# TREATMENT OF COPPER FLUE DUST FROM FLASH FURNACE WASTE HEAT BOILER FOR IMPURITIES CONTROL

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## ABSTRACT

During the pyrometallurgical processing of copper sulphide raw materials, part of the charge leaves the furnace space together with the exhaust gases in the form of dust entrainment. Volatile components also pass into the dustgas flow, due to which it is enriched with impurities, some of which are harmful to the technological process. The formed dust-gas flow passes through dust collection equipment, where the main part of it is captured. Most often, the captured dust is recirculated, which leads to a decrease in the productivity of the furnace unit and a gradual increase in the content of impurities in the condensed products of the smelting.

To overcome these disadvantages, it is necessary to take part or all the recirculating dust out of the melting cycle and process it independently to extract the valuable metals and dispose of the harmful substances.

In metallurgical practice, industrial application for the processing of recirculating dust entrainment has mainly been accomplished by hydrometallurgical methods based on treating the flue dust in aqueous solutions of acid or alkaline reagents.

In the present research work, laboratory results on hydrometallurgical leaching of flue dust from a flash furnace waste heat boiler (FF-WHB) are presented. The effect of the main technological parameters affecting the degrees of recovery of the main metals in solution was studied. Based on the experimental results, the optimal conditions for hydrometallurgical treatment of the FF-WHB flue dust were determined. By treating the dust under optimal conditions, the main tasks of the research are achieved - removal of impurities harmful to the technological process and reduction of the amount of flue dust processed in the flash furnace.

Keywords: flash smelting, waste heat boiler, copper flue dust, sulfuric acid, leaching.

### **INTRODUCTION**

Globally over 80% of the primary copper production comes from smelting of sulphide concentrates [1]. All smelting furnaces generate flue dusts which are captured in the gas handling equipment.

The most common method for handling with the flue dust is its recirculation in the smelting furnace. If copper dust is directly returned to smelting furnace, the content of the impurities in the feed materials could rise significantly and the processing capability of furnace will be reduced. The process of copper electrorefining and the quality of copper cathodes will also be affected greatly by the accumulation of the recirculating impurities [2]. Among the most harmful impurities in the copper smelting and refining processes are arsenic, antimony, bismuth, lead, and zinc. Methods for concentrate pretreatment like blending with pure concentrates and roasting are applied to prevent impurity increment. Impurities can also be controlled by using slagging at the smelter and electrolyte purification in the copper refinery. The removal of impurities is crucial in copper metallurgy to produce high-quality copper, but it is also of great importance from an environmental aspect. Due to the constant increase in impurities in copper ores over the years, an even more critical situation can be reasonably expected in the future. It is essential to keep impurities below the levels accepted by national and international standards for the copper industry, and to decrease the total emission of harmful components. Currently, the majority of smelters around the world face this problem [3].

Therefore, in case of high impurity levels, it is necessary to separate flue dust from the copper smelting system and reclaims valuable metals. The hydrometallurgical treatment of flue dusts (from smelting and/or converting unit) is a potential way to control the impurity levels. Morales et al. assumed that an ideal process for the treatment of copper smelter flue dusts should be a hydrometallurgical process, operating at room temperature and atmospheric pressure, with dilute reagents, few unit operations and low energy costs [4].

The choice of a suitable hydrometallurgical technology for processing of these dusts depends exclusively on their chemical and mineralogical composition. The hydrometallurgical methods for treatment of the dusts can be divided in two main groups - acidic and alkaline methods, as well as by applying bioleaching [5 - 9]. The reagent used most frequently by the acidic methods is sulfuric acid, and by the alkaline methods - sodium hydroxide.

In the case of sulfuric acid treatment, the copper, zinc, cadmium and iron sulphates and arsenic oxides are solubilised. The lead and silver compounds present in the flue dust remain in the leach residue. The alkaline methods have not found wide application till the present day. From economical viewpoint copper from the flue dust must be recovered. This can be done in various ways such as copper cathodes, precipitate, or as copper sulphate, which is a powerful coolant when returned to the smelting furnace [10].

The aim of this study is to investigate the possibility

of hydrometallurgical removal of the harmful impurities from FF-WHB flue dust and reduction the amount of recirculating dust.

#### **EXPERIMENTAL**

A sample from flash furnace waste heat boiler (FF-WHB) dust was provided by a copper smelter. The representative sample was characterized by chemical, qualitative XRD and granulometric analyses. The chemical analysis considering the main metals was determined using inductively coupled plasma optical emission spectroscopy (ICP-OES), using an iCAP 7000 series from Thermo Scientific. The results from ICP-OES are shown in Table 1.

As can be seen from Table 1, the concentration of copper and iron in the studied material is the highest. Besides these, it contains zinc, bismuth, arsenic, cadmium and small amounts of antimony, nickel and selenium. The concentration of the trace metals is not presented. A characteristic feature of the flue dust is the presence of sulphur mainly in sulphate form.

A mineralogical study of the dust is carried out by means of X-ray diffraction. Qualitative XRD was carried out on a Rigaku Dmax C Diffractometer. According to the applied diagram of the material (Fig. 1) the identified phases in it are: magnetite, hematite, chalcanthite, goethite and delafossite.

Granulometric analysis was performed by means of laser ray diffraction using Mastersizer 3000 apparatus from Malvern Panalytical. The samples were prepared in a solid-isopropyl alcohol suspension to avoid particle agglomeration. The results are shown in Fig. 2 and reveal that the particle size range is wide, and the maximum size was over 3000  $\mu$ m. The FF-WHB dust consists of two fractions - fine up to 300  $\mu$ m (around 70 % of the dust mass) and coarse 1000 - 3000  $\mu$ m, which shows that the mechanism of dust formation is complex. Evidently there are two types of dust in the process off gases: a) mechanically formed dust consisting of small particles of the charged material which are carried away by the

Table 1. Chemical composition of the FF-WHB dust, %.

Cu	Fe	As	Cd	Pb	Bi	Zn	S <sub>tot</sub>	S <sub>SO4</sub>	Sb	Ni	Se
22.00	18.46	1.23	0.27	0.64	0.11	1.75	7.58	7.17	0.03	0.07	0.004



Fig. 2. Size distribution for FF-WHB dust.

process technological gas; b) chemically formed dust consisting of vaporized components that condense into particles as the gas cools.

## **RESULTS AND DISCUSSION**

The hydrometallurgical treatment of FF-WHB flue dust is carried out in three series - i) water leaching; ii) leaching with synthetic sulfuric acid solutions and iii) leaching with weak scrubbing acid.

The experiments were carried out in a glass flask placed in a thermostat to maintain the set temperature, and the stirring was carried out with a mechanical stirrer with adjustable revolutions (Fig. 3). Sample of 20 or 40 g dust was mixed with 200 mL leaching liquid for 60 min at agitation  $300 \pm 5$  min<sup>-1</sup>.

After completion of the experiments the obtained pulp is filtered on vacuum pump and the residue is desiccated at 80°C for 24 hours and weighed on an assay balance. At all experiments, the solid phase obtained as a result of the hydrometallurgical leaching tests was analysed for determination the concentration of the main metals (Fe, Cu, As, Bi, Zn, Pb and Cd) by means of ICP-OES analysis. Based on these results, the recovery



Fig. 3. Picture of the experimental equipment.

rate of metals into solution was calculated.

In this study the sample of FF-WHB dust was leached with water at 80°C temperature to determine the quantity of water-soluble compounds in the dusts. The experiments on leaching FF-WHB dust in synthetic sulfuric acid solutions aim to investigate the effect of temperature, sulfuric acid concentration, and solid:liquid ratio on main metals recovery rates and their distribution between the solution and the leach residue. Leaching the dust with real production solution (scrubbing acid from the gas scrubbing/cooling section of sulfuric acid plant) aims to evaluate the possibility for practical implementation of the process (tests 15 - 20).

The conditions of the conducted experiments are given in Table 2, and the results of the laboratory experiments are presented in Table 3.

The main priority of the research is an assessment of the possibility of removing harmful impurities (mainly arsenic) in solution and reduction the amount of recirculating dust.

		Parameter								
Test №	Initial mass of the	Concentration of	Temperature	Solid : liquid ratio						
	sample, g	$H_2SO_4$ , g L <sup>-1</sup>	T,⁰C							
1	2	3	4	5						
1	20.0	100	40	1:10						
2	40.0	100	40	1:5						
3	20.0	100	60	1:10						
4	40.0	100	60	1:5						
5	20.0	100	80	1:10						
6	40.0	100	80	1:5						
7	20.0	50	40	1:10						
8	40.0	50	40	1:5						
9	20.0	50	60	1:10						
10	40.0	50	60	1:5						
11	20.0	50	80	1:10						
12	40.0	50	80	1:5						
13	20.0	Water	80	1:10						
14	40.0	Water	80	1:5						
15	20.0	15	40	1:10						
16	40.0	15	40	1:5						
17	20.0	15	60	1:10						
18	40.0	15	60	1:5						
19	20.0	15	80	1:10						
20	40.0	15	80	1:5						

Table 2. Parameters of the conducted laboratory experiments on dissolution of FF-WHB dusts with synthetic and production solutions.

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Degree of extraction in solution	Cd	%	17	85.07	81.47	86.24	81.05	85.80	80.62	84.55	80.73	85.01	79.50	84.00	78.19	74.74	72.53	76.17	72.13	76.53	74.16	76.77	74.19
	Bi	%	16	71.21	66.49	73.46	61.35	72.00	63.29	55.32	53.55	56.66	42.98	57.95	43.68	0.01	0.00	9.74	0.00	0.00	0.00	0.00	0.00
	Zn	%	15	44.73	44.00	47.99	44.46	50.41	48.74	51.39	47.98	48.45	45.50	47.06	46.90	49.90	48.85	51.16	50.55	52.68	53.76	53.15	54.08
	Pb	%	14	4.92	0.00	9.45	0.00	1.98	0.14	11.38	3.14	4.56	0.00	2.49	4.07	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	As	%	13	84.27	83.39	91.24	88.48	92.92	90.88	79.66	75.32	84.21	77.50	85.96	82.22	41.40	39.29	76.84	72.86	77.55	72.02	76.69	69.41
	Cu	%	12	78.25	78.05	81.07	79.47	84.49	83.62	76.50	74.60	78.03	75.19	80.21	78.22	68.88	66.71	70.40	69.43	72.62	71.44	72.92	70.76
	Fe	%	11	21.32	22.22	28.43	25.71	31.98	30.37	24.48	23.42	25.38	20.24	26.96	23.46	10.88	13.26	16.09	17.64	19.67	19.69	19.20	19.12
	Cd	%	10	0.10	0.12	0.10	0.13	0.11	0.14	0.10	0.12	0.10	0.13	0.11	0.14	0.14	0.15	0.14	0.16	0.14	0.15	0.14	0.15
	Bi	g/t	6	0.08	0.09	0.08	0.11	0.09	0.11	0.12	0.12	0.12	0.15	0.12	0.15	0.23	0.25	0.22	0.25	0.26	0.28	0.27	0.28
position	Zn	%	8	2.40	2.35	2.45	2.47	2.49	2.40	2.04	2.10	2.23	2.24	2.36	2.21	1.80	1.81	1.86	1.84	1.83	1.74	1.83	1.73
idue com	Ъb	%	7	1.51	1.56	1.56	1.65	1.80	1.71	1.36	1.43	1.51	1.54	1.59	1.46	1.60	1.37	1.58	1.48	1.62	1.53	1.58	1.49
Solid res	$\mathbf{As}$	%	9	0.48	0.49	0.29	0.36	0.25	0.30	0.60	0.70	0.48	0.65	0.44	0.52	1.48	1.51	0.62	0.71	0.61	0.74	0.64	0.81
	Cu	%	5	11.87	11.58	11.21	11.42	9.79	9.64	12.40	12.89	11.95	12.82	11.09	11.39	14.06	14.81	14.17	14.30	13.31	13.51	13.30	13.85
	Fe	%	4	36.04	34.43	35.56	34.85	36.03	34.39	33.43	32.61	34.05	34.58	34.35	33.60	33.78	32.38	33.71	32.33	32.77	31.88	33.29	32.14
% ər	Solid esidı, ield,	л г	Э	40.30	41.70	37.15	39.35	34.85	37.37	41.70	43.35	40.45	42.57	39.25	42.05	48.70	49.45	45.95	47.02	45.25	46.50	44.80	46.45
Solid Solidue, g		ıc	2	8.06	16.68	7.43	15.74	6.97	14.95	8.34	17.34	8.09	17.03	7.85	16.82	9.74	19.78	9.19	18.81	9.05	18.60	8.96	18.58
Test Nº		1	1	2	ю	4	5	9	7	8	6	10	11	12	13	14	15	16	17	18	19	20	

#### Water leaching of FF-WHB dust

Experiments were carried out at a temperature of 80°C, agitation rate of 300 min<sup>-1</sup>, solid:liquid ratio of 1:5 and 1:10, for a period of 60 min. The purpose of the experiments is to determine the number of water-soluble compounds in the flue dust. The experimental results are presented graphically in Fig. 4.

Arsenic in the flue dust can exist as  $As_2O_3$  or  $As_2O_5$ . As the data presented below depict, the solubility of  $As_2O_3$ in water is significantly lower than that of  $As_2O_5$  [11]:  $As_2O_3$  - from 12.0 to 37.0 g L<sup>-1</sup> at 20°C;  $As_2O_5$  - 658 g L<sup>-1</sup> at 20°C.

Arsenic pentoxide is readily soluble in water, yielding arsenic acid:

$$As_2O_5 + 3H_2O \rightarrow 2H_3AsO_4 \tag{1}$$

Copper in the FF-WHB dust is predominantly in the form of sulfate (sulphating air is blown in WHB), which is easily soluble in water. According to the results of the chemical analyses of the solutions 66.71 and 68.88 % of the copper passes into it, as shown in Table 3 (tests 13 and 14). Zinc, arsenic and cadmium are also partially in the form of water-soluble compounds. Iron degree of recovery was negligible, as lead and bismuth remained in the solid residue.

As a result of the water leaching, the pH of the resulting solutions diminished to 2.4 and 2.3 respectively. The mass of water-soluble compounds was about 50 % of the initial mass of FF-WHB dust.

#### Leaching FF-WHB dust with synthetic H,SO<sub>4</sub> solutions

The experiments on leaching FF-WHB dust in synthetic sulfuric acid solutions aim to investigate

the effect of main technological parameters on metals recovery rates and their distribution between the solution and the leach residue.

#### Effect of initial H<sub>2</sub>SO<sub>4</sub> concentration

The tests were carried out at a temperature of 40, 60 and 80°C, solid:liquid ratio of 1:10 and 1:5, agitation 300 min<sup>-1</sup> for 60 min. From the results presented in Fig. 5, depending on the acidity the metals extraction degree changes significantly. The maximum degree of metal extraction is achieved at 100 g L<sup>-1</sup> H<sub>2</sub>SO<sub>4</sub> and a temperature of 80°C. The highest bismuth and cadmium degree of extraction is achieved at a concentration of 100 g L<sup>-1</sup> H<sub>2</sub>SO<sub>4</sub> and a temperature of 40°C. Zinc degree of recovery changes only by 4 %.

#### Effect of the temperature

### Initial $H_2SO_4$ concentration - 50 g $L^{-1}H_2SO_4$

The laboratory experiments were conducted at initial sulphuric acid concentration of 50 g L<sup>-1</sup>, temperature of 40, 60 and 80°C, solid:liquid ratio of 1:10 and 1:5, agitation 300 min<sup>-1</sup> for 60 min. It is evident from the test results that with the increase of temperature a minor increase in the extraction degrees of all metals is observed (Fig. 6.). Considering the insignificant increment of the metals recovery degree it can be concluded that temperature has a minor effect on metals dissolution at 50 g L<sup>-1</sup> H<sub>2</sub>SO<sub>4</sub> initial acidity.

#### Initial $H_2SO_4$ concentration - 100 g $L^{-1}H_2SO_4$

The effect of this parameter was studied at initial



Fig. 4. Degree of Fe, Cu, As and Bi extraction upon water leaching.



Fig. 5. Effect of initial  $H_2SO_4$  concentration on the degree of metals recovery at solid:liquid ratio = 1:5.



Fig. 6. Effect of temperature on the degree of metals recovery -  $50g L^{-1} H_2SO_4$  at solid:liquid ratio = 1:10.

sulphuric acid concentration of  $100 \text{ g L}^{-1}$ , temperature 40, 60 and 80°C, solid:liquid ratio of 1:10 and 1:5, agitation 300 min<sup>-1</sup> for 60 min. From the data presented in Fig. 7, with the rise of temperature the extraction degrees of Fe, Cu, As and Bi increases. The increment of metals recovery is within 10 %. Only the degrees of Bi and Cd extraction did not increase at temperature of 80°C, which is due to the effect of PbSO<sub>4</sub> formed in the pulp. The pulp contains CaSO<sub>4</sub>, which also has the ability to co-precipitate metals like bismuth and cadmium.

#### Effect of solid:liquid ratio

Analysis of the results obtained in the laboratory tests showed that the reduction of the solid: liquid ratio (respectively an increase in the amount of solid phase in the solution) leads to a decrease in the extraction rate of the metals (Fig. 8). This influence is most pronounced on the extraction rate of bismuth and cadmium, which decreases the most with an increase in the amount of solid phase in the solution.

#### Leaching FF-WHB with scrubbing acid

Laboratory experiments on leaching the FF-WHB dust with real production solution (scrubbing acid from the gas scrubbing/cooling section of sulfuric acid plant) are conducted for evaluation the practical possibility for implementation of the leaching process. A sample of 10 L of scrubbing acid from the sulphuric acid plant was provided. Table 4 presents the chemical analysis of the scrubbing acid used in this study.

The effect of temperature and solid:liquid ratio on the distribution of main metals between the solution and the solid residue was investigated. The duration of dissolution process in all experiments was 60 min.



Fig. 7. Effect of temperature on the degree of metals recovery -  $100g L^{-1} H_2SO_4$  at solid: liquid ratio = 1:10.



Fig. 8. Effect of solid:liquid ratio on the degree of metals recovery at 100 g  $L^{-1}$  H<sub>2</sub>SO<sub>4</sub>.

and the agitation rate was 300 min<sup>-1</sup>. The effect of temperature is shown graphically on Fig. 9.

It has been found experimentally that the extraction degrees of Fe, Cu and As in the solution increases slightly with increasing the solution temperature. Bismuth is extracted insignificantly only at a temperature of 40°C, at a higher temperature Bi practically does not pass into the solutions. This fact is due to the low final acidity of the solutions (around pH = 1) and the presence of compounds such as  $PbSO_4$  and  $CaSO_4$  in the pulp, which co-precipitates metals like bismuth, cadmium, arsenic, cobalt etc.

The influence of the solid:liquid ratio on the extraction degree of FF-WHB dust with scrubbing acid is illustrated on Fig. 10.

Experimental results showed that an increase in the amount of solid phase in the solution leads to a slight

decrease in the extraction rate of iron and copper. The extraction rate of arsenic is affected to a greater extent by this parameter. Bismuth passes into the solution only at a temperature of 40°C and a ratio of solid:liquid = 1:10. This parameter has negligible effect on zinc and cadmium recovery degrees (tests 16 - 20 in Table 3).

Based on the results from the carried out tests on leaching the dust with scrubbing acid is evident that iron degree of recovery did not exceed 20 %. The optimal conditions for dust leaching with scrubbing acid are: temperature 40°C, solid:liquid ratio = 1:10 and test duration 60 min. At these conditions iron and arsenic concentrations in solution are 6.51 g L<sup>-1</sup> Fe and 2.36 g L<sup>-1</sup>. As respectively, which corresponds to a Fe:As molar ratio of 3.7. Excessive iron dissolution will lead to precipitation of high quantities of hazardous waste, during solution purification step, instead of smelting slag which

Cu	As	Sb	Bi	Pb	Cd	H <sub>2</sub> SO <sub>4</sub>	
0.026	0.57	0.0009	0.0196	0.023	0.058	15.0	
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	٦	1					
	Bi 0	0.74					
		9.74					

Table 4. Chemical composition of the scrubbing acid, g L<sup>-1</sup>.

As

Cu

Fe

0

192

20

19.67

40

Degree of recovery, %



60

80

100



Fig. 10. Effect of solid:liquid ratio on the degree of metals recovery.

can find various applications. The final pH of the solution diminished to pH = 1.04 with copper concentration of 30.55 g L<sup>-1</sup> Cu. This pH value is appropriate for direct copper solvent extraction from the leach solution.

Based on the experimental findings the process to be proposed for the treatment of FF-WHB dust can include the following steps: i) Leaching the dusts with scrubbing acid (15 g  $L^{-1} H_2 SO_4$ ), where 53 % of dust is dissolved; ii) Copper recovery from the leach solution by solvent extraction and electrowining; iii) Precipitation of the arsenic from the resulting solution, such as crystalline or amorphous ferric arsenate [12]; iv) Recycling of the leach residue to the flash furnace after drying. This proposed process could be integrated with industrial Wastewater treatment plant (WWTP) removing arsenic in the form of ferric arsenate.

■ 1:10, 80°C
■ 1:10, 60°C

■ 1:10, 40°C

#### CONCLUSIONS

Based on the conducted laboratory study of the process of hydrometallurgical treatment of a representative sample of FF-WHB flue dust, the following conclusions can be drawn:

• Cadmium passes predominantly into the solutions regardless of the test conditions (its minimum degree of extraction is 72.53 %.

- Copper degree of extraction is slightly influenced by the technological parameters of the process and ranges from 66.71 to 84.4 9 %.
- Lead dissolves negligible in sulfuric acid medium and mainly concentrates into the solid residue. Part of the lead sulphate passes through the filter paper during filtration of the solution, because of which the degree of its dissolution is calculated up to 11.38 %.
- Arsenic dissolution is greatly affected by the conditions of sulfuric acid treatment. Arsenic dissolves best at increased acidity and temperature. A maximum extraction rate of 92.92 % was achieved when dissolving the dust with a sulfuric acid solution with a concentration of 100 g  $L^{-1}$  H<sub>2</sub>SO<sub>4</sub> and a ratio of solid:liquid = 1:10.
- The degree of bismuth dissolution increases with increasing temperature and decreasing pulp density. As the solution acidity decreases from 100 to 15 g L<sup>-1</sup> H<sub>2</sub>SO<sub>4</sub>, its extraction degree decreases significantly (not exceeding 9.74 %).
- The degree of iron extraction in solution increases with increasing initial acidity, temperature and decreasing pulp density, reaching a maximum value of 31.98 %.
- Zinc is only partial leached during the conducted experiments with recovery rate between 44.00 and 54.08 %.
- The optimal conditions for dissolving the FF-WHB dust are the following: temperature 40°C, duration 60 min., solid:liquid ratio = 1:5, reagent scrubbing acid (15 g  $L^{-1}$  H<sub>2</sub>SO<sub>4</sub>). Under these conditions, 72.86 % of arsenic extraction is achieved with low degree of iron dissolution (17.64 %), resulting in solution with Fe:As molar ratio of 3.7.
- The main valuable component in the production solution is copper, which can be recovered from the leach solution as cathode by solvent extraction and electrowining.
- The obtained undissolved residue has a 47.02 % of the mass of the initial sample. It can be recycled to the flash furnace as coolant of the reaction shaft.
- The single-stage sulfuric acid treatment of FF-WHB dust with scrubbing acid provides a solution to the main tasks of the research removal of the impurities harmful to the technological process and reduction of the amount of recirculated material processed in the flash furnace.

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