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PROJECTING OF THE AUTOCLAVE-DISTILLATE UNIT AND ITS TESTING ON EXTRACTION OF OSMIUM OUT OF INDUSTRIAL INTERMEDIATE PRODUCTS OF COPPER PRODUCTION WITH USAGE OF HYDROGEN PEROXIDE

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ABSTRACT

Researches on elaboration of the technology of osmium extraction from lead cake, a pressure leaching product of lead dusts of copper production, allowed to define technological conditions of the dissolution processes in sulfuric acid cake with oxidants-hydrogen peroxide (H_2O_2) and distillation out of osmium from the resulting solution. Pressure leaching processes of lead cake and distillation out of osmium from solution were carried out in hermetic autoclave-distillation unit, which prevented the loss of osmium and harmful toxicity effect of osmium tetroxide on the environment. In this case, the condensation of osmium was implemented beyond that unit in an alkaline solution.

Key words: Osmium, Lead cake, Pressure leaching, Osmium tetroxide, Alkaline distillate.

INTRODUCTION

During the processing of copper production's lead dusts with autoclave-distillation method the majority of rhenium (96.8%), osmium (64.5%), copper (75.3%), cadmium (78.9%), zinc (81.4%) pass into the solution of oxidative pressure leaching. From the solution, directly in the autoclave, osmium tetroxide distils and condenses into alkaline solution in the form of sodium osmate. The bulk of lead (96%) and 30-35% of osmium of the initial content of osmium in lead dusts concentrates in no decomposed cake. From clarified solution (filtrate) rhenium is extracted, copper, cadmium, zinc, iron and other components are concentrated in reextract.

Subsequent recovery of valuable components (Os, Pb, Cu, Cd, Zn) of industrial

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products from processing of copper production's lead dusts of Zhezkazgan copper smelting plant (hereinafter-ZHP), i.e. recycling industrial products (cakes, solutions) provides an opportunity for obtaining more valuable products, increasing the economic profitability of the primary production of copper and rhenium, contributes to the social development of the area, where the technology will be used. However, utilization of the above-mentioned industrial products allow also solve environmental issues in the area of their storage. Therefore, the elaboration of technologies for processing copper manufactures industrial products is an important task.

Analysis of existing technologies on the subject showed –

- Most of the technologies are characterized by high losses of osmium tetroxide to the gas phase, due to its physical properties - a low boiling point (131.2^{o}C)[1-7].
- Dissolution of osmium containing industrial products and distilling of osmium into solution is carried out in different units and accompanied by losses of it into the atmosphere due to lack of tightness of unit[8-13].

This work is aimed to –

- The obtaining additional production (sodium osmate and lead cake purified of the osmium).
- Reduce of environmental pollution, which has been achieved by creating hermetic autoclave-distillation unit. This installation has a simplified construction and system to servicing, and provides safe and favorable transporting conditions of the gas phase on the further its distillation and condensation.

EXPERIMENTAL

After pressure leaching of poor by osmium (6.8 g/t) of lead dusts of the copper production of ZHP was obtained lead cake, the content of basic components of which are given in Table 1.

Table 1: The content of the lead cake main components

Elements	Os (g/t)	Re (g/t)	Pb (%)	Zn (%)	Cu (%)	Cd (%)
Content	2.26 (0.000226 %)	6.3 (0.00063 %)	50.12	0.91	3.52	0.39

Sufficiently high content of osmium (2.26 g/t) in the lead cake is the basis for his subsequent recovery to the desired product. To obtain the desired osmium product from the lead cake of pressure leaching was processed by flowsheet shown in Fig. 1.

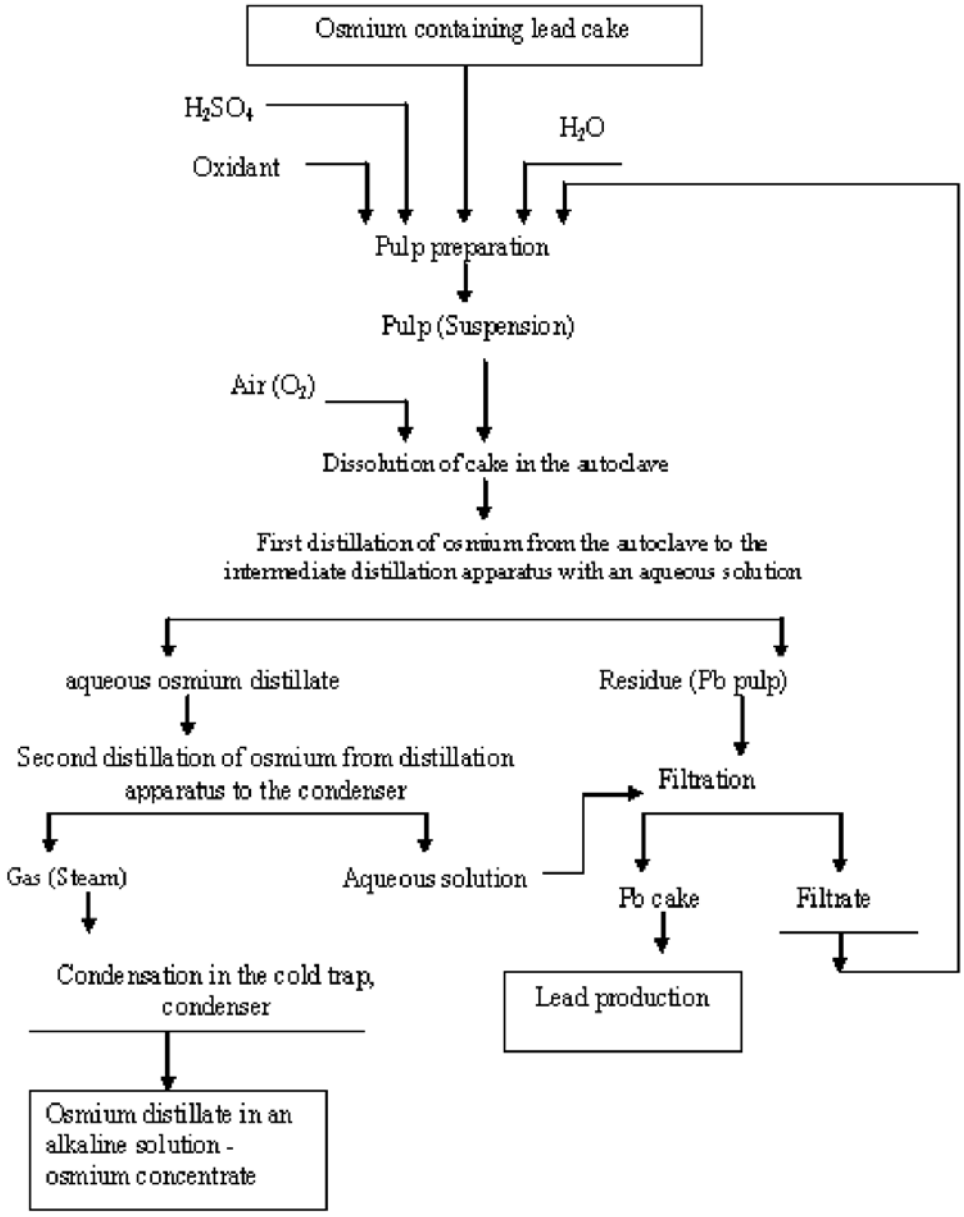

Fig. 1: Flowsheet of obtaining osmium concentrate from the osmium containing cake by pressure leaching of lead dusts of copper production

Autoclave-distillation unit (Fig. 2), designed to extract osmium consist of agitator reactor making pulp (position 1); hermetic autoclave (pos. 5) equipped with a heating system and technological pipes for input and output of liquid and solid components and gas, mechanical mixer (pos. 10) mounted within the housing attached to the lid and connected to the actuator; sealed distillation apparatus (pos. 12), equipped with a heating system and a partition separating the cavity of the body into two vertical cavities, each of them communicates with the system inlet and outlet vapor (gas), a vacuum pump (pos. 14); cooled condenser (pos. 15), equipped with a steam supply system (gas) and the withdrawal of liquid and solid products, vacuum filter (pos. 17) to separate suspension. In addition, the unit is provided with control systems and regulation of temperature, pressure on all devices of autoclave-distillation unit.

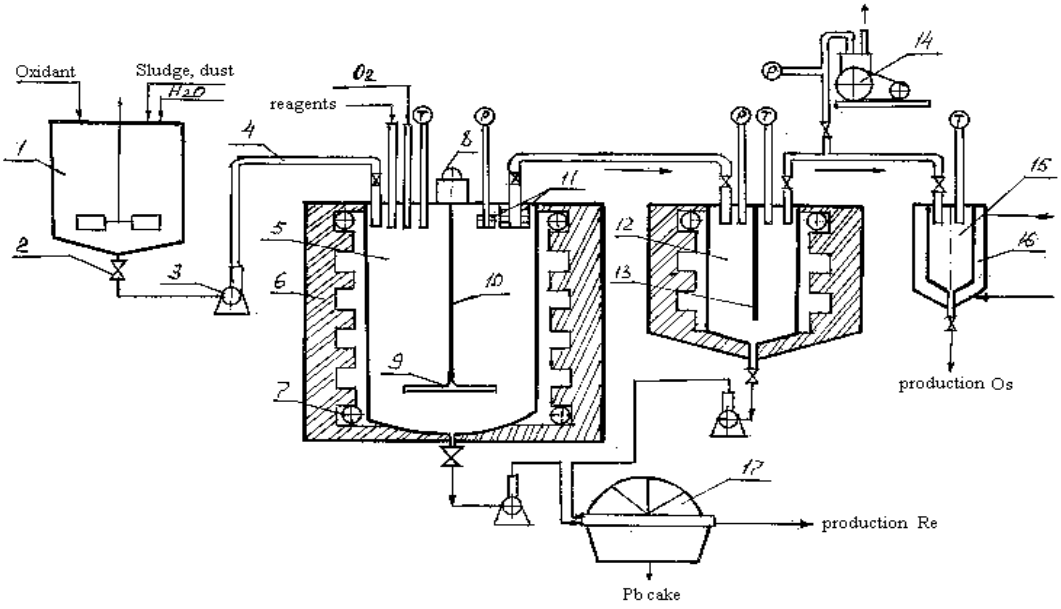

Fig. 2: Autoclave-distillation unit

(1) Agitator reactor for pulp preparation, (2) Shut-off valve, (3) Pulp pump, (4) Pipeline, (5) Autoclave, (6) Lining, (7) Heater, (8) The drive, (9) Mechanical mixer, (10) Shaft, (11) Reflectors, (12) Distillation apparatus, (13) Partition, (14) Vacuum pump, (15) Condenser, (16) Cooling jacket, (17) Drum vacuum filter, (P) Pressure control, (T) Temperature control

To prevent corrosion, while working in different environments internal surfaces of the autoclave, a mechanical mixer, flange seals, reagents supply pipes and drain suspensions lined by lead sheet or fluoroplastic-4.

Unit operates as follows: lead cake loaded into the reactor agitator making pulp (pos. 1) to the same feed solution H_2SO_4. Heated to 70-80^oC suspension was loaded into the autoclave, which comprises heating the autoclave and mechanical mixing. The autoclave (pos. 5) was added oxidant, in necessity in addition further fed pure O_2 or air oxygen. Then began the process of distillation of osmium tetroxide in the distillation apparatus with water (pos. 12) and caught an osmium tetroxide in alkaline solution in a cooled condenser (pos. 15).

As refrigerants were used: water, ice, ice + water, NaCl, $NaNO_3$, KNO_3, NH_4Cl, $(NH_4)_2SO_4$ and etc. Upon completion of the process the suspension in the autoclave was cooled and was sent to the vacuum filter with solution of distillation apparatus (pos. 17). Lead cake cleared of osmium sent to lead production for obtaining marketable lead, and the clarified solution (filtrate) to prepare a suspension in reactor agitators (pos. 1).

RESULTS AND DISCUSSION

To elaborate the technology of lead dissolution cake was carried out in an autoclave by sulfuric acid without using an oxidant and with oxidant-hydrogen peroxide (H_2O_2). In experiments of dissolution autoclave cake by sulfuric acid without oxidant formation of osmium tetroxide is not happening, and in experiments with hydrogen peroxide, we observed the formation of osmium tetroxide.

Technological conditions and the results of experiments without oxidant are shown in Table 2, with the oxidant H_2O_2 in Table 3 and Fig. 3.

Table 2: Technological conditions and the results of experiments on the sulfuric acid dissolution of lead cake without oxidant (weight 100 g (Os -0.226 mg); H_2SO_4 -50%; L/S of 20:1; 45 % NaOH solution -0.1 dm^3)

Temp. (^oC)	Duration (min)	Content of osmium in the distillate of alkaline solution (g/t) %
120	30	not found
130	30	not found
140	30	not found
150	30	not found
120	45	not found

Cont…

Temp. (^oC)	Duration (min)	Content of osmium in the distillate of alkaline solution (g/t) %
130	45	not found
140	45	not found
150	45	not found
120	60	not found
130	60	not found
140	60	not found
150	60	not found
120	75	not found
130	75	not found
140	75	not found
150	75	not found

Experiments have shown (Table 3, Fig. 3) that the extraction of osmium to alkaline distillate, increases at rising temperature and duration of distillation, extraction reaches 98.67% at 140^{o}C in 20 minutes distillation, at a consumption of H_2O_2 equal to 50% by weight of lead cake.

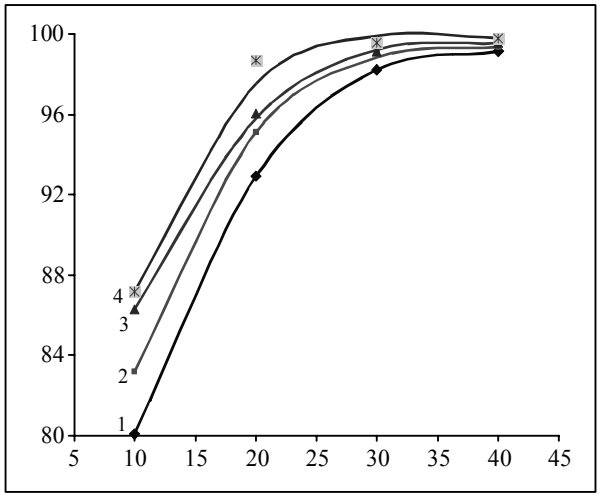

Fig. 3: Extraction of osmium to distillate depending on the duration and process temp. (const: 100 g weight; H_2SO_4 -50%, 50 g of 100% H_2O_2 content, L/S of 20:1; 45% NaOH -0.1 dm^3). Temperature by the process: 1-110^{o}C; 2-120^{o}C; 3-130^{o}C; 4-140^{o}C

Table 3: Technological conditions and the results of experiments on the sulfuric acid dissolution of lead cake with an oxidant-hydrogen peroxide (100% H_2O_2 content, weight 100 g (0.226 mg Os); H_2SO_4 -50%, L/S of 20:1; 45 % NaOH solution -0.1 dm^3)

Temp. °C	Duration, min	The amount of H_2O_2 dependent by weight of lead cake (%)	Volume of alkaline osmium distillate (dm^3)	Content of Os			Os extraction	
				In a cake (mg)	In alkaline distillate		To alkaline distillate (%)	To cake (%)
					(mg)	mg/dm^3		
1	2	3	4	5	6	7	8	9
110	10	50	0.108	0.0436	0.181	1.676	80.09	19.19
120	10	50	0.111	0.0366	0.188	1.696	83.19	16.15
130	10	50	0.115	0.0298	0.195	1.698	86.28	13.13
140	10	50	0.118	0.0282	0.197	1.670	87.17	12.21
110	20	50	0.113	0.0147	0.211	1.864	92.92	6.37
120	20	50	0.116	0.01	0.215	1.857	95.13	4.33
130	20	50	0.121	0.00825	0.217	1.79	96.02	3.47
140	20	50	0.126	0.0025	0.223	1.772	98.67	1.01
110	30	50	0.121	0.0034	0.222	1.835	98.23	1.44
120	30	50	0.128	0,0018	0.224	1.750	99.12	0.67
130	30	50	0.137	0.00147	0.224	1.638	99.12	0.51
140	30	50	0.145	0.00056	0.225	1.554	99.56	0.20
110	40	50	0.135	0.0016	0.224	1.660	99.12	0.64
120	40	50	0.148	0.001	0.225	1.519	99.34	0.32
130	40	50	0.156	0.0008	0.225	1.443	99.56	0.29
140	40	50	0.162	0.00034	0.226	1.392	99.78	0.09

Note: discrepancy: sensitivity of analysis, the loss in a distillation apparatus, mechanical losses

Then extracting osmium to alkaline distillate decelerates to 99.78% in the same conditions and the duration of 40 minutes.

Dissolution reaction cake in sulfuric acid with an oxidant H_2O_2 proceeds rapidly with foaming and extraction of osmium to alkaline distillate at 110°C, duration 10 minutes,

reaches 80.09%, at 120^{o}C extraction of osmium - 83.19%, at 130^{o}C – 86.28%, at 140^{o}C - 87.17% at the same duration.

Maximum extraction of osmium to alkaline distillate: 99.12% (110^{o}C), 99.34% (120^{o}C), 99.56% (130^{o}C), 99.78% (140^{o}C) was achieved by process duration of 40 minutes the amount of addition of hydrogen peroxide 50% by weight of lead cake. A further increase oxidant additives-hydrogen peroxide is ineffective because, took place excessive rough foaming of solution, increased the pressure in the unit, there was a probability of emissions of the solution and in that case it may occur to the loss of control of the process the installation of leakage.

CONCLUSION

Installed autoclave-distillation unit provides two different processes in one unit: pressure leaching cakes by sulfuric acid with usage of oxidant-hydrogen peroxide with result-obtaining enriched osmium solution; distillation of tetroxide osmium from enriched osmium solution to alkaline distillate that is second aimed product. Comparative researches of pressure leaching lead cakes in sulfuric acid without oxidant and with the presence of H_2O_2 showed the effectiveness of the oxidant, providing a high degree of extraction of osmium.

In this case, the elaborated technology of extraction of osmium oxidant H_2O_2 provides the following benefits:

(i) Processes of extraction osmium out of osmium solutions to alkaline distillate flow at relatively low temperatures and short duration;

(ii) Undecomposed lead cake and the filtrate were not contaminated by the oxidant (H_2O_2).

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