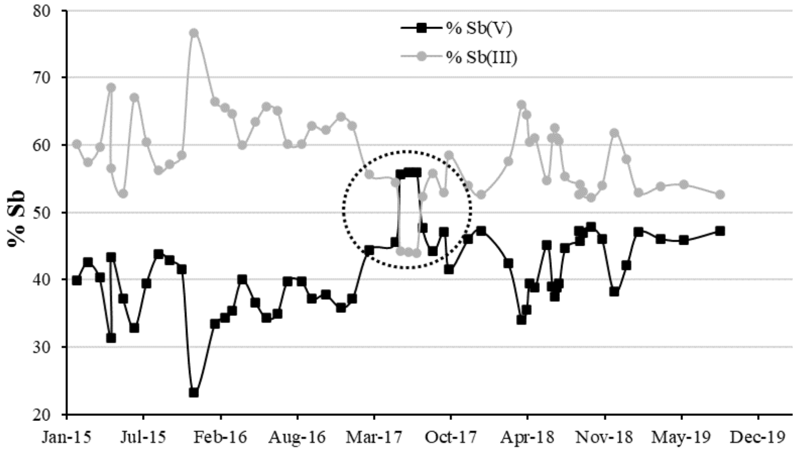
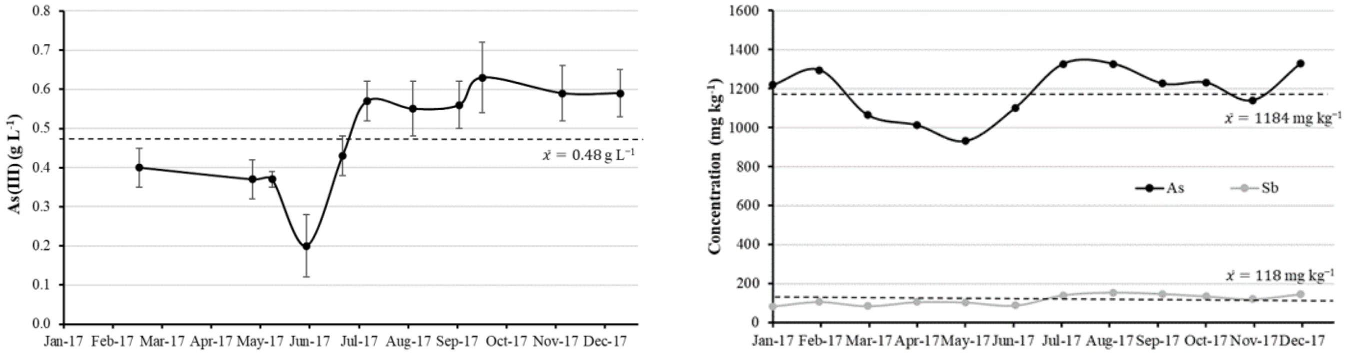


Figure 3. Percentages of Sb species in copper electrolyte during the period 2015–2019. Dashed circle indicates an anomalous period in the distribution of Sb species.

In the anomalous period of May-July 2017, the distribution of the Sb species changed with Sb(V) becoming the predominant Sb species (ca. 40% Sb(III) and 60% Sb(V)). This was accompanied by a statistically significant decrease in the total As content of the electrolyte from ca. 9 to 8 g L^{-1} during these months (*t*-test, $p < 0.05$). This decrease was not only observed in the total As content but also in the As(III) concentration, which was at its lowest during this time frame. Figure 4a shows the concentrations of As(III) in the copper electrolyte during 2017, in which the annual mean As(III) was 0.48 g L^{-1}. It can be seen that during the May–July period, As(III) concentrations were below the mean, with a minimum concentration of 0.20 g L^{-1} in June 2017. The presence of As(III) in the electrolyte is desirable, as it prevents or minimized the oxidation of Sb(III) to Sb(V). Similarly, As(V) showed lower concentrations during this period (7.66–7.87 g L^{-1}) than the 8.28 g L^{-1} annual mean. These results are in agreement with the literature, which indicates the importance of maintaining a high concentration of As(V) in the copper electrolyte [26].



(**a**) (**b**)

Figure 4. (**a**) Concentration of As(III) in copper electrolyte during the year 2017. The dashed line corresponds to the mean annual concentration; (**b**) Concentrations of As and Sb in anodes for 2017.

This diminution of As in the electrolyte was studied in relation to the anode composition during 2017. The monthly concentrations of As in the anodes (Figure 4b) indicated a decrease in the concentration of As between the months of March and June (932–1102 mg kg^{-1}) below the annual mean of 1184 mg kg^{-1}. Both periods of minimum As in the anode and in the electrolyte overlap. The one-month displacement from the decrease in anode arsenic to the arsenic decrease in the electrolyte is due to the time elapsed between anode casting and electrorefining. In the same way, the concentration of Sb in the anodes during the months of March and June (85.8–106.5 mg kg^{-1}) was below the annual average of 118 mg kg^{-1}. These results demonstrated that the low level of impurities in the electrolyte was likely related to changes in the anode composition for the March-June 2017 period.

Regarding iron in the electrolyte, the Fe concentration in the electrolyte remained fairly stable from 2015 to 2019 (0.9–1.1 g L^{-1}). It was determined that Fe(II) was always the major species with an average proportion of 95% (Figure 5). Fe(III) which can potentially poison the ion exchange resin and lower cathode current efficiency represented 5–6% of the total Fe electrolyte content of the electrolyte. At the beginning of this study, the mean annual Fe(III) concentration was 0.06 g L^{-1}. The use of the copper shavings column favored the decrease of Fe(III) concentration, which resulted in a mean concentration of 0.04 g L^{-1} in 2019, thus lowering the proportion of Fe(III) in the electrolyte to less than 4%.

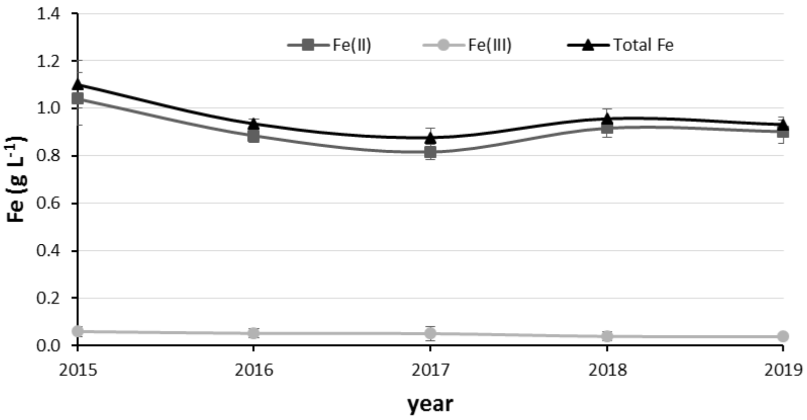


Figure 5. Annual trend of Fe species and total Fe concentration in copper electrolyte from 2015 to 2019.

The annual As concentration in the electrolyte increased during the study period from an initial mean value of 7.7 g L^{-1} in 2015 to 9.2 g L^{-1} in 2019 (Figure 6). The study of the redox species indicated that the main As species in the electrolyte was As(V) with an average annual proportion in the range of 93–95%. The increase in As concentration corresponded to only increases in As(V), whose concentration raised from 7.2 to 8.7 g L^{-1}. During the same period, the mean annual concentration of As(III) remained consistent at 0.5 g L^{-1} (Figure 6). As indicated in Figure 3, that concentration of As(III) in the electrolyte helps to prevent significant oxidation of Sb(III) to Sb(V), a fact that is only observed when the As(III) concentration drops below 0.3 g L^{-1}.

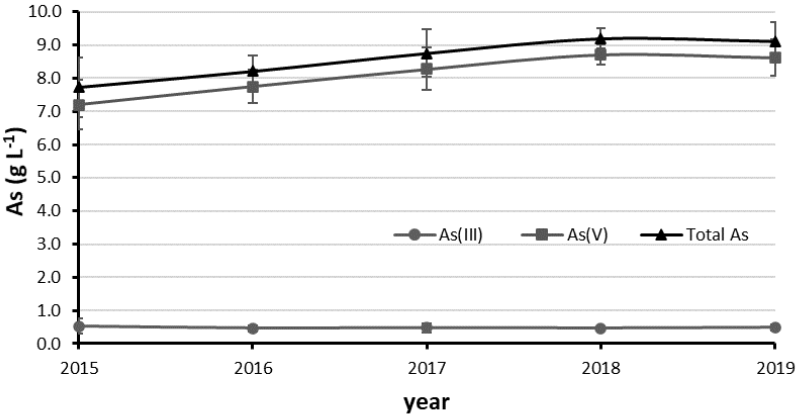


Figure 6. Annual trend of As species and total As concentration in copper electrolyte from 2015 to 2019.

4. Conclusions

The removal of antimony from the industrial copper electrolyte at the Atlantic Copper Refinery was performed on a laboratory-scale and industrial scale. The removal process includes a pre-treatment with copper shavings followed by ion exchange with Lewatit TP 260. The concentrations of the stable oxidation species of Sb, Fe and As in the copper electrolyte were determined. The major species were Sb(III), Fe(II) and As(V) for most time periods.

The laboratory-scale pilot plant successfully removed ca. 100% of Sb(III) and ca. 50% of Sb(V) from the industrial copper electrolyte. The pre-treatment with copper shaving promoted the reduction of Fe(III) to Fe(II) thus avoiding the rapid poisoning of the Lewatit TP 260 resin. The ion exchange treatment process did not alter the concentration and distribution As species in the copper electrolyte.

Since the start of the industrial Sb/Bi removal plant in 2018, a reduction of ca. 50% of Sb(III) and 36% of Sb(V) in the copper electrolyte has been noted. Overall, the total concentration of Sb in the electrolyte has decreased by 45% which should minimize the possible formation of floating slimes and scale formation in piping and on stainless steel blanks. The relative proportions of As (ca. 94% As(V) and Fe (ca. 95% Fe(II) redox species in the electrolyte remained steady during the study period (2015–2019).

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