The American Journal of Applied sciences
(ISSN – 2689-0992)
Published: October 30, 2021 | **Pages:** 18-30
Doi: https://doi.org/10.37547/tajas/Volume03Issue10-05

IMPACT FACTOR
2021: 5. 634

OCLC - 1121105553

Journal Website: https://theamericanjournals.com/index.php/tajas

Technology For Processing Industrial Waste Containing Non-Ferrous Metals

Zulfizar Mirzanova
Head Of The Laboratory Of Metallurgical Research And Production Of High Purity Metals Of The Scientific And Technological Center Under The Association Of Rare Metals And Solid Alloys JSC "Almalyk MMC", Uzbekistan

Abdurashid Hasanov
DSc, Professor, Deputy Chief Engineer For Science Of JSC AMMC, Uzbekistan

ABSTRACT

The article presents the results of analyzes to study the possibility of extracting valuable components from industrial waste from non-ferrous metallurgy. Of particular interest for the production of new types of products, in particular lead and additional extraction of precious metals (gold, silver), are the fine converter dust of the copper smelting plant of Almalyk MMC, the accumulation of which necessitated the search for technical solutions for their processing. Dust captured in electrostatic precipitators mainly contains metal sulphates, which violate the autogenousness of the melt and contribute to a decrease in the process temperature.

KEYWORDS

Sulfurous Gas, Sulfuric Acid, Slurry, Converter Dust, Vanyukov Furnace, Copper, Lead, Zinc, Autogenous Process, Concentrate, Leaching, Extraction.

INTRODUCTION

The mining and metallurgical industry of Uzbekistan is one of the important branches of development, which is unthinkable without the development and implementation of improved and new technologies for the processing of mineral raw materials, in particular polymetallic ores, in order to comprehensively extract valuable components. the largest mining and metallurgical enterprises in Central Asia. The production capacity of the plant is based on the reserves of a group of porphyry copper, lead-zinc and gold-silver deposits located in the Tashkent, Jizzakh, Namangan and Surkhandarya regions of the Republic of Uzbekistan. In the action strategy for the further development of the Republic of Uzbekistan "The tasks of raising the industry to a qualitatively new level, deep processing of local sources of raw materials, accelerating the production of finished products, mastering new types of products and technologies" - important tasks are set. In this aspect, scientific research aimed at improving and developing new technologies for the complete extraction of non-ferrous and precious metals from the dust of copper smelting of the CMP (Copper melting plant) of the Almalyk Mining and Metallurgical Combine is of great importance. The production of metals from industrial waste plays an important role in the overall balance of their production and consumption in our country. One of the problems in copper smelting is the capture, cleaning and disposal of dust and gas emissions.

In the copper-smelting production of Almalyk MMC, when melting a copper charge, a dust and gas flow (GWP) is formed, consisting of sulphurous gas, coarse (85% of the gross dust volume) and fine dust (15%) [1].

In the dust of metallurgical units with a large volume of waste gases (shaft furnace, converter, Vanyukov furnace and others), there is a high (up to several percent) concentration of non-volatile metals (copper, lead, nickel, iron, and others), which are mainly represented by particles of the processed charge or the resulting products (matte, slag) Of particular interest for the production of new types of products, in particular lead and additional extraction of noble metals (gold, silver), are the fine converter dust of the copper smelter of Almalyk MMC, the accumulation of which necessitated the search for technical solutions for their processing.

Dust captured in electrostatic precipitators mainly contains metal sulphates, which violate the autogenousness of the melt and contribute to a decrease in the process temperature.

OBJECTS AND METHODS OF RESEARCH

The study by laboratory experience the composition of the dust obtained during the converter processing of copper mattes is shown in Table 1. Fine converter dust of copper-smelting production is a white powder with a particle size of -0.02+0.1 mm (80-90%) with a bulk density of 1.3 g/cm^3.

Table 1

Results of chemical analysis of converter dust

Component	Content, %	Component	Content, %
Copper	1,9-2,3	Cadmium	0,19
Lead	16-50	Silica	0,65
Zinc	9-14,7	Magnesium oxide	0,33
Total sulfur	11,47	Calcium oxide	2,84
Sulphate sulfur	8,52	Gold	1 г/т
Gland	0,46	Silver	170-200 г/т

The main theoretical premise justifying the autonomous processing of fine dust by chemical enrichment methods is the phase-chemical composition of dust and the futility of secondary processing of the material where it was formed.. [2]

Dust processing technological schemes used at different plants differ from each other. The study of most of the existing schemes abroad and the domestic scheme is based on the selective dissolution of non-ferrous metals contained in the dust. In this case, noble metals remain in the undissolved residue, which is sent to refining production. A solution containing sulphates or chlorides of non-ferrous metals, in the main production. On the territories of Almalyk MMC, fine dust from the copper smelter is accumulating, which are technogenic deposits of polymetallic raw materials of unique composition, which are practically not used at present.

Technological and environmental shortcomings of the pyrometallurgical scheme for extracting metals from waste and industrial products determine the need to find better ways for their complex processing.

In this aspect, the development of new technologies and the improvement of existing technologies to increase the end-to-end extraction of non-ferrous and precious metals are urgent tasks of science and practice in the production of non-ferrous metals. In order to expand this task, of particular importance is the development of a method for the complex extraction of non-ferrous metals using a complex of beneficiation and metallurgy processes.

RESEARCH RESULTS AND THEIR DISCUSSION

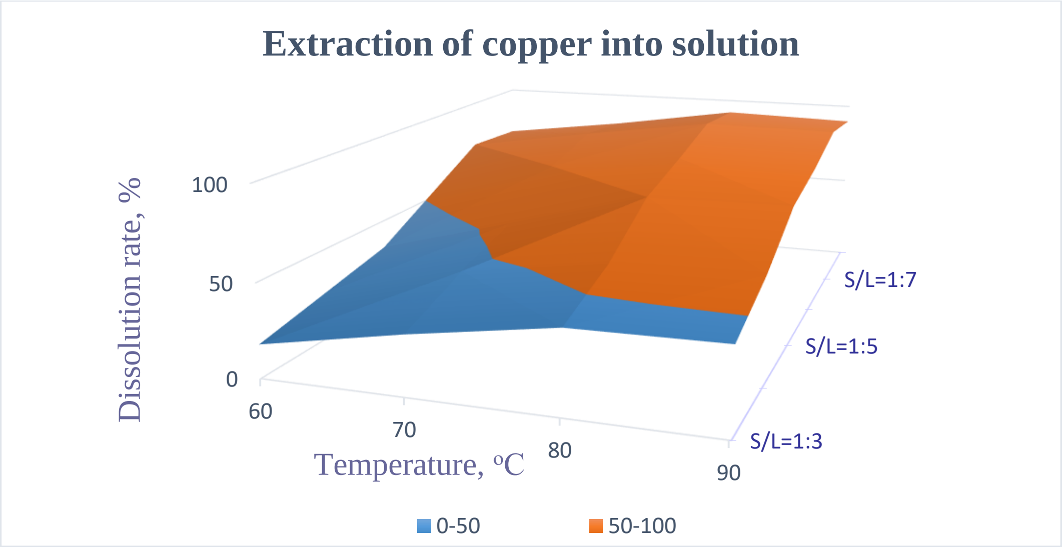
Fine converter dust of VF electrostatic precipitators and converters with an average content of chemical elements (%): Pb - 31.56%; Cu - 2.20%; Zn - 14.70%; Fe - 0.46%; Cd - 0.19% SiO2 - 0.65%; Stot - 13.15%; SSO4 - 8.56%; CaO - 0.91%; MgO - 0.06%; enters the acid leaching in the acidified washing acid of sulfuric acid plant of CMP. The concentration of sulfuric acid in the solution is 80-90 g / l, temperature is 80.0-85.0 °C, duration is 2.0 hours. Copper, zinc, iron, cadmium in the form of sulfates go into the solution. Lead and precious metals remain in the solid sediment (cake). The solid sediment is an additional sulfur for the extraction of lead and other non-ferrous and noble metals. After filtration, hydrolytic purification is carried out in the solution. The solid residue is sent to a two-stage leaching with sodium chloride. Leaching is carried out at the following operating parameter: the salt concentration of the first leaching stage is 250 g / l, at the second stage the leaching with sodium chloride is 150 g / l, the process temperature is 85-90 °C, the duration is 2.0 hours. After two-stage leaching with sodium chloride in solution, lead carbonization is carried out with the addition of sodium carbonate (Na2CO3) to pH = 8.5-9. The resulting cake PbCO3 is calcined at a temperature of 550-570 °C, the calcination duration is 1 hour. After calcining, we obtain litharge and carry out reduction smelting with the addition of carbon-containing material and flux. Graphite was used as the carbon-containing material.

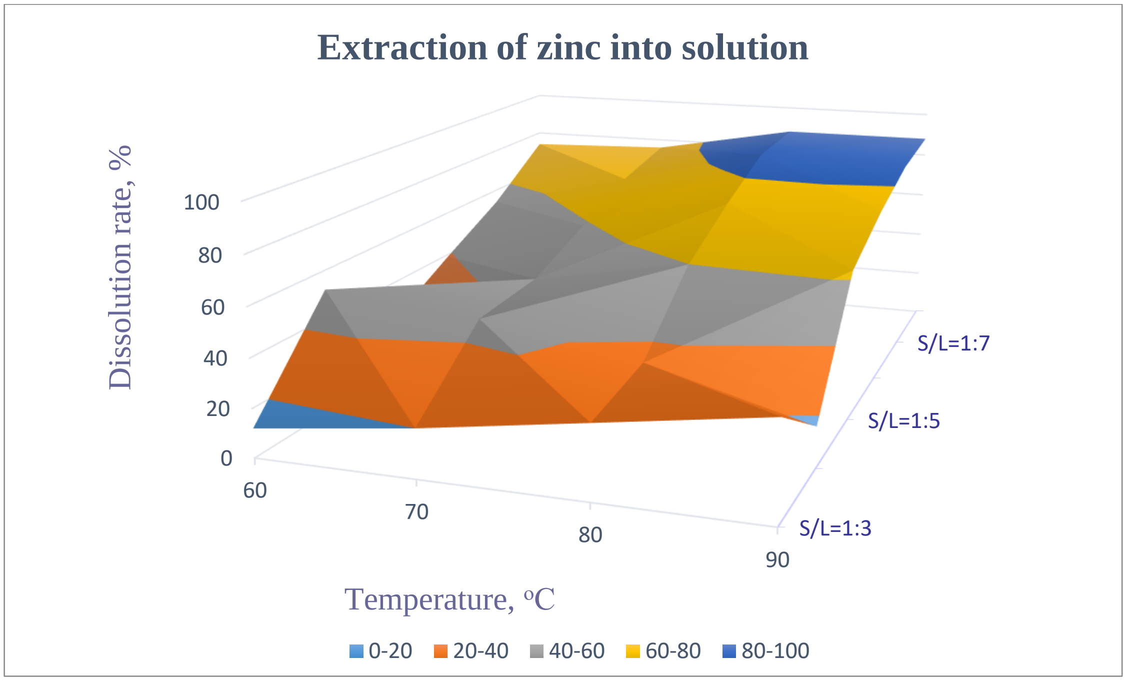
Acid leaching is carried out to leach valuable components from fine dusts of the electrostatic precipitator of converters; it is proposed to use at the first stage a representative washing acid from Sulfuric acid plant of CMP with a sulfuric acid content of more than 37.2 g / l, as well as containing copper, zinc, rhenium and other components. As a result of dust leaching at a given S: W = 1: 4 ratio, sulfuric acid is neutralized from an initial concentration of 80 g / l to a pH value of 0.8-1 (30-35 g / l). Leaching was carried out at a sulfuric acid concentration of 80, g / l. Leaching temperature 80 - 85°C. With sulfuric acid leaching with a higher content of S: L, an increase in temperature has a positive effect on the degree of transition of copper and zinc into solution. The influence of the concentration of sulfuric acid, temperature on the extraction of honey zinc, lead and bismuth into solution is shown in Table 2.

Table 2.

Sample No.	Mode parameters				Content in solution				Content in the cake, %			
	Temperature, °C	Leaching time, min.	Acidity, g/l	S/L	Pb, mg/l	Zn, g/l	Cu, g/l	Bi, g/l	Pb	Zn	Cu	Bi
1/1	20-25	120	37,2	1:8	8,0	13,0	5,9	48,0	55,15	0,71	0,51	0,42
1/2	20-25	120	37,2	1:8	8,0	14,0	6,1	51,0	51,11	0,64	0,54	0,41
1/3	20-25	120	37,2	1:8	7,0	14,0	6,3	54,0	46,20	0,72	0,57	0,40
2/1	20-25	120	70-80	1:8	5,0	14,0	6,0	79,0	44,09	0,68	0,55	0,37
2/2	20-25	120	70-80	1:8	5,0	14,0	6,1	70,0	52,70	0,71	0,58	0,37
2/3	20-25	120	70-80	1:8	5,0	14,0	6,0	62,0	55,12	0,78	0,64	0,38
3/1	25-30	120	120	1:8	4,0	14,0	6,4	78,0	50,59	1,03	0,60	0,35
3/2	25-30	120	120	1:8	7,0	14,0	6,0	77,0	47,78	0,66	0,55	0,36
3/3	25-30	120	120	1:8	7,0	14,0	6,3	72,0	52,17	0,81	0,55	0,35
4/1	60-70	120	30-40	1:8	17,0	19,0	8,2	46,0	57,26	0,62	0,33	0,43
4/2	60-70	120	30-40	1:8	6,0	18,0	7,9	64,0	60,78	0,46	0,24	0,41
4/3	60-70	120	30-40	1:8	6,0	20,0	9,8	64,0	61,57	0,62	0,40	0,42
5/1	60-70	120	80-90	1:8	6,0	17,0	8,2	119,0	37,41	0,71	0,45	0,35
5/2	60-70	120	80-90	1:8	6,0	21,0	9,7	143,0	42,51	0,72	0,48	0,35
5/3	60-70	120	80-90	1:8	5,0	16,0	7,3	112,0	59,37	0,71	0,43	0,38
6/1	50-60	120	100-120	1:8	2,0	20,1	7,8	136,0	61,13	0,68	0,29	0,35

6/2	50-60	120	100-120	1:8	2,0	19,2	8,7	154,0	52,87	0,70	0,68	0,36
6/3	50-60	120	100-120	1:8	2,0	17,5	8,6	116,0	60,42	0,56	0,39	0,39
7/1	80-90	120	30-40	1:8	3,0	17,3	8,3	30,0	51,11	0,86	0,41	0,44
7/2	80-90	120	30-40	1:8	2,0	18,8	8,9	42,0	62,01	0,55	0,41	0,46
7/3	80-90	120	30-40	1:8	3,0	15,3	6,4	8,6	56,91	0,45	0,26	0,37





As can be seen from table No. 2, at a sulfuric acid concentration of 90 g / l, at a temperature of 70 °C, and at a ratio of S:L = 1: 8, more positive results were obtained. After sulfuric acid leaching in order to remove undissolved residues from the solution, filtration is performed. The precipitate was washed with water to pH = 5.5 ÷ 6 at a water temperature of 80 °C. The filtrate obtained after filtration with a copper content of 9 g / l and zinc content of 21 g / l is a productive solution for the extraction of zinc and copper. As a result of sulfuric acid leaching, the resulting solution goes from light blue to dark blue.

The solubility of lead chloride in sodium chlorides depends on their concentration and the temperature of the solution. So, for example, an increase in the concentration of sodium chloride from 190 to 330 g / l, and the temperature of the solution from 25 °C to 80 °C increases the solubility of lead from 3.1-3.5 g / l to 76.6-81.1 g / l ...

The dissolution of lead sulfate proceeds with the following reaction.

$$PbSO_4 + 2\ NaCl = PbCl_2 + Na_2SO_4$$

To withdraw the reaction product - sodium sulfate - and shift the reaction to the right, a mixed solvent consisting of sodium chlorides is used. Lead goes into solution.

After acidic leaching of the cake, with a moisture content of 20% and a lead content of 64%, salt leaching was performed. The operating parameters affecting the extraction of lead chloride are given in Table 3.

Table 3.

Results of the study of salt leaching of lead cake obtained from sulfuric acid leaching of converter dust

No.	Mode			Content of elements in solution, mg/l				Content of elements in the cake, %			
	T, °C	Leaching time, min	Sodium chlorine concentration	Cu	Zn	Pb	Bi	Cu	Zn	Pb	Bi
1	2	3	4	5	6	7	8	9	10	11	12
I	70-80	90	100	30,6	59,0	62,80	103,0	0,26	0,17	14,16	0,43
			150	28,0	101,0	63,6	42,0	0,34	0,15	9,39	0,42
			200	28,5	147,0	63,07	353,0	0,35	0,14	4,78	0,30
			250	25,0	212,0	64,7	135,0	0,29	0,05	2,5	0,3
			300	20,0	230,0	64,2	626,0	0,34	0,04	0,92	0,1

1	2	3	4	5	6	7	8	9	10	11	12
II	90-95	90	100	40,5	212,0	62,8	247,0	0,29	0,16	13,30	0,5
			150	36,2	263,0	60,95	760,0	0,34	0,18	10,30	0,44
			200	29,4	302,0	63,86	508,0	0,26	0,04	5,90	0,42
			250	26,4	300,0	60,15	483,0	0,30	0,16	1,49	0,1
			300	23,1	275,0	51,94	495,0	0,33	0,04	0,86	0,07

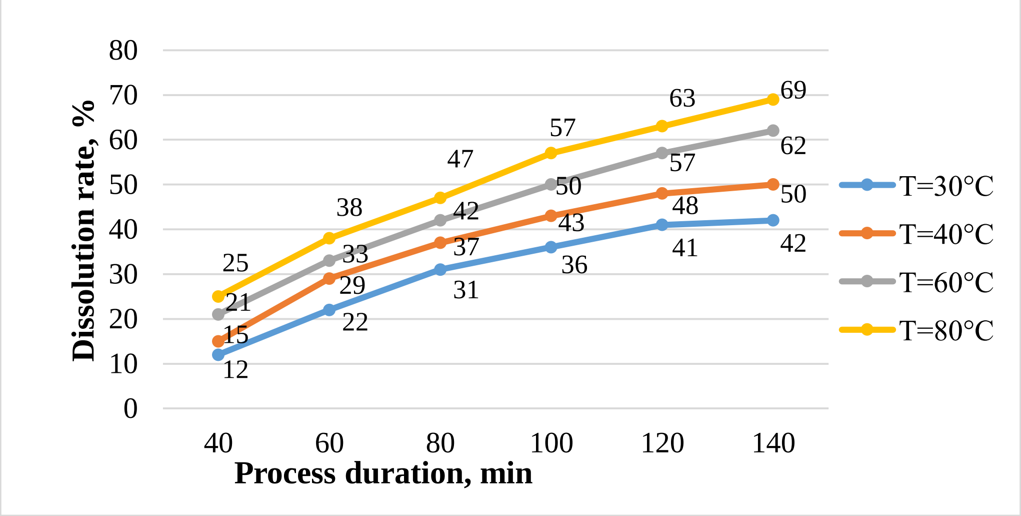


Fig. 1. Dependence of the degree of dissolution of lead after stage I of salt leaching into solution on the duration of the process and temperature

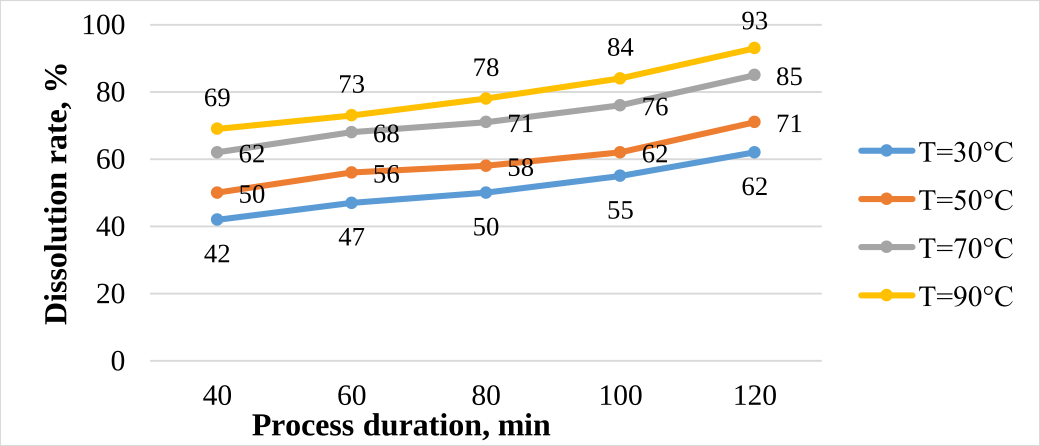


Fig. 2. Dependence of the degree of dissolution of lead after stage II of salt leaching into solution on the duration of the process and temperature

The precipitation of lead from chloride solutions can be carried out using the following methods:

- Crystallization of lead chloride followed by electrolysis of the melt containing lead and sodium chloride;
- Cementation of lead with iron scrap or spongy iron;
- Electrolysis of solutions with soluble (iron or cast iron) or insoluble (graphite) anodes;
- Precipitation of lead in the form of a hydrate by the action of lime on the solution, followed by melting of the hydrate on the metal in a shaft furnace;
- Carbonation with addition of soda ash and calcination.

The simplest in technological design, regulation of process parameters, the possibility of regeneration of salt solutions and subsequent processing of sludge, is the method of carbonization with the addition of soda ash followed by calcining the precipitate of lead carbonate. Proceeding from this, we have chosen this method to isolate lead from chloride solutions.

Lead carbonization was carried out by adding a solution of calcined technical grade to a solution of lead chloride, which leads to an increase in the pH of the medium to 8.5-9.0 and the following reaction [10].

$$PbCl_2 + Na_2CO_3 = 2PbCO_3\downarrow + 2NaCl$$

After carbonization, the pulp was filtered off, and the solutions were returned to circulation. The resulting cake contains more than 60% $PbCO3$.

Table 4 shows the results of laboratory studies to study the effect of the consumption of soda ash solution with a concentration of 170 g / l on the pH values of the solution, the sediment yield (the degree of precipitation of lead carbonate)

Carbonation Analysis Results

Table 4.

№	**Indication pH**	**Content in solution, mg/l**		**Content in the cake, %**	
		Pb	**Bi**	**Pb**	**Bi**
1	7	260,2	304	45	0,39
2	7,5	146,5	298	50,2	0,42
3	8	124,8	246	56,4	0,40
4	8,5	106,3	201,3	60,06	0,43
5	9	97	189,6	67,63	0,5

Calcining of lead carbonate sediments in laboratory conditions was carried out as follows. The wet cake of lead carbonate was dried in an oven at a temperature of 120 0 for 60 minutes. After the end of the specified time, a sample of lead carbonate was removed from the drying oven and cooled to room temperature. During the drying process, the cake was compacted in the form of a "cake" (Fig. 3)

Fig. 3. Dried sludge of lead ca

For the efficient course of the process of thermal decomposition (burning) of lead carbonate, a developed specific surface area of the material to be calcined is required. For this reason, the resulting "sintered" precipitate of lead carbonate was abraded by hand in a porcelain mortar.

After attrition, the lead carbonate precipitate was weighed on an electronic balance and loaded into a refractory crucible, which was placed in a preheated muffle furnace. After the completion of calcination, the crucible was removed from the furnace and cooled to room temperature. After cooling down, the calcined material was weighed.

To determine the optimal conditions for the process of calcining the sediments of lead carbonate, a series of laboratory tests were carried out at various durations and temperatures. Calcined lead carbonate precipitate of the following composition, in%: Pb - 67.63; Bi - 0.5;

According to the results of laboratory studies, the following optimal operating parameters of the process of calcining lead carbonate sediments were obtained:

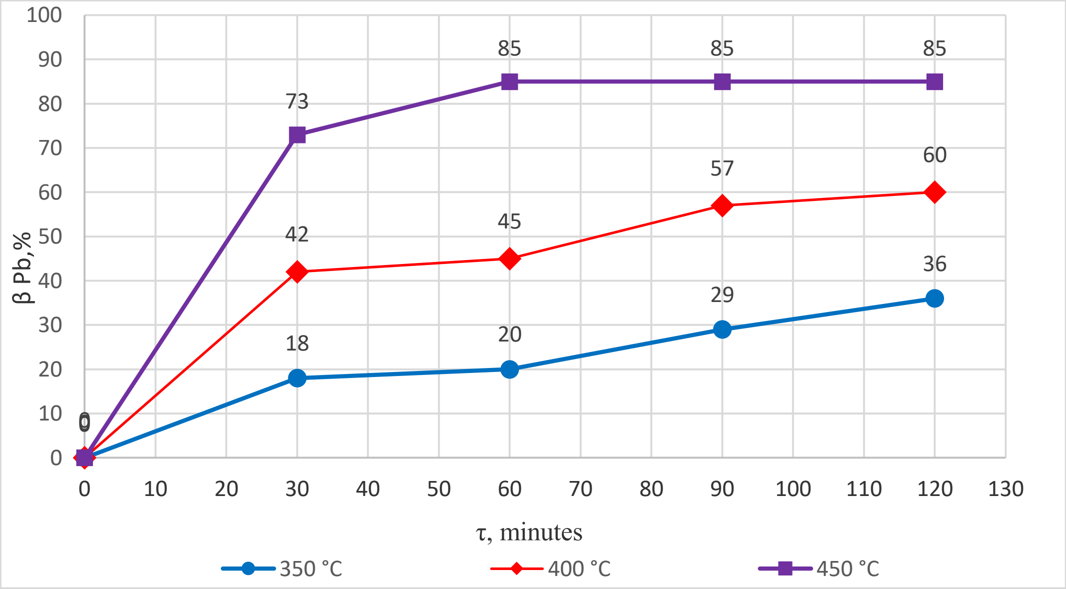
- The duration of the process of calcining the sediments of lead carbonate 60 min;
- Temperature of the process of calcination of lead carbonate sediments 450 °C.

The results of laboratory studies are shown in table. 5 and Figure 4.

Table 5

Study Results on burning Lead Carbonate Sediments

№	Material name	Temperature, °C	Time, min.	Pb content in the burned product, %
1	Lead carbonate precipitate	350	30	18
			60	20
			90	29
			120	36
2	Lead carbonate precipitate	400	30	42
			60	45
			90	57
			120	60
3	Lead carbonate precipitate	450	30	73
			60	85
			90	85
			120	85



1 – 350 ˚C; 2 – 400 ˚C; 3 – 450 ˚C

Fig. 4 Dependence of the lead content in the calcined product on time and on the burning temperature

After calcining the precipitates of lead carbonate, lead oxide PbO was obtained - lithot with a lead content of 85%. The appearance of the lattice after calcination is shown in Fig. 5.

Laboratory studies on the carbon-thermal reduction of lead oxide to the metallic state were carried out in a laboratory muffle furnace of the SNOL type (Fig. 6) The maximum heating temperature of the SNOL muffle furnace is 11000C, the heating rate to the maximum, 40 min.

Carbon-thermal reduction of lead oxide to the metallic state in laboratory conditions was carried out as follows. Lead litter after calcination of lead carbonate precipitates having the following chemical composition in terms of the recoverable components, in Pb - 89.13; Bi - 0.36. The bulk density of the obtained lead slurry is 1.7-2.2 g / cm3, weighed on an electronic balance.

The charge for the carbon-thermal reduction consisted of 51 g of slurry and 3 g of graphite, the ratio of slurry to graphite is 17: 1. The components of the charge were thoroughly mixed, then the charge was poured into the crucible. The crucible with the charge was poured into a preheated muffle furnace.

The effect of temperature and duration of carbon-thermal reduction on the lead content in the finished product was investigated. The research results are shown in Table 4 and Fig. 7.

Table 6.

Results of the study of carbon-thermal reduction of lead from slurry

№	Material name	Melting temperature, °C	Melting time, min.	Content of Pb, %
1	Slurry	700	30	44,7
			60	51,02
			90	48,9
			120	47,3
2	Slurry	800	30	77,6
			60	88,23
			90	85,6
			120	85,1
3	Slurry	900	30	93,5
			60	99,9
			90	98,93
			120	95,92

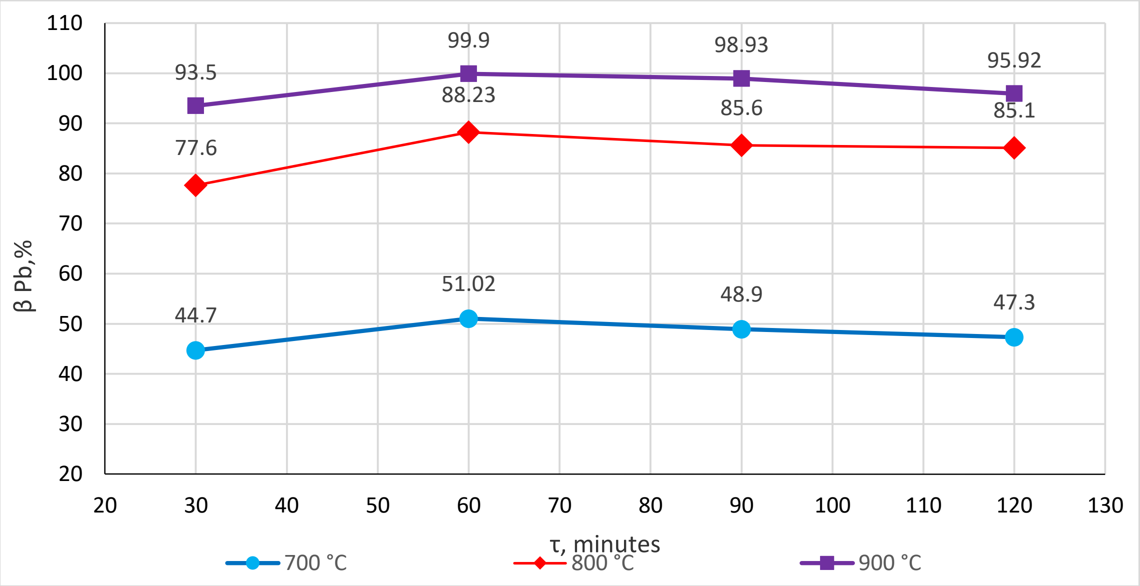


Fig. 5. Dependence of the degree of purity of lead in metal products on temperature

CONCLUSIONS

According to the results of the experimental studies, the optimal operating parameters of the process of coal-thermal recovery of carbon dioxide with a lead content of 85 -89% were selected:

- The duration of the process of coal-thermal recovery of gluten is 60 minutes;
- Temperature of the process of coal-thermal reduction of gluten 900 °C.

Metallic lead with a purity of 99.9% was obtained after carbon-thermal reduction of the slurry. The appearance of metallic lead with a purity of 99.9%, obtained as a result of laboratory tests, is shown in Figure 8.

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