Article

Pyrometallurgical removal of arsenic from dusts collected in electrostatic precipitators of copper

Part II:-Dust from a Fluidized Bed Roasting Furnace

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Abstract: This paper, the second in a series of two, describes the experimental results of removing arsenic from the dust collected in electrostatic precipitators of a fluidized bed roasting furnace (RP dust). The fluidized bed roasting process 600 kilotons of copper concentrate per year with 3-6 wt% of concentration of arsenic, producing a roasted product with a low content of arsenic below 0.3 wt%. The process generates 27 kilotons of the RP dust per year with a concentration of arsenic of the order of 5 wt% and copper concentration of around 20 wt%. Subsequently, the dust collected in the electrostatic precipitators is treatment by hydrometallurgical methods allowing the recovery of copper, and the disposition of arsenic as scorodite. Based on the successful experimental results obtained in part I of this series of papers, this work proposes to use the pyrometallurgy sulfidation-decomposition-oxidation process to carry out the volatilization of arsenic from the RP dust. Then, the obtained material can be recirculated in copper smelting furnaces allowing the recovery of valuable metals. The set of experiments carried out in the roasting of the mixture of copper concentrate/RP dust and sulfur/RP dust used different ratios of mixtures, temperatures, and roasting times. By different techniques used, the characterization of the RP dust determined its size distribution, morphology, chemical, and mineralogical composition. RP dust is a composite material of small particles (<5 μm) in 50 μm agglomerates, mostly amorphous with a complex chemical composition of sulfoxide. The results of the roasting experiments indicated that for a 75/25 weight ratio of the mixture of the copper concentrate /PR dust under 700 ° C, 15 minutes of roasting time with air atmosphere, the volatilization of arsenic maximum reached was of 96% by weight. The arsenic concentration after the roasting process is less than 0.3% by weight. For a 5/95 mixture of sulfur/RP dust, at 650 ° C, the volatilization of arsenic reached a promissory result of 67 %.

Keywords: Copper smelter dust; electrostatic precipitators of copper; removal of arsenic; sulfidation; roasting process, fluidized bed roasting.

1. Introduction

Worldwide, sulfide concentrate of copper, gold, and nickel with high arsenic content are treated in roasting furnaces, obtaining calcines with low concentrations of arsenic. The obtained calcine is appropriate for further metallurgical operations for the recovery of valuable metals [1-5]. In Chile, a copper concentrate with a high content of arsenic is roasted in a fluidized bed furnace, which process 600 kilotons of copper concentrate per year generating an off-gas rich in arsenic that during the process of gas cooling produce 26 kilotons of fine particles dust (RP dust) [6, 7]. This dust is collected in electrostatic precipitators [6-8] and further processed by hydrometallurgical techniques allowing to recover copper and other valuable metals [6, 7]. The arsenic dissolved during the leaching process is disposed as a precipitate of scorodite [6, 7].

In the literature is reported that the treatment of dust by hydrometallurgy process obtains a low recovery of copper (60-70 wt%), a considerable generation of solid residues (around 40 wt% concerning the original material) [9, 10], a high consumption of chemicals materials, and there is uncertainty regarding the long-term stability of the scorodite or other arsenic oxides [1, 11, 12]. In terms of stability, it reports that the fixation of arsenic as a glassy material is the best option just as it keeps the arsenic stable for geological periods of time. To produce a glassy residue, it is desirable a waste material with a high concentration of arsenic oxides [1, 11].

Pyrometallurgical treatment of RP dust has the potential to increases the recovery of copper (and other valuable metals), reduce solid residues, and generate condensed gases with a high concentration of arsenic oxides [13-17].

In published articles describing the pyro-metallurgical process for removing arsenic from copper smelting dust, the literature has not reported the characterization or works related to the volatilization of arsenic from RP dust. In the first article of this series of two, was demonstrated the technical possibility of pyro-metallurgical volatilizing arsenic from a Flash Smelting Furnace dust [13]. The thermodynamic information related to the reactions of sulfidation of arsenic oxides, thermal decomposition of enargite/tennantite, and oxidation of arsenic sulfides indicated that all of them were spontaneous at temperatures between 600 and 700 °C [13]. Thus, based on the results of volatilization of arsenic of the first paper, the main aim of this work is to characterize the RP dust, evaluate at a laboratory scale, what temperature, roasting time, and atmosphere could increase an arsenic volatilization in mixtures of copper concentrate/RP dust, sulfur/RP dust or pyrrhotite/RP dust through the sulfidation-decomposition-oxidation processes described in the literature [13].

2. Materials and Methods

2.1Copper concentrate with high concentration of arsenic

An essential section of this article is related to roasting experiments of copper concentrate mixtures with a high concentration of arsenic and RP dust. The characterization and roasting behavior of this concentrate was reported in the first article in this series of two papers [13]. The obtained information will be part of the study material to be used during the development of this work.

2.2 Characterization of the RP dust

The set experimental worked on the dust collected from the electrostatic precipitators of a Bed Roasting Furnace (RP dust). By using QEMSCAN (TSCAN, Model: VEGA3 LM) carried out the analysis of the mineralogical composition and the distribution of each of the phases of the dust. Table 1 shows the results obtained (QEMSCAN), where arsenic is present as a multi-component materials of Cu-Fe-As-S-O and chenevixite. Table 2 displays the elemental composition by Atomic Adsorption Spectroscopy (AAS) (AAS Perkin Elmer, Model: PinAAcle 900F Flame) analysis, reporting a concentration of copper of 17.2 (wt%) and a concentration of arsenic of 4.4 wt %. The table include the concentration of copper with high concentration of arsenic used in the experiments. The quantitative data collected for the arsenic concentration by AAS analysis will be used in future calculations.

Table 1. QEMSCAN analysis of RP dust (analysis conditions: Probe current = 4.55 nA, beam intensity: 25 kV).

Phase	Normalized Mineralogical Composition (wt%)	As distribution per phase (wt%)
Si-Fe-Cu-S-As-Al-O-K-Cl-Sb Amorphous complex oxides	58.2	88.1
Bornite: Cu_2FesS_4	17.1	0
Micas (Muscovite/illita) $KAl_2(AlSi_3O_{10})(F,OH)_2$/ $(K, H_3O)(Al, Mg, Fe)_2(Si, Al)_4O_{10}[(OH)_2,(H_2O)]$	10.5	0
Chalcocite/Digenite: Cu_2S/Cu_9S_5	3.7	0
Quartz: SiO_2	2.3	0
Chenevixite $Cu_2Fe_2(AsO_4)_2(OH)_3$	1.9	11.0
Tenorite CuO	1.7	0
Hematite/magnetite: Fe_2O_3/Fe_3O_4	1.1	0
Aluminium Clays	0.5	0
Wurtzite: ZnS	0.4	0
Sphalerite: (Zn, Fe)S	0.4	0
Others	1.8	0.9

Table 2. Atomic Absorption (AAS) elemental chemical analysis of RP dust

	Elemental composition (wt%) or (ppm)								
	Cu	**Fe**	**S**	**As**	**Ag (ppm)**	**Sb**	**Bi (ppm)**	**SiO_2**	**Fe_3O_4** (insoluble)
RP dust	17.2	13.6	7.7	4.4	460	1.0	100	20.1	11.4
Copper concentrate	42.1	11.1	30	5.2	486	0.3	68	5.0	6.5

Figure 1. exhibits the size distribution of an RP dust obtained by using hydro cyclone sizers, indicated that 99.4 wt% of the sample corresponds to size under 53 µm and 90 wt% to size under 10 µm. By using SEM/EDS (SEM: Zeiss Evo Model: MA10, EDS: Oxford X-Act, Detector BS, Software INCA) were determined the phases present in the RP dust. Figure 2 includes SEM micrographs, and Table 3 summarizes the elemental chemical composition of the selected points analyzed by EDS. Both analyses indicated that the particles have a few microns in size, and some of them appear as

agglomerates, where there are no appreciable visual differences between the particles or indication of any crystal formation. The EDS analysis indicated that the RP dust corresponds to amorphous polymetallic sulfoxide.

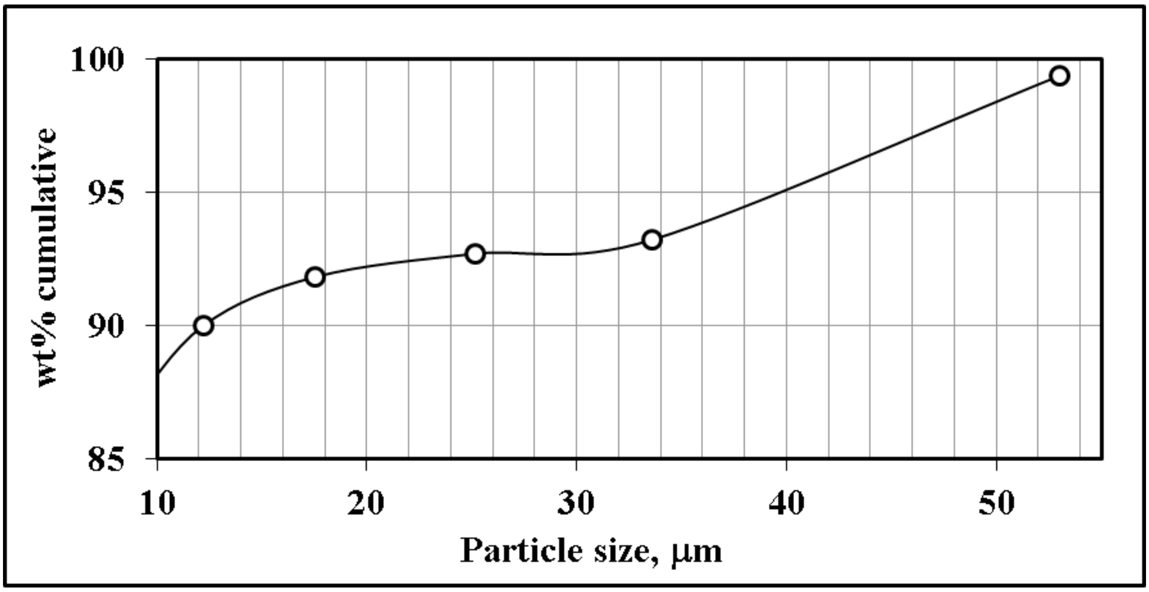

Figure 1. RP dust sample particles size distribution

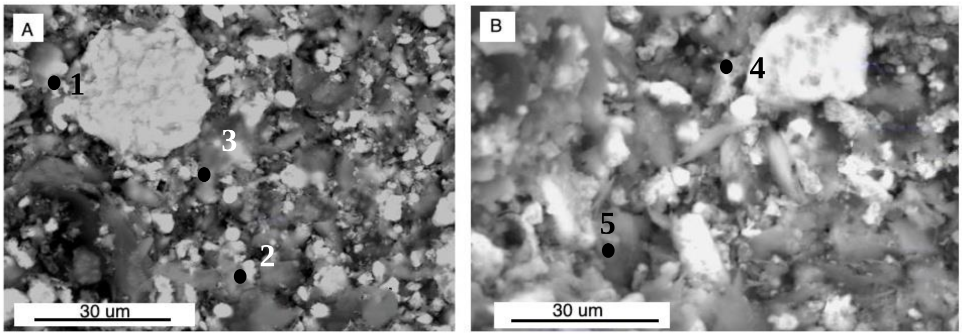

Figure 2. Scanning electron micrograph of RP dust (A) EDS analyzed points 1, 2 and 3 reported in Table 3, (B) EDS analyzed points 4 and 5 reported in Table 3.

Table 3. SEM/EDS analysis of RP dust (analysis condition: Acceleration voltage = 15 kV, beam current = 11 nA).

Nº	Normalized elemental composition (wt %)									
	O	Al	Si	S	K	Fe	Cu	Zn	As	Sb
1	4.9	1.4	2.0	10.5	0.4	19.1	60.6	--	1.1	--
2	27.8	1.5	2.6	6.2	0.3	28.4	23.6	0.4	7.6	1.6
3	17.7	1.9	2.5	10.2	0.4	21.2	41.1	0.9	3.3	0.8
4	26.5	2.3	2.4	8.8	0.3	24.3	29.1	--	5.2	1.1
5	4.2	1.5	2.3	15.4	0.5	23.9	47.1	--	4.1	1.0

The result of the X-ray diffraction (XRD) (XRD STOE, Model: STADI_MP, Detector DECTRIS MYTHEM 1K, Database Match) analysis is presented in Figure 3, which shows an amorphous material with the presence of a few peaks corresponding to complex phases of Cu-Fe-S-As-Al as gartrellite, johillerite, and kidwellite.

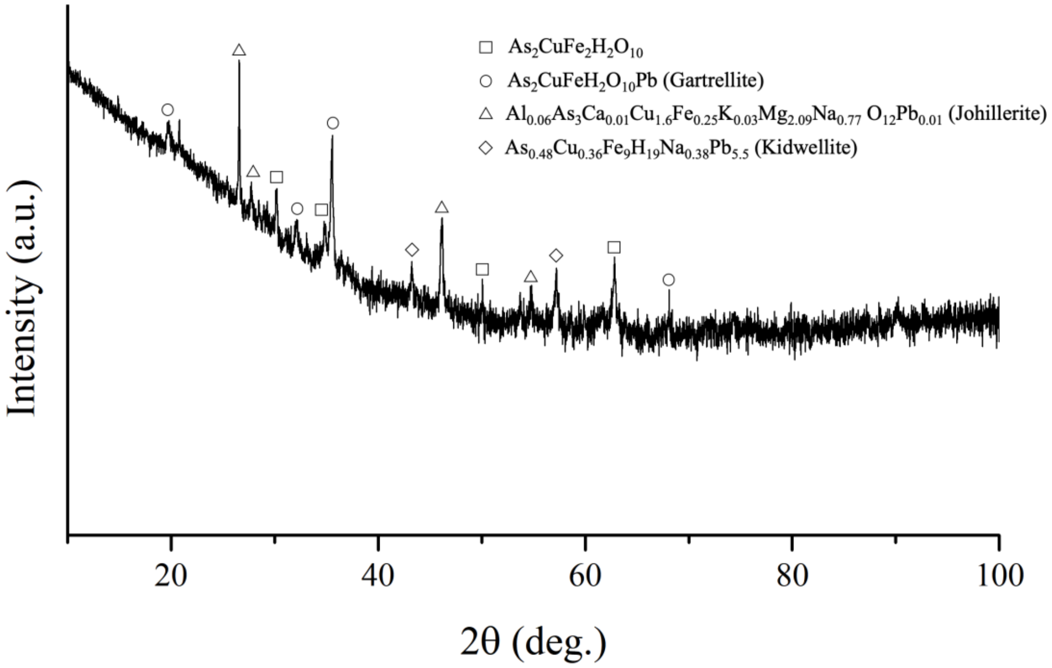

Figure 3. XRD analysis of RP dust (analysis conditions: t/step = 17 s, step = 2.1, 2θ = 10–100, time = 15 min).

A qualitative agreement exists between the results of QEMSCAN, SEM/EDS, XRD regarding the presence in the sample of Si-Fe-Cu-S-As-Al-O-K-Cl-Sb complex oxides described as Chenevixite, bornite, gartrellite, johillerite and kidwellite.

2.3. Experimental equipment and method

The experiments carried out used the same copper concentrate, equipment, and procedure previously described [13] with a reactor built from boron silicate glass to resist temperatures of 750 ° C, with a sintered glass disk (fritted), forming the base of the reactor bed. All experiments realized achieved a homogeneous mixture between the gases and the sample, to maintain a stable temperature for both, allowing the same operating conditions of an industrial fluidized bed roaster. 5 g of silica was as a bed on the fritted, and the sample used was 5 grams of copper concentrate or elemental sulfur mixed with RP dust placed on the silica bed. Then, inside a muffle preheated to 200 °C, the reactor was held. During the tests, the temperature of the reactor kept a range between 600 and 700 ° C, with gas (air, nitrogen, or SO_2) flow of 10 ml/min. The criterion used to select the flow of gases was to create the conditions of a fluidized bed in the reactor. Already the roasting time finished, the sample once extracted from the reactor was separated in a screen of 100 μm meshes. The oversized material contained the silica, and the undersize consisted of the calcined sample, whose elemental composition was determined by AAS.

As indicated in the previous paper [13], the percentage of volatilization of species (X) was defined as:

$$\% \text{ Volatilization } X = 100x\,([\text{wt } \% \text{ X}]\ \text{I}_{\text{nitial}} - [\text{wt } \% \text{ X}]_{\text{ final}}) / [\text{wt } \% \text{ X}]_{\text{ initial}} \qquad (1)$$

Where [wt % X] $_{\text{initial}}$ is the measured concentration of arsenic (or antimony) in the charged sample and [wt % X] $_{\text{final}}$ is the measured concentration of arsenic (or antimony) after the roasting process.

3. Results

3.1 Results of roasted mixture of copper concentrate/RP dust

Preliminary roasting experiments were performed at different conditions of mixtures of copper concentrate/RP dust at 700 °C. The temperature was selected according to the results obtained in the previous paper [13], where at 700°C was observed highest arsenic volatilization for the copper concentrate. The results indicated in Figure 4 includes a mass balance for the calculation considering a volatilization of arsenic of zero from the RP dust in a mixture of concentrate/RP dust, the results are indicated in this figure as squares while the experimental results of the mixture concentrate/RP dust as circles. By comparing the lines of trends, it is possible to state that only for mixtures with ratios of copper concentrate/FS dust above 50/50 had a significant volatilization of arsenic. It is interesting to observe that FS dust [13] and RP dust mixed with copper concentrate show a similar behavior in terms of arsenic volatilization. It is important to note that, for mixtures with ratios lower than 30/70, the volatilization of arsenic is negligible. Thus, from an operational point of view, a large proportion of copper concentrate would be required to volatilize the arsenic contained in the RP dust.

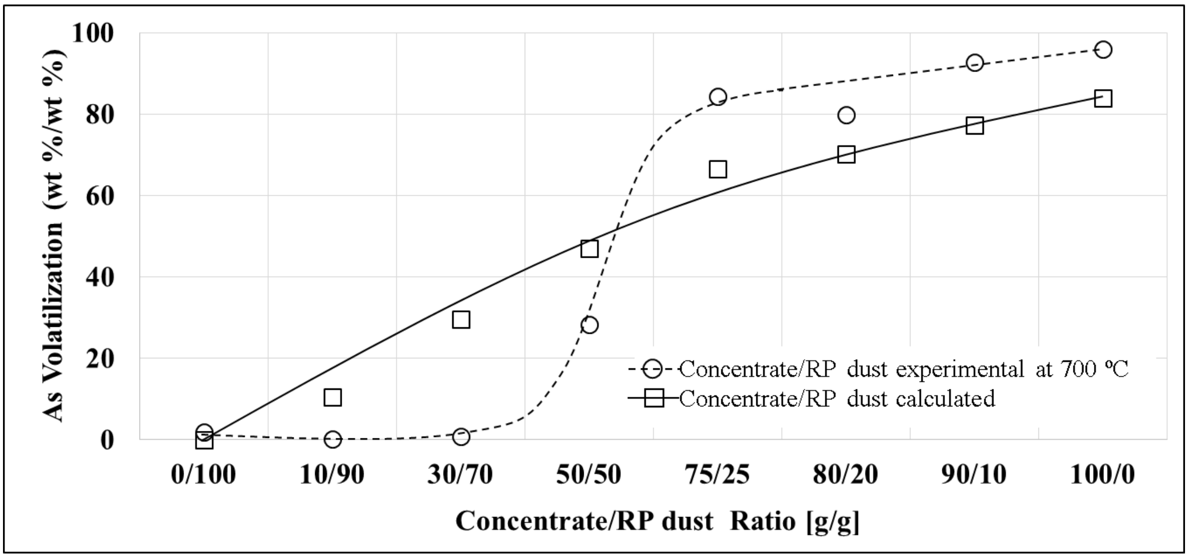

Figure 4. Volatilization of arsenic for different ratios of copper concentrate / RP dust mixture at 700 °C and 15 minutes of volatilization.

Based on the results of Figure 4, a set of experiments were carried out for the mixture of copper concentrate / RP dust in a ratio of 75/25, under a roasting time of 15 minutes varying the temperature in the range of 600 to 700 degrees. The experimental conditions, results of AAS chemical analysis of arsenic and volatilization of arsenic are includes. The table also comprises experiments for a mixture of sulfur/RP dust in a weight ratio of 5/95.

Table 4. Volatilization of arsenic for a mixture of copper concentrate/RP dust of 75/25 and Sulfur/ RP dust of 5/95, under different temperatures and roasting times

		Experimental conditions		Chemical Analysis (wt%)	
		Time	Temperature	As	As Volatilization
Mixture	Experiment	minutes	(°C)		
Copper concentrate/RP dust 75/25 (wt%/wt%)	1	2	600	2.9	34.8
	2	5	600	2.9	33.6
	3	10	600	3.0	33.0
	4	15	600	2.9	34.5
	5	2	650	2.9	34.3
	6	5	650	2.7	40.2
	7	10	650	2.3	47.9
	8	15	650	1.8	59.4
	9	2	700	2.2	49.9
	10	5	700	1.3	69.8
	12	10	700	0.5	88.0
	13	15	700	0.2	96.4
Sulfur/ RP dust 5/95 (wt%/wt%)	14	15	600	3.0	24.0
	15	15	620	1.7	57.3
	16	15	650	1.3	67.5
	17	15	700	1.4	65.8

Table 5 summarize the experimental results obtained at the conditions of 700 °C and 15 minutes includes in Table 4. Addition al experiments of mixtures of Fe_7S_8/RP and gas flow of nitrogen, air and SO_2 gases are included in the Table. For this set of experiments was includes the chemical analysis and volatilization of As and Sb.

Table 5. Composition and arsenic volatilization for different samples and mixtures at 700 °C and roasting time of 15 minutes

	Experimental Condition		Chemical composition (wt%)					Volatilization	
Experiment	Mixture	Atmosphere	Cu	Fe	S	As	Sb	As	Sb
18	RP Dust	Nitrogen	18.2	14.0	2.2	3.9	0.8	0.0	28.6
19	RP Dust	Air	16.2	14.1	2.5	4.2	0.7	0.0	33.3
20	RP Dust	SO_2	----	----	----	4.0	0.6	0.0	44.8
13*	Copper concentrate/ RP Dust	Air	40.8	11.8	17.2	0.2	0.4	96.4	27.6
21	Sulphur/RP Dust	Nitrogen	18.5	12.4	5.4	1.4	0.3	64.0	69.0

17*	Sulphur/RP Dust	Air	18.9	13.2	6.1	1.4	0.3	65.8	72.5
22	Fe_7S_8 /RP Dust	Air	----	----	1.9	1.8	0.3	0.0	15.0

*Experiments included in Table 4

The results in Table 5 indicated negligible arsenic volatilization for the RP dust in atmospheres of nitrogen, air, and SO_2. However, the antimony reached volatilizations of 29, 33.3, 45 wt% in the mentioned atmospheres, respectively. The pyrrhotite/RP dust mixture under the air atmosphere obtained negligible volatilization of arsenic and antimony. For copper concentrate/RP dust mixture achieved a volatilization of arsenic of 96% by weight with a low volatilization of antimony close to 28 wt. The sulfur /RP dust mixture showed significant volatilization of arsenic and antimony in both atmospheres, air, and nitrogen.

3. Discussion

Figure 5 represents data for the mixture of the copper concentrate/RP dust with results indicating a strong dependence on the volatilization of arsenic concerning the temperature and roasting time. Arsenic volatilization above 90 % obtained at temperatures of 700 °C and a roasting time of 15 minutes.

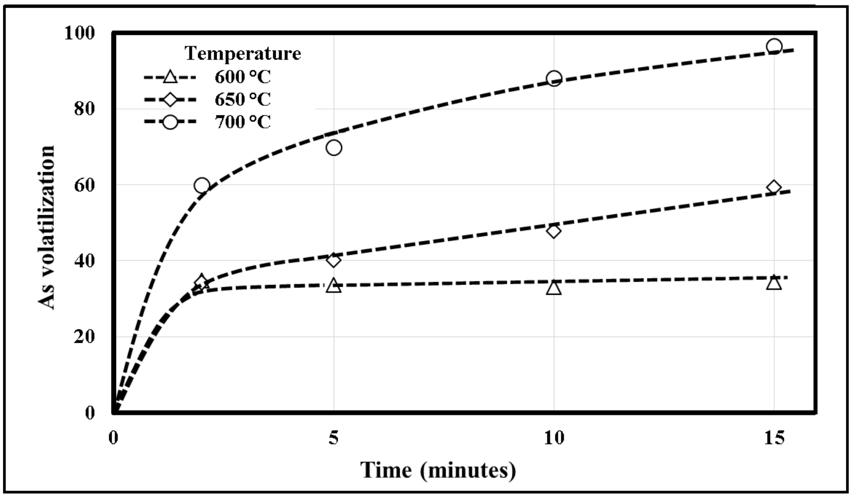

Figure 5. Relationship between the volatilization of arsenic and roasting time for a weight ratio of a mixture concentrate/RP dust of 75/25 at temperatures of 600, 650 and 700 °C.

Figure 6 represents the experimental results for a reaction time of 15 minutes for the mixture of the copper concentrate/RP dust and sulfur/RP dust. For the sulfur/RP dust mixture, the volatilization of arsenic peaked at approximately 65 wt% at 650 ° C, higher temperature values did not show any increment on the arsenic volatilization for this mixture. Further studies are recommended to improve the arsenic volatilization on the sulfur / RP dust mixtures. One option is to perform roasting tests at higher ratios of sulfur/RP dust.

The reaction mechanism of sulfidation-decomposition-oxidation proposed by SEM/EDS analysis in roasted mixtures with RP dust could not be confirmed. The results obtained indicate the absence of clear enargite/tennantite crystals. However, as indicated in Table 5, the experiment with nitrogen and air atmospheres for the sulfur/RP dust mixture showed remarkably similar results (about 65%) of arsenic volatilization for both tests. These results can compare with those reported in the same table for RP dust roasted in a nitrogen (or air) atmosphere without the presence of sulfur, where a zero volatilization of arsenic obtained. Therefore, it is possible to infer a process of sulfidation-volatilization of arsenic. However, verification of the reaction mechanism may be subject to further study. Some questions require answers: why the mixtures with copper concentrate reached the highest arsenic volatilization? Why pyrrhotite/RP dust and mixture obtained zero volatilization of arsenic? What is the reason to explain that the volatilization of arsenic of sulfur with FS dust is only a 10%, compared with 65% for the RP dust?.

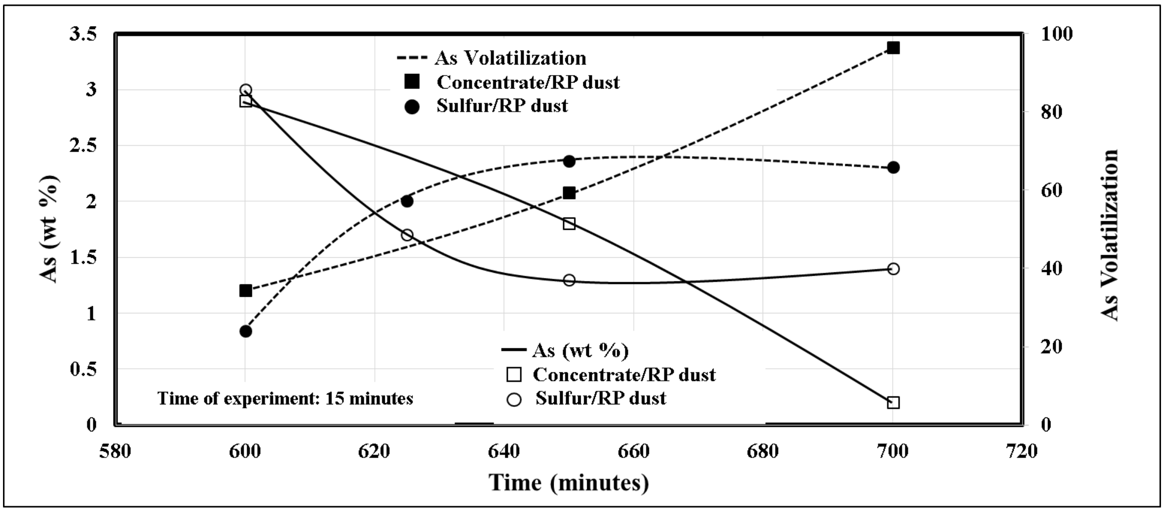

Figure 6. Volatilization of arsenic from mixtures of copper concentrate/RP dust and Sulfur/RP dust vs. temperature

5. Conclusions

The characterization of RP dust indicated a morphological composition different from FS dust. Meanwhile, the RP dust is composed of fine agglomerated particles (90 wt% <10 µm) the results indicated that the FS dust is composed of particles with sizes between 10 and 100. The RP dust corresponds to an amorphous materials and polymetallic sulfoxide. The FS dust contain particles with a glassy matrix and crystals of iron oxides (hematite/magnetite) and also complex polymetallic materials

By comparing the data for the two papers, the differences found are the mixture of copper concentrate/FS dust reached volatilization of arsenic of more than 90%, at a low roasting temperature of 600 ° C. In contrast, a mixture of copper concentrate / RP dust required a roasting temperature of than 700 ° C but achieved a higher arsenic volatilization of 96%. Arsenic concentration after the roasting process was less than 0.3wt% for both mixtures of RP dust as FS dust. Thus, despite the initial differences between FS dust and RP dust, after roasting in mixtures with concentrate or sulfur, both materials are appropriate material to be recirculated into smelting processes. The combined process of roasting of concentrate/dust and recycling in smelting has the potential to significantly reduce the tons of toxic solid waste and increase the recovery of valuable metals.

Another option is the recycling of FS dust and RP dust into a high arsenic concentrate fluidized bed roasting furnace. However, due to the size of the dust, operational inconveniences in the gas cleaning system and sulfuric acid production equipment can be caused. Thus, this work recommends the construction of an independent roasting furnace for the treatment of metallurgical FS and RP dust.

Given the promissory results obtained with two kinds of metallurgical dust in the elimination of arsenic, it is reasonable to extend the investigation for a similar treatment for the dust generated in the process of roasting high arsenic gold and nickel concentrates. Another line of research correspond to explore the recovery of silver in the gas phase exploring the differences of temperature precipitations of different compounds of silver, arsenic and antimony. The paramount importance to complement the present work is research the fixation of the residual arsenic (antimony and bismuth) in a glassy slags matrix.

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