

CHARACTERIZATION AND PROCESS DEVELOPMENT FOR THE SELECTIVE REMOVAL OF Sn, Sb, AND As FROM ANODE SLIME OBTAINED FROM ELECTROLYTIC COPPER REFINING

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Abstract

The aim of this work was to develop a process for the removal of Sn, Sb and As from anode slime out of copper refinery to disburden a subsequent pyrometallurgical processing for precious metals refinement. For this reason, a detailed literature survey was conducted, followed by a characterization to find the present compounds/alloys and their morphology. A newly developed process concept for the separate extraction of the afore mentioned three target metals was developed and verified by leaching experiments, combined with thermodynamic calculations on their behavior under varying conditions. In this context, the influence of leaching temperature, alkalinity of leaching solution, and solid-liquid ration were evaluated on the extraction yields of Sn, As, and Sb, as well as how to exploit these findings to obtain separate streams enriched in the respective metals.

Keywords: Anode slime; Arsenic; Tin; Antimony; KOH; Selective leaching

1. Introduction

Copper ores are well known to be carriers of a wide range of other valuable metals apart from the main metal. These can be partly recovered as by-products; however, some of them accumulate in side streams of the copper industry and form residues or cause problems in the further processing of by-products. One of these by-products arises in the last step of the high purity copper production via the pyrometallurgical process route, in the electrolytic refining. This step removes all elements less noble than copper by dissolution, while the more noble ones remain in the metallic state and accumulate in the so-called anode slime of the electrolytic copper refining [1,2]. Ignoble elements can also appear in the anode slime if they form insoluble compounds after their dissolution in the electrolyte [3]. As a consequence, anode slime from the electrolytic copper refining is rich in elements such as As, Sb, Sn, Bi, Pb, Au, Ag, PGM, Se, Te, or Ba, in addition to a considerable amount of disproportionate copper [1,2]. The occurrence of aforementioned compounds varies from metallic to sulphatic or oxidic state, in pure or also alloyed form.

The common way of processing anode slime is split into three steps, including pretreatment, concentration and refining. In the first step, pretreatment, elements like Cu, Sb, Pb or also As, which are less noble than copper, are removed.

Although the separation of these elements in a later step would be possible as well, it is typically done preliminary to the precious metals winning to avoid qualitative and environmental problems during the further processing. As the obtained separated by-products are still toxic and show a complex chemistry, the aim of the research described in this paper is to recover Sn, As, and Sb as separately as possible from anode slimes by an alkaline leaching process before lead and other elements are removed by pyrometallurgical means.

Table 1 summarizes the results obtained from the literature survey, showing the composition of various anode slimes from copper plants worldwide. It underlines the fact that there is significant potential for a process capable of separating the elements As, Sb, and Sn effectively to form separate mass/solution-streams.

The first part of this paper deals with a detailed characterization, identifying the chemical compounds as well as the morphology based on EDX mappings in combination with existing literature. This knowledge regarding the morphology, combined with the behavior of present compounds/alloys under varying chemical conditions, forms the base for a metallurgical process development. In addition, prior tentative trials carried out with different leaching reagents showed that an alkaline leaching process is the first choice for the targeted removal of afore mentioned metals.

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Table 1. Chemical composition of copper anode slimes at various refining plants (concentration in mass-pct)

Origin of slime (Plant, country)	Cu	Au	Ag	Ni	Se	Te	As	Sb	Sn	Pb	Ref.
Aurubis, Germany	15.5	0.5	13	n.m.	6	1.5	4	6	n.m.	14	[4]
Outokumpu, Finland	4.2	0.9	7.7	n.m.	3.3	1.5	4.9	5.1	n.m.	5.3	[4]
Kazakhmys, Kazakhstan	1.2	0.25	19.5	n.m.	7	1.1	0.85	5	n.m.	35	[4]
Pol'skaya med', Poland	11.8	0.02	32.8	n.m.	2.3	n.m.	1.8	0.3	n.m.	16.5	[4]
Boliden, Sweden	9.8	0.65	21.3	n.m.	3.3	1.6	0.95	2.6	n.m.	7.8	[4]
Kennecott, USA	20	0.5	5	n.m.	5	1	5	1	n.m.	30	[4]
Noranda, Canada	17	1	25	n.m.	7	4	1.5	1	n.m.	10	[4]
Uralk Elektromed', Russia	2	0.7	17.5	n.m.	9	3.5	2	12.5	n.m.	24	[4]
Kyshtym MEZ, Russia	11.7	n.m.	8.92	n.m.	5.97	3.3	3.3	24.8	n.m.	18	[4]
Mexicana de Cobre, Mexico	12.7	0.035	15.1	0.003	4.2	0.37	5	5	n.m.	14.8	[5]
Sarkuysan Copper, Turkey	25.8	0.23	2.8	0.29	4.68	0.9	3.93	0.99	8.1	12.93	[6]
Hindustan Copper Ltd., India	12.3	0.006	1.5	36.8	10.5	3.4	0.036	0.01	n.m.	0.16	[7]
SWIL Ltd., India	1	n.m.	12	23	1.2	n.m.	0.3	n.m.	2.4	11	[8]
Nat'l Sarcheshmeh Copper Co., Iran	4.3	0.12	4.5	n.m.	7.3	n.m.	n.m.	2.16	n.m.	3.54	[9]

2. Experimental

The experimental part covers the description of the investigated material, the characterization, as well as the performed experiments for a hydrometallurgical Sn, As and Sb separation.

2.1. Material and characterization

The anode slime investigated in this study was obtained from a lab-scale electrolysis operated at the Chair of Nonferrous Metallurgy, Montanuniversitaet Leoben in Austria. Prior to the characterization and leaching tests, the obtained material was washed in an acidic-sulphatic solution, in the same way as carried out in industrial plants, for keeping the copper losses at a minimum level. The chemical analysis of the washed material is shown in Table 2.

Table 2. Chemical analysis of elements of washed anode slime (in wt-%)

Species	Te	SiO ₂	BaO	Cl	S	Ag	Au	Pt	Pd
wt-%	1	0.2	8.3	0.7	3.1	7.2	0.43	0.01	0.06
Species	Cu	Sb	As	Sn	Pb	Zn	Ni	Bi	Se
wt-%	7.1	11.5	3.5	3.3	22.8	0.6	3.6	1.2	1

The above table shows the content of the investigated elements antimony, arsenic and tin with 11.5 pct, 3.5 pct and 3.3 pct, respectively. Other elements forming the main matrix include 22.8 pct lead and 8.3 pct BaO in addition to 7.1 pct copper. As the source of barium is not represented by the ore or a chemical additive taking part in the copper winning process itself, it is introduced in a step prior to the electrolytic refining. The origin of the barite (BaSO₄), as present in the slime, derives from the use as mold dressing in the dies of the anode casting. This mold dressing partly still sticks on the anodes when they are put into the cells. The 8.3 pct BaO consequently correlates with 12.6 pct BaSO₄. During the refining, the inert barite sinks to the bottom and accumulates in the slime of the electrolysis cells together with other elements. Typically, it does not take part in reactions with any of the investigated elements.

In general, the focus of the characterization was primarily set on the elements Sn, As and Sb. As these are present in a wide variety of compounds or alloys with other elements, the morphology of Pb, Bi, Ni, Ag and other elements is partly described as well. Due to the fact that the x-ray diffraction analysis does not lead to a clear result concerning compounds of Sn, As and Sb because of an exceeding amount of different other elements, the investigation was performed with an EDX unit at a scanning electron microscope (Jeol JSM-IT300).



2.2. Pourbaix diagrams (Eh-pH) for Sn, As and Sb

Stability- or equilibrium diagrams were calculated with the software HSC Chemistry, on the one hand to determine the required conditions for the leaching

Additionally, the influence of the concentration, if decreased to $1/100 \text{ M/kg H}_2\text{O}$ tin and arsenic, does not shift the stability regions significantly. This is the reason why the inclusion of figures for these cases was dispensed.

Fig. 1-c shows the Pourbaix diagram for $1 \text{ M/kg H}_2\text{O}$

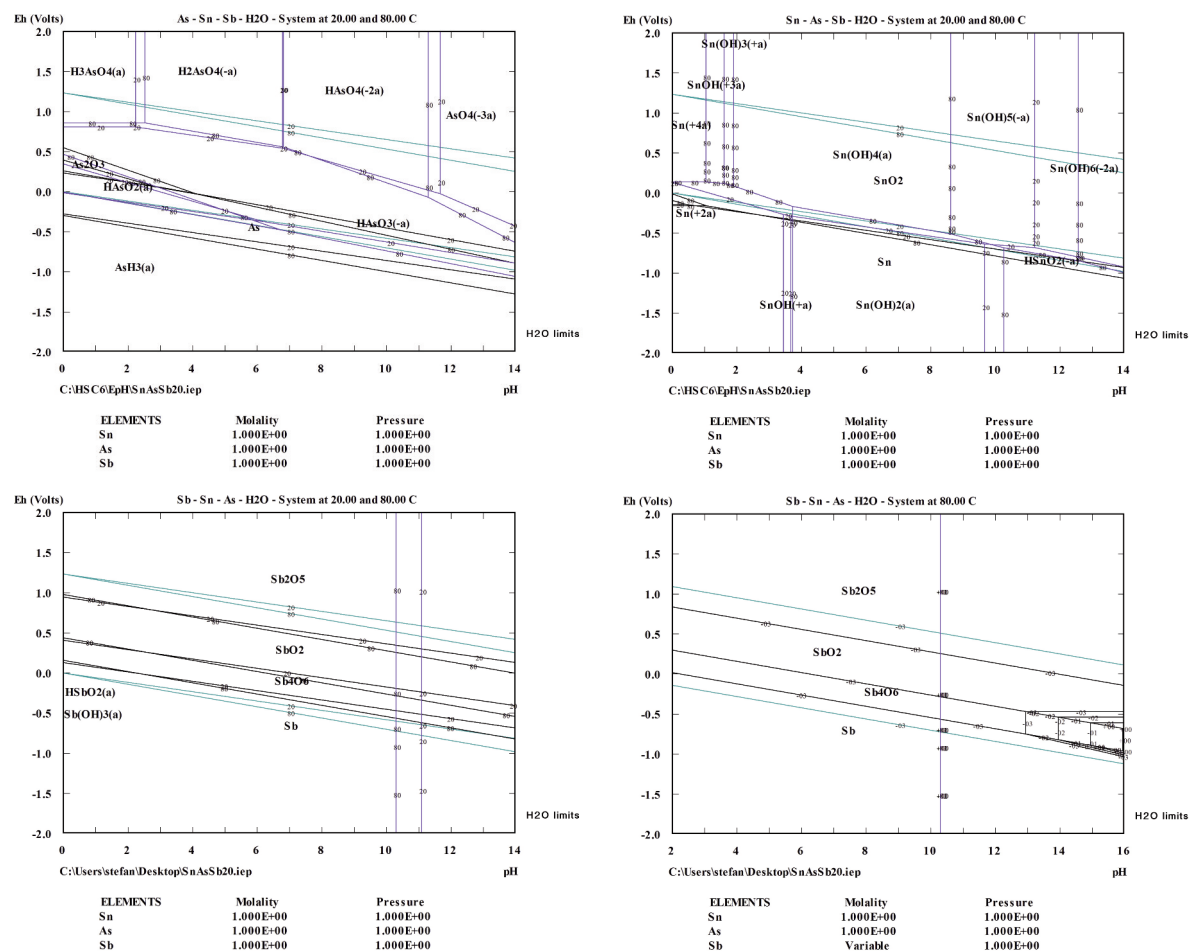


Figure 1. 1 – a) Pourbaix dia. for 1 M As/kg H₂O b) Pourbaix dia. for 1 M Sn/kg H₂O c) Pourbaix dia. for 1 M Sb/kg H₂O d) Pourbaix dia. for varying Sb molarity

experiment and on the other hand to further investigate the obtained result, especially for the Sb yield.

The calculated data is therefore summarized in a so called Pourbaix diagram. It uses thermodynamic data and represents the stability of a metal as a function of the electrochemical potential and the pH.

Figs. 1-a and 1-b show the stability regions for $1 \text{ M/kg H}_2\text{O}$ of tin and arsenic for a minimum temperature of 20 °C and a maximum temperature of 80 °C. The stability regions for the further two investigated leaching temperatures of 40 °C and 60 °C lie in-between the two calculated cases.

antimony for the area of pH 0 to 14. Similar to the diagrams for tin and arsenic, the change of temperature has no significant influence on the equilibrium regions. However, the decrease of concentration does have an influence on the stability regions (see Fig. 1-d). In the area of pH 13 to 16 a shift of the equilibrium lines for $\text{Sb(OH)}_4\text{(aq)}$ takes place in case of a decrease down to $1/1000 \text{ M/kg H}_2\text{O}$ Sb. The label of the line gives the exponent of antimony, like for instance -2 for $1 \cdot 10^{-2} = 0.01$. Further calculations showed that the concentrations of tin and arsenic have no significant influence on the stability region of antimony.



2.3 Experimental setup and procedure

The fundamentals of the setup of the leaching experiment are based on the publication by Fernández et al. [10]. The present experimental parameters were chosen in a way to investigate a possible separate winning of As and Sb. In addition, the behavior of Sn was investigated as a third element in the present trials.

The required molarity of the KOH solution is based on thermodynamic calculations of the solubility of tin, arsenic and antimony, as shown in the prior chapter. A pH of around 14 seems to be sufficient for a dissolution of tin and arsenic in case of a high or a low concentration of dissolved element. For antimony, the concentration of the dissolved element needs to be low in order to shift the stability region of a soluble compound into the mentioned pH area, resulting in a lower solid-liquid ratio for the experiment.

In addition to the thermodynamic background, kinetic aspects also have an influence. Therefore, the temperature and the solid liquid ratio will be investigated further, based on the parameters given in Table 3. The leaching experiments are carried out in alkaline media, in a 4 M KOH aqueous solution, representing a theoretical starting pH of 14.6. The washed anode slime was stored at atmospheric

Table 3. Summary of experimental parameters for the alkaline leaching trials

Parameter	Unit	Experiment №					
		1	2	3	4	5	6
T	K	293.15	313.15	333.15	353.15	353.15	353.15
	(°C)	-20	-40	-60	-80	-80	-80
t	s	1800	1800	1800	1800	1800	1800
S/L	1	1/4	1/4	1/4	1/4	1/10	1/20
c(KOH)	mol/l	4	4	4	4	4	4

conditions preliminary to the leaching experiments, leading to the circumstance that it is at least partly oxidized by the atmosphere. As shown in Table 3, the trial series consists of six experiments, varying the temperature and the solid/liquid ratio, respectively, in the range from room temperature at 293.15 K (20 °C) up to 353.15 K (80 °C) (experiments 1, 2, 3 and 4) and between a S/L ratio of $1/4$ to $1/20$ (experiments 4, 5 and 6). The treatment time and KOH concentration were kept constant for this trial series.

The leaching was conducted in either an 800 ml or a 250 ml beaker, depending on the investigated S/L ratio. In each case 30 gram samples (dry mass) of anode slime are leached. First, the KOH solution was

prepared and heated to the desired temperature with an electronically controlled heater. In order to keep the solids in suspension and to maintain a quick reaction the beaker was continuously stirred with a magnetic stirring unit. After 30 minutes (1800 s) of leaching, the solids were separated from the solution with a laboratory filter paper and a filtering flask operated under negative pressure. The separated solid particles (filter cake) were dried at 378.15 K (105 °C) before the filter cake and the solution were analyzed.

3. Results and discussion

Based on the carried out literature survey and the thermodynamic calculations, the present anode slime was characterized and leached to develop and find the optimum parameters for the separation of Sn, As, and Sb, and the verification of a corresponding process flowsheet.

3.1. Morphology of the investigated anode slime related to Sn, As and Sb

Fig. 2 illustrates the mapping of the investigated anode slime, showing an interesting combination of different relevant particles, their morphology and composition. Arrow 1 in the first row proves the existence of barite (BaSO_4), as described in the “2.1 Material and characterization” section. Cu, Ni and Sb (arrow 3 in the second row of the EDX mapping) form a chemical compound, with the structure of $\text{Cu}_3\text{Ni}_2\text{-xSbO}_6\text{-x}$. Often, but not solely, Cu, Ni and Sb appear in a needle-shaped form in the present sample. In the stoichiometric calculation of the analyzed grains via quantitative EDX analysis multiple times very pure particles, with little tramp elements and quite accurate stoichiometry correlation of elements, were found. However, particles with hyperstoichiometric amount of Ni or Sb, or also combined with a sulphatic lead phase (arrow 2) are also present within the material.

The correlation of antimony and arsenic in different compounds was detected as well as illustrated by the areas indicated by arrow 4. However, it is important to keep in mind that arsenic-antimony oxides (Sb_2O_3) $_x$ (As_2O_5) $_{1-x}$ appear in a wide range of their stoichiometric relation, which was confirmed by different grains of several investigated areas of the sample. This is the reason why no definite proportion between these elements can be determined in the EDX spectra.

Sn was found together with copper in needle-shaped bodies (see Fig. 2, arrow 5), in similar appearance as observed in the case of Sb, Ni or Cu. This was confirmed by other investigated grains, like shown in Fig. 3 (spectrum 2).



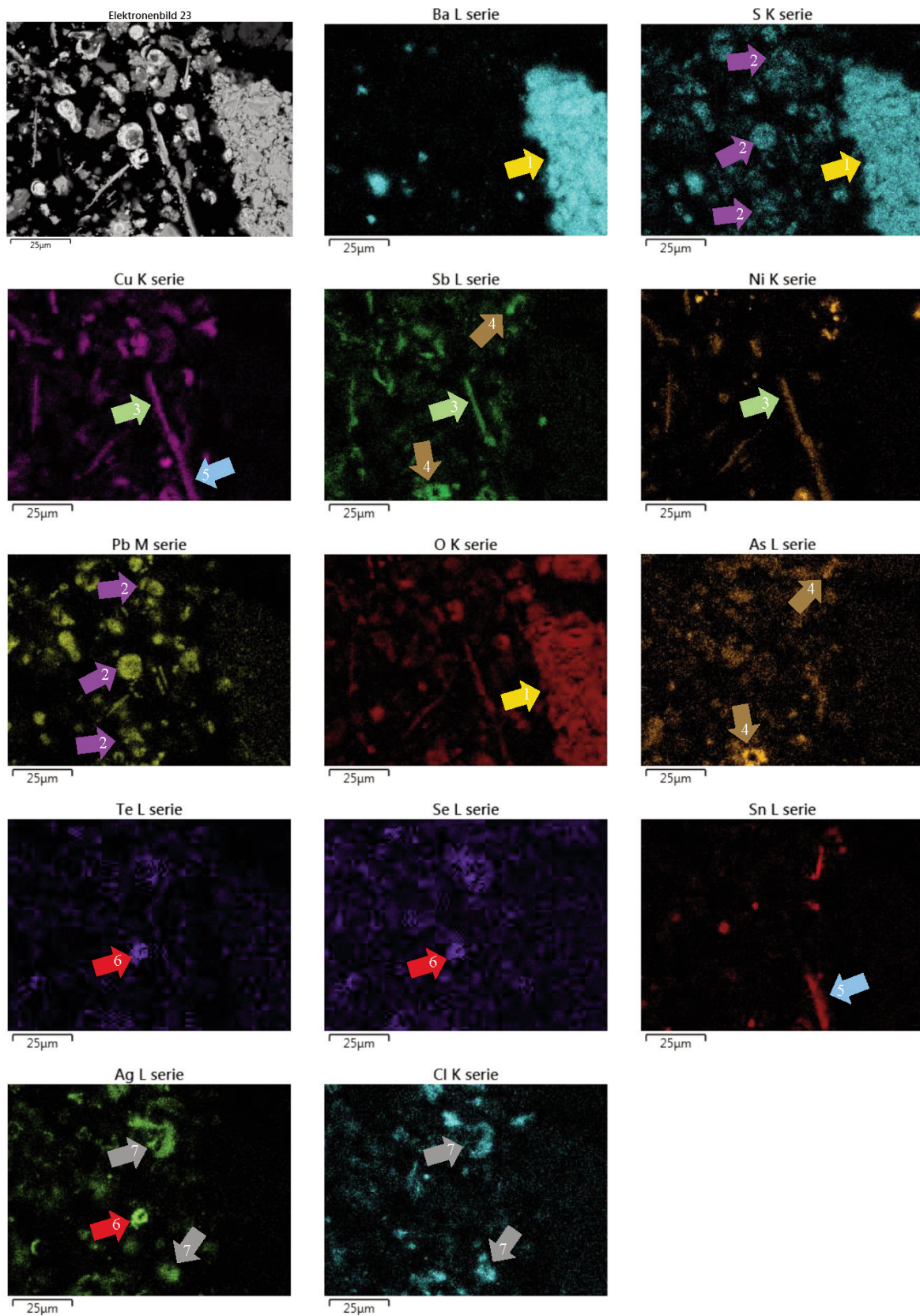


Figure 2. EDX mapping of a washed anode slime sample

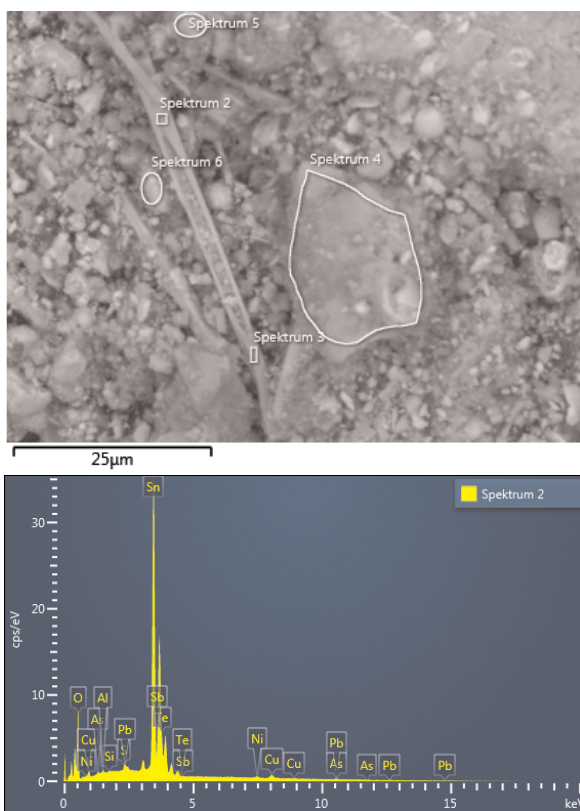


Figure 3. Sn-Sb needle, analyzed by an EDX scan

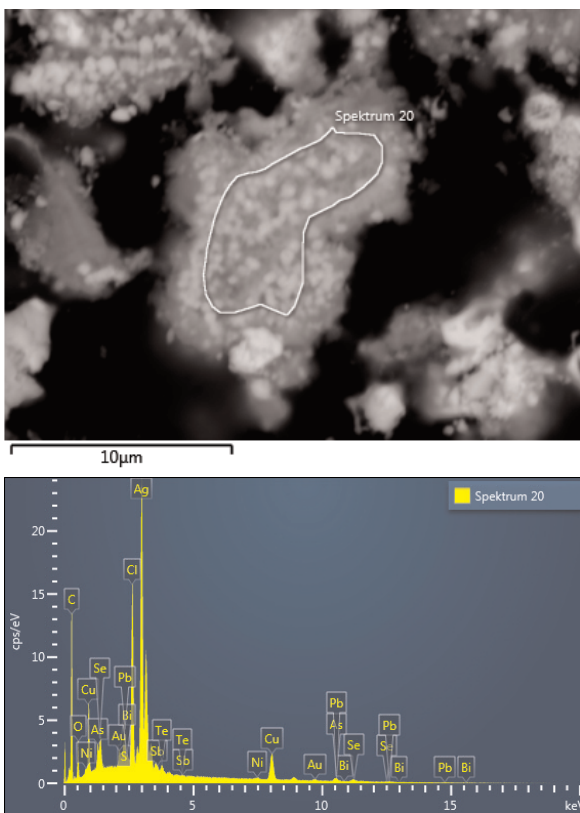


Figure 4. AgCl particle, analyzed by an EDX scan

Silver-mixed selenides/tellurides (arrow 6) are suspected multiple times in the stoichiometric calculations of quantitatively analyzed grains of the anode sample. Their origin can be found in dissolved silver which reacts with selenide particles, forming different copper-silver-selenides. As reported in literature, Ag, Se, Cu and Te form for example silver/copper-di-selenide/telluride $(Ag,Cu)_2(Se,Te)$ or also Cu_2Se next to $AgCuSe$ or Ag_2Se [5].

Arrow number 7 in Figure 2 points at a silver chloride particle. It is obvious that Se and/or Te also partly correlate with this Ag-Cl grain. The presence of AgCl was confirmed by several other particles, as shown in Fig. 4. However, in this particle a small amount of Se was found as well.

3.2. Extraction yields of the leaching experiments

Table 4 summarizes the extraction yields of the analyzed elements obtained during the KOH leaching, calculated as the percentage of the initial amount of each element contained in the untreated dried anode slime. The base is formed by the analysis of the filter cake and is validated via an additional calculation based on the obtained analysis of the pregnant solution.

Table 4. Removal rates of selected elements due to KOH leaching (based on analyses of the dried filter cake)

Parameter	Unit	Experiment №					
		1	2	3	4	5	6
As	pct	37.2	45.9	47.9	70.5	84	87.7
Sb	pct	2.3	-	-	-	25.3	37.2
Sn	pct	15.9	30.1	36.9	48	80.1	79.8
Ag	pct	6.1	14.7	14	14.6	23.8	9.6
Cu	pct	9	6.5	15.5	15.1	24.3	29.1
Ni	pct	3.4	-	8	9.2	-	-
Se	pct	-	1.8	2.9	2.8	11.4	-
Te	pct	18.2	18.9	19.4	23.6	33.8	25.9

Fig. 5 shows a clear tendency of the increased leachability of As and Sn with an increasing temperature. The solid/liquid ratio, KOH concentration and leaching time were kept constant, respectively $1/4$, 30 minutes and 4 mol/L. About 70 pct of the contained arsenic were removed. Increased temperature and treatment time will further increase the yield but very likely with more tramp elements dissolved aside. An interesting detail beside the 48 pct Sn yield is the fact that almost zero Sb was dissolved at the present conditions.

In addition to As, Sn, and Sb, Ag and Cu were also dissolved up to approximately 15 pct. Due to their noble character, a cementation step is easily realizable. While Se was not leached in significant amounts at all investigated temperatures, Te and Ni were partly dissolved as well.

Fig. 6 shows the achieved extraction yields depending on the solid/liquid ratio at a constant leaching temperature of 353.15 K (80°C). With a decreasing S/L ratio, the extraction yield increases for Sn and As, but also for Sb, up to nearly 90 pct As, 80 pct Sn and 37 pct Sb.

The high amount of solution can be identified as the reason for the better extraction rate, which decreases the relative amount of dissolved element per volume-unit solution compared to the S/L ratio of the experiment 1, 2, 3 and 4.

Although 37 pct extraction yield for Sb was achieved, higher yields can be expected based on the results mentioned in the literature [10]. This is underlined by the carried out thermodynamic calculations (see Fig. 1-d), illustrating an equilibrium between the solid Sb compound and the soluble $\text{Sb(OH)}_{4(\text{aq})}$ for a concentration of 0.01 Mol Sb at a pH of ~ 14 . This correlates with the dissolved amount of Sb in the executed trial 6 (starting pH of 14.6) well, within $1.7 \cdot 10^{-2}$ Mol Sb per kg H_2O were dissolved. It seems that a lowering of the ratio of solid to liquid can lead to higher extraction rates.

Furthermore, in the filtered solution crystallization took place, leading to a product rich in Sb and As. The reason for this is the high dependence of the Sb solubility on the solution temperature. As a consequence, filtering must take place at the same temperatures as leaching, if Sb should be successfully removed from the anode slime. Nevertheless, these described effects can be exploited for the recovery of

Sb from the solution, after a “hot” solid/liquid separation has been carried out.

3.3. Process flowsheet for a Sn, As, and Sb separation by an alkaline leaching of anode slime

Referring to the characterization, thermodynamic calculation and carried out leaching experiments, a selective leaching requires at least two steps. First, a maximum of As (and Sn) is extracted, while Sb remains in the solid fraction. The remaining solids are then leached in the second step, when As and Sn are already removed to a great extent, resulting in a Sb-rich stream with a low Sn and As content. With the aim of keeping the process as simple as possible, the acidity as well as the leaching solution are aimed to be kept the same. Based on this target the separation can be achieved by a high solid liquid ratio (1/4) in the first step and a low (1/20) ratio in the second leaching step. In order to achieve a fast extraction, the temperature needs to be high, followed by a hot filtration step with the aim to avoid precipitation, especially of dissolved Sb. By exploiting the strong dependency of the Sb solubility a simple cooling after the filtration in the second step leads to a Sb-rich crystallized product. The solution, which is then low in Sb, can be recycled to the first step to remove the dissolved Sn, which can still be present from the second step in small amounts.

A flowsheet showing the developed core process for a selective separation of Sn, As and Sb from anode slime treatment is shown in Fig. 7.

The Sn-As-rich solution from the first step can further be treated to obtain a Sn-rich and an As-rich phase. As described by Junwei Han et al. [11], the separation of Sn and As from an alkaline solution can be achieved with a simple cooling crystallization. The

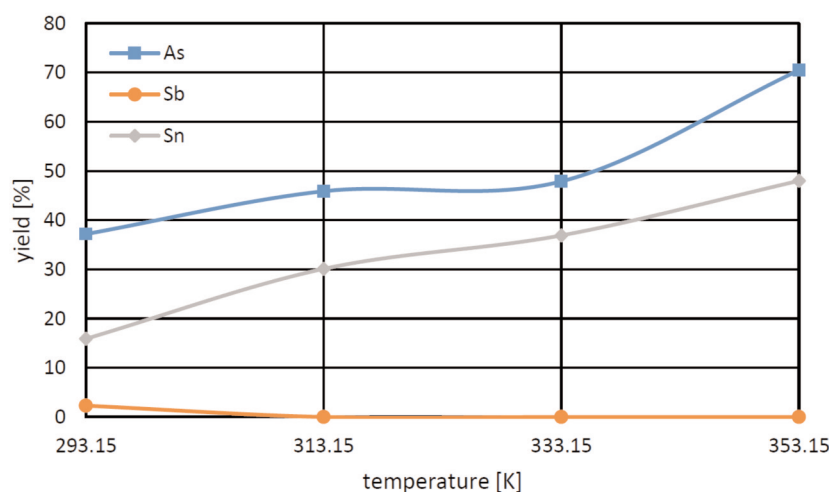


Figure 5. Removal yield of As, Sb and Sn depending on the leaching temperature (experiments 1, 2, 3 and 4), $S/L = 1/4$, $t = 30$ min, $c(\text{KOH}) = 4.0$ mol/l



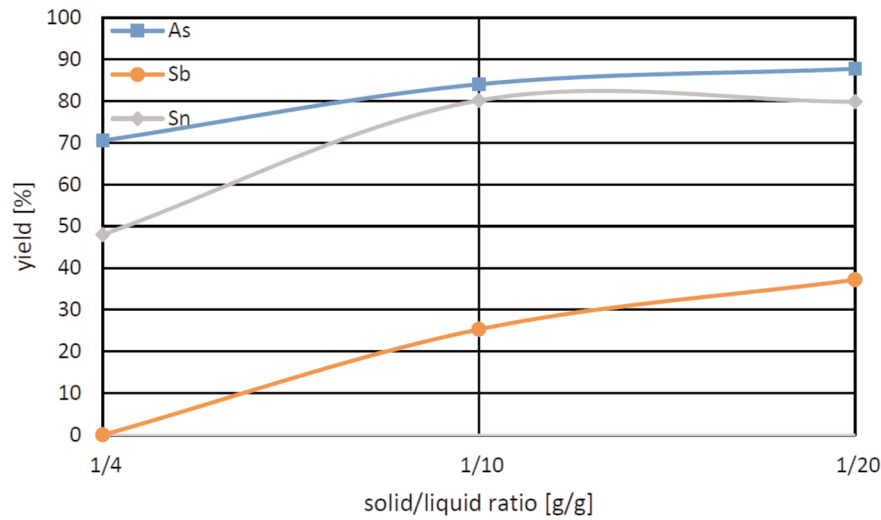


Figure 6. Removal rates of As, Sn and Sb depending on the solid/liquid ratio (experiments 4, 5 and 6), $T = 353.15 \text{ [K]}$, $t = 30 \text{ [min]}$, $c(\text{KOH}) = 4.0 \text{ [mol/l]}$

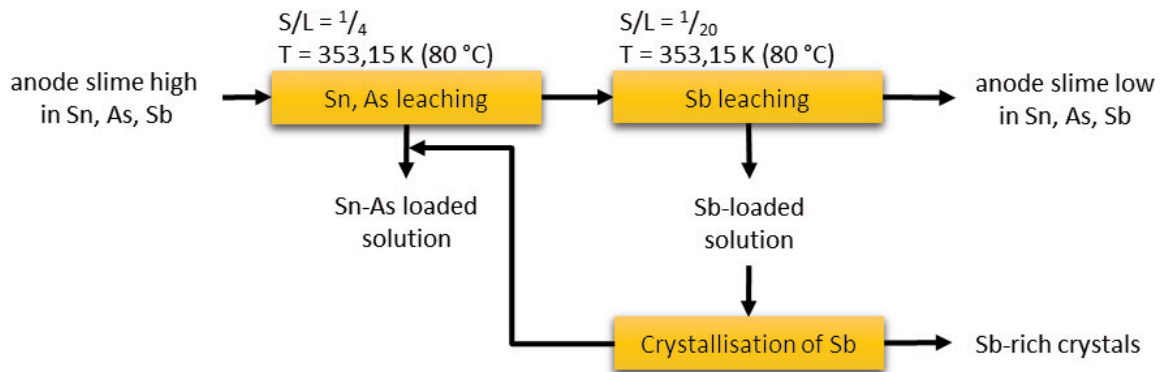


Figure 7. Process flow sheet for KOH leaching to remove Sn, As and Sb from anode slime

base for this is formed by the different solubility of arsenic and tin depending on the temperature. While the Sn stays mainly in the alkaline solution, arsenic can be removed very effectively by cooling to room temperature, in a similar way the crystallization of Sb can be exploited to produce a Sb-rich product. One possibility to achieve cooling can be, for instance, mixing with the already cooled-down solution from the filtration executed in the second step.

4. Conclusions

The characterization showed that specific elements occur together in alloys or compounds of the anode slime. Obtained correlations were found for:

- Pb, Bi, Ba and S
- As, Sb, Se and Te
- Ag, Cl and Te, Se
- Cu, Ni and Sb
- Sn with Cu

Based on the aforementioned fact of elements frequently appearing together, it is obvious why also Ni, Cu, Se, Te, and Ag are partly dissolved together with Sn, As, and Sb, and can consequently be found in the process solution as well. This leads to the necessity of further recovery steps. However, in the present paper the separation of Sn, As, and Sb by an alkaline leaching with KOH was the main target and evaluated successfully. In this context a process flow was developed to effectively remove tin, arsenic and antimony from the copper refinery anode slime. In case of antimony it was shown via the executed thermodynamically calculations that a further increase of the extraction yield might be linked to the exchange of leaching liquor or the decrease of solid-liquid ratio to keep the dissolved Sb-content below the equilibrium molarity. This can be achieved by increasing the solution amount or by utilizing a counter current leaching process.

Furthermore, the arsenic extraction increased with

decreasing S/L-ratio. This leads to the possible conclusion that the remaining arsenic as well as antimony occur in the same compound, for example as $(\text{Sb}_2\text{O}_3)_x \cdot (\text{As}_2\text{O}_5)_{1-x}$. With this, the dissolved antimony would be the limiting factor for a further increased arsenic removal as well.

Summing up, a process was developed successfully for the removal of tin, arsenic as well as antimony from anode slime in separate streams. Next to an increased antimony also a most probably linked arsenic extraction yield can be realized, by a specific system configuration, like increased S/L ratio realized by a counter current leaching.

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KARAKTERIZACIJA I RAZVOJ PROCESA ZA SELEKTIVNO UKLANJANJE Sn, Sb I As IZ ANODNOG MULJA DOBIJENOG ELEKTROLITIČKOM RAFINACIJOM BAKRA

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Apstrakt

Cilj ovog rada je razvoj procesa za uklanjanje Sn, Sb i As iz anodnog mulja nastalog rafinacijom bakra, da bi se rasteretio kasniji pirometalurški postupak rafinacije plemenitih metala. Zbog toga je urađen detaljan pregled literature, nakon čega je usledila karakterizacija da bi se otkrili prisutna jedinjenja/legure i njihova morfologija. Nedavno razvijeni koncept za odvojenu ekstrakciju tri prethodno pomenuta metala razvijen je i verifikovan uz pomoć eksperimenata luženja kombinovanih sa termodinamičkim proračunima njihovog ponašanja pod različitim uslovima. U ovom kontekstu, uticaj temperature luženja, alkalnost rastvora za luženje i odnos čvrstog – tečnog su vrednovani na prinosima ekstrakcije Sn, As i Sb, kao i kako iskoristiti ove nalaze u cilju dobijanja posebnih tokova obogaćivanja ovim metalima.

Ključne reči: Anodni mulj; Arsen; Kalaj; Antimon; KOH; Selektivno luženje

