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Satbayev University

ХАБАРЛАРЫ

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
Satbayev University

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PROCESSING OF INDUSTRIAL PRODUCTS WHEN DISPOSING OF COPPER ELECTRO-REFINING SOLUTIONS

Abstract: at enterprises that process secondary copper raw materials, it is customary to extract copper from the spent electrolyte by electroextraction. After electroextraction, the solution is neutralized with lime milk to obtain a gypsum nickel-containing cake or evaporated with the release of a cake containing metal sulfates (Cu, Ni, Zn, Fe), which is sold as a commercial product. The search for effective technological solutions for the selective isolation of secondary non-ferrous metals during the regeneration and complete utilization of copper electro fining solutions is relevant. The paper presents studies of the processing of spent copper electrolyte from the processing of non-ferrous metal scrap at a copper smelter in Kazakhstan. For the processing of the spent electrolyte, a stage-by-stage neutralization was performed using zinc sublimates and potash. As a result of the first stage of neutralization with zinc sublimations to pH 4.7, a precipitate with a content of PbO 44.69 %; SnO_2 16,36 %.was obtained. After processing the sediment with an alkaline solution, carbonization and melting at a temperature of 900ºC, metallic lead and tin-containing slag with a content of SnO_2 of 16.36 % were obtained. As a result of the second stage of neutralization with potash to pH 7.1, a precipitate was obtained-with a CuO content of 76.45 %.After the third stage of neutralization with potash to pH 9.5, a precipitate with a content of NiO 27.63 % and ZnO 55.75 % was obtained. After treatment of the precipitate with a solution containing 100 g / dm^3 KOH, a zinc-containing solution with a ZnO content of 225.0 g/dm^3 and a precipitate were obtained, after calcination of which nickel oxide with a NiO content of 89.14 % was obtained.

Key words: copper electrolyte, zinc-containing product, potash, copper-containing precipitate, metallic lead, slag.

Introduction. During the processing of non-ferrous scrap metals at the electro fining copper recovery stage unwanted impurities such as nickel, zinc and other impurities accumulate in the spent electrolyte and affect the quality of the copper cathode. Part of the electrolyte from the saleable baths is periodically removed from the electrolysis cycle and reprocessed. The productivity of the plant depends on the processing of the solutions; therefore, the development of an efficient technology is relevant.

When refined copper is produced from mineral raw materials the spent electrolyte is treated by regeneration. For purification of the electrolyte from impurities extraction, sorption, membrane and combined sorption and electrochemical methods have been proposed [1-5].

At the majority of the enterprises processing scrap of non-ferrous metals the two-stage scheme of processing of spent electrolyte is applied, including evaporation and crystallization with obtaining copper sulphate and extraction of residual copper from the evaporated solution by electroextraction [2,6]. After extraction of copper from solution, nickel is extracted in the form of sulphuric acid salt by evaporation, crystallization and subsequent refining. The method does not allow to separate non-ferrous metals present in the solution selectively enough.

The method of processing of spent electrolyte is also used according to which copper is first extracted by electro extraction. After electroextraction solution is neutralized with lime milk to obtain gypsum nickel-containing cake or evaporated to obtain cake containing sulphate metals (Cu, Ni, Zn, Fe), which is sold as a commercial product [2,6,7,8]. The method also does not allow to obtain a selective extraction of non-ferrous metals.

In order to develop an efficient technology for processing spent copper electrolyte, research was performed on solution utilization and selective

extraction of non-ferrous metal concentrates by a stage-by-stage neutralization method using a zinc-containing intermediate product.

Zinc-containing intermediate product - zinc sublimations formed during processing of nonferrous metal scrap in the process of copper refining and captured during waste gases purification in bag filters. These distillates cannot be used to produce zinc white metal due to the presence of lead and tin and are normally mixed with sulphur-containing flux, granulated and subjected to distillation of lead and tin in a rotary kiln [9,10,11].

Research objective. Development of technology for the disposal of spent copper electrolyte from non-ferrous scrap processing by means of stage-by-stage neutralization using zinc sublimations and producing selective concentrates of non-ferrous metals.

Research methods and techniques. X-ray fluorescence, chemical and X-ray phase analyses were used in the research. X-ray fluorescence analysis was performed on a Venus 200 spectrometer with wave dispersion (PAN alyical B.V., Holland).

Chemical analysis of samples was performed on optical emission spectrometer with inductively coupled plasma Optima 2000 DV (USA, Perkin Elmer).

Semi-quantitative X-ray phase analysis was performed on diffractometer D8 Advance (BRUKER) using Cu-Kα radiation at accelerating voltage 36 kV, current 25 mA.

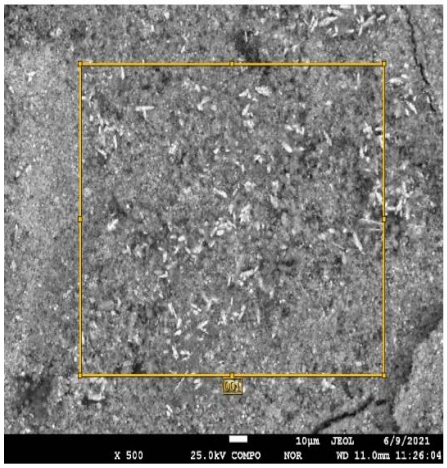
Microphotographs were made on a low-vacuum scanning electron microscope with thermal emission cathode (LaB6) JSM-6610LV of "JEOL" company.

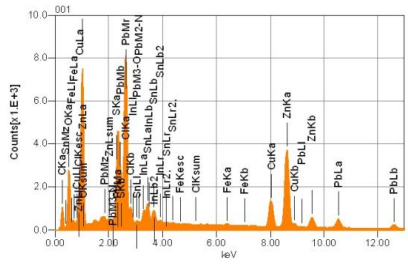
Research results. Spent copper electrolyte and zinc sublimations from the copper smelting plant "Casting" PLC in Kazakhstan were used for the research.

Chemical composition of spent copper electrolyte, wt. g/dm^3: Cu 67.14; Ni 36.41; Fe 11.43; Zn 10.96; SO_4 125.9; N 4.1; As; 0.03; Bi 0.002; Na 2.6; Pb 0.014; Sb 0.05; Si 0.047; Sn 0.0.

Chemical composition of zinc sublimations wt.%: F 0.97, Al_2O_3 0.15, P_2O_5 0.82, SO_3 4.0, Cl 11.64, K_2O 0.93, CaO 0.36, $Fe_2O_3$0.29, NiO 0.05, CuO 7.86, ZnO 39.46, Br0.19, MoO_3 0.1, CdO 0.23, SnO_2 7.16, WO_3 0.36, PbO 19.4, Bi_2O_3 0.04, op 0.04.

The electron microscopic analysis of the zinc substrates is shown in Figure 1.





Elements	ms%	mol%	Sigma	Net	K ratio	Line
C	16.55	39.81	0.30	138222	0.0035840	K
O	18.54	33.49	0.30	550982	0.0652360	K
S	0.49	0.44	0.07	146317	0.0043180	K
Cl	10.32	8.41	0.09	2869289	0.0827736	K
Fe*	0.22	0.12	0.18	39006	0.0023840	K
Cu	7.03	3.20	0.34	794308	0.0723914	K
Zn	24.81	10.97	0.42	2412878	0.2572257	K
In	1.50	0.38	0.26	243198	0.0111408	L
Sn	3.21	0.78	0.27	525069	0.0245118	L
Pb	17.32	2.42	0.34	2567008	0.1247003	M
Total	100.00	100.00				

Figure 1 - Microphotograph of zinc sublimations x 500

According to the X-ray phase analysis shown in Figure 2, the composition of the zinc sublimations is as follows, wt.%: matlockite (PbClF) - 43.1; copper sulphate ($Cu_5(SO_4)_2(OH)_6 \cdot 5H_2O$) - 20.0; copper chlorate ($Cu(ClO_4)_2$ - 10.3; zinc stonate ($Zn_2(SnO_4)$) - 9.8; moolooite ($C_2CuO_4 \cdot xH_2O$) - 4.4; lead hydrate acetate ($C_4H_8Pb_2O_6 \cdot H_2O$) - 3.8; zinc oxalate (C_2O_4Zn) - 3.2; phedotovite (K_2Cu_3+2O$(SO_4)_3$ - 2.8 and zinc chloride ($ZnCl_2$) - 2.6.

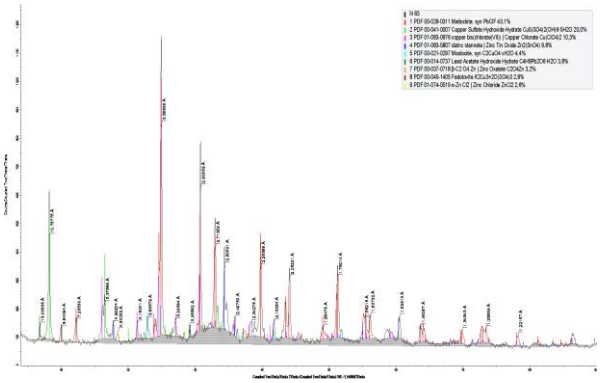


Figure 2 - X-ray appearance of zinc sublimations

A stage neutralization of spent copper electrolyte using zinc sublimations was performed to utilize industrial products of non-ferrous scrap processing and selective extraction of non-ferrous metals.

The first stage of neutralization of electrolyte was performed by zinc sublimations to pH 4.7 at L:S=5:1, temperature 20°C, stirring time 3 hours. As a result of neutralization we obtained black leadtin precipitate the composition of which is given in table 1.

The second and third stages of neutralization were performed with potash. The choice of potash instead of cheaper reagent Na_2CO_3 was determined by the possibility of obtaining after solution purification and evaporation a marketable, highly-liquid product - potassium sulphate K_2SO_4, instead of Na_2SO_4.

After the second stage of electrolyte neutralization to pH 7.1 a copper-containing precipitate with low impurity content was obtained which can be returned to the copper electrofining solution.

Table 1 - Chemical composition of neutralization precipitation

Name	Contents, %		
	pH		
	4.7	7.1	9.5
F	0.44	0.26	0.3
Na_2O	0.71	-	-
MgO	-	-	0.11
Al_2O_3	1.56	0.34	1.3
SiO_2	4.08	0.24	0.54
P_2O_5	0.41	0.01	0.009
SO_3	11.12	5.56	3.42
Cl	0.72	4.7	0.19
K_2O	0.19	-	0.05
CaO	0.85	0.03	0.38
Fe_2O_3	5.48	0.31	0.14
NiO	0.15	1.1	27.63
CuO	2.97	76.45	0.68
ZnO	4.79	2.8	55.75
Br	0.09	0.01	-
MoO_3	0.23	-	-
CdO	0.08	0.01	-
SnO_2	16.36	-	-
WO_3	0.29	-	-
PbO	44.69	0.06	-
Bi_2O_3	0.09	-	-
п.п	4.7	5.12	8.131
Total	100	100	100

The precipitate of the first neutralization stage was processed with spent electrolyte solution at a ratio L:S=10:1. As a result, a filtrate with pH 2.2 and a lead-tin-containing product were obtained, wt. %: Na_2O 0.16; Al_2O_3 0.34; SiO_2 1.83; P_2O_5 0.17; SO_3 20.53; Cl 0.51; K_2O 0.29; CaO 0.23; Fe_2O_3 0.95; NiO 0.05; CuO 1.33; ZnO 0.57; As_2O_3 0.07; Br 0.07; MoO_3 0.1; CdO 0.1; SnO_2 9.5; WO_3 0.18; PbO 62.24; BiO_3 0.11; pp. 0.67.

For processing of lead-tin-containing product the method of processing of production wastes was used [12]. The method is suitable for processing of production wastes containing compounds of lead, tin, antimony, copper, iron, zinc, bismuth, arsenic, silver, calcium, sodium, potassium.

In accordance with [12] the carbonization of obtained lead-tin product was performed in a solution containing 135 g/dm^3 K_2CO_3, at L:S=4:1 and temperature 20°C. After filtration a composition precipitate, wt. %: Al_2O_3 0.42; SiO_2 2.29; P_2O_5 0.21; SO_3 2.9; Cl 0.1; K_2O 0.29; CaO 0.23; Fe_2O_3 0.95; NiO 0.06; CuO 1.66; ZnO 0.71; As_2O_3 0.1; Br 0.01; MoO_3 0.12; SnO_2 11.8; WO_3 0.22; PbO 77.8; BiO_3 0.13.

The phase composition of the carbonation precipitate is represented, wt.%: cerussite ($PbCO_3$) 91.4; ferrous tin oxide ($(Sn_{0.9}Fe_{0.1})O_2$) – 3.0; cassiterite (SnO_2)–2.8; ferrosilite($Fe_3Si_{0.93}$) –1.5 and zinc oxide (ZnO) –1.3 (Figure 3).

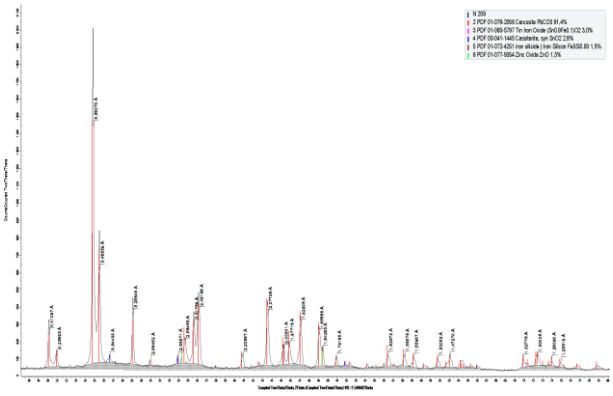


Figure 3 - X-ray appearance of the precipitate after carbonation

Carbonation precipitate in a mixture of 10 % charcoal and 5 % K_2CO_3 was smelted at 900°C for 30 minutes. Metallic lead and tin-bearing slag were obtained with composition, wt. %: PbO 3.36; SnO_2 47.84; Al_2O_3 1.08; $SiO_2$9,2; P_2O_5 1.96; SO_3 0.54; K_2O 15.1; CaO2,7; $Fe_2O_3$4,8; NiO 0.64; CuO 5.5; ZnO 0.3; As_2O_3 0.6; $WO_3$3.0; o.p. 3.38.

The third stage of the neutralization of the spent electrolyte to pH 9.5 resulted in a nickel-zinc-containing precipitate (Table 1). The precipitate was processed with a solution containing 100 g/dm^3 KOH at temperature 90 °C, time 30 minutes and L:S= 3:1. The alkaline zinc-containing solution with ZnO content of 225,0 g/dm^3 and nickel-containing

precipitate with composition, wt. %: NiO 76.2; MgO 3.2; Al_2O_3 0.32; SiO_2 0.35; SO_3 0.31; CaO 1.4; Fe_2O_3 0.11; CuO 0.2; ZnO 0.5; o.p. 17,41.

After calcination of the precipitate at 350°C for 30 minutes the precipitate of nickel oxide was obtained, wt. %: MgO 5.44; Al_2O_3 0.54; SiO_2 0.59; SO_3 0.53; CaO 2.38; Fe_2O_3 0.19; NiO 89.14; CuO 0.34; ZnO 0.85.

As a result of the conducted research the technological scheme of processing of the spent electrolyte of copper with use for neutralization of zinc sublimations was developed (figure 4).

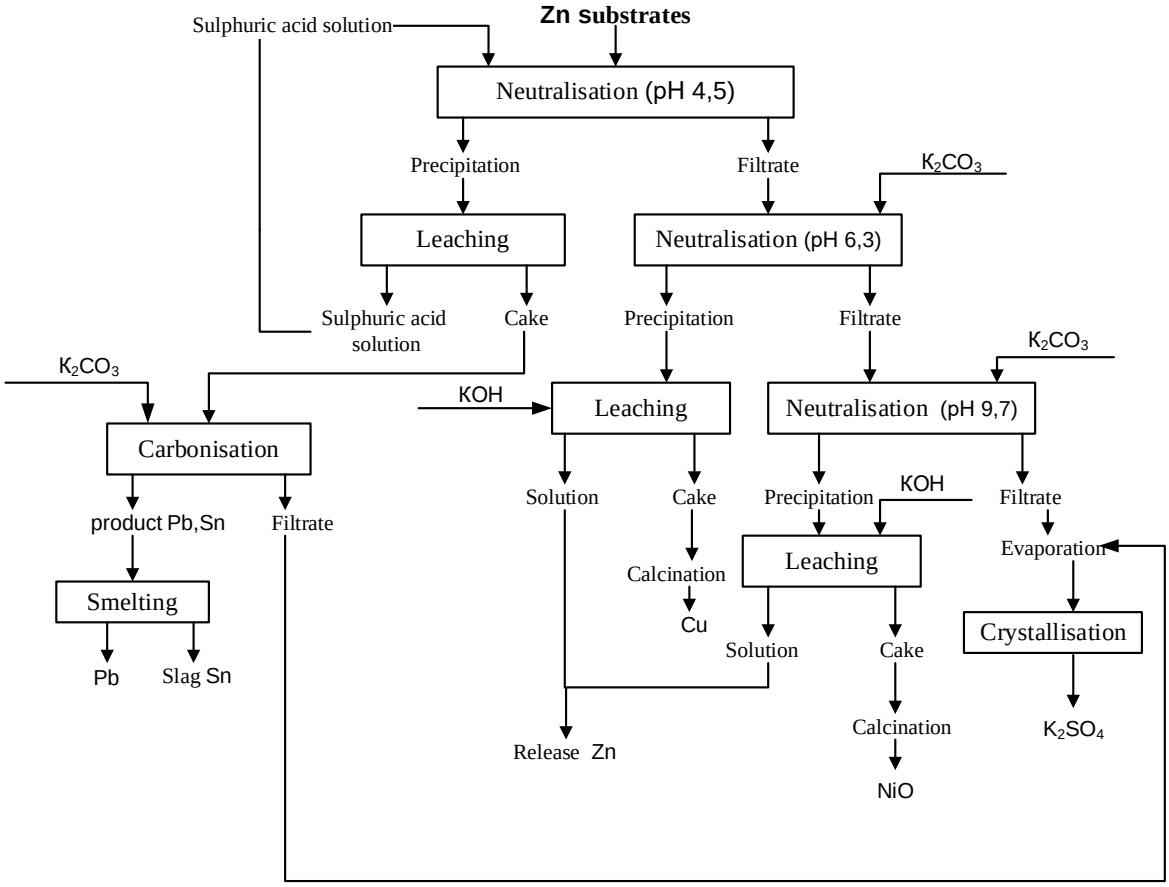


Figure 4 - Process flow diagram for processing spent copper electrolyte from non-ferrous metal scrap production

Conclusions. The physical and chemical research of content of the spent copper electrolyte and zinc-containing industrial product of the nonferrous metals scrap processing at the copper smelting plant "Casting" PLC in Kazakhstan was conducted.

As a result of the stage-by-stage neutralization of the used copper electrolyte with zinc sublimations and potash the following was obtained: copper-containing precipitate that can be returned into copper electro fining solution, nickel oxide, alkaline zinc-containing solution, metallic lead and tin-containing slag.

The technological scheme of utilization of industrial products of non-ferrous scrap processing - spent electrolyte and zinc slag - was developed.

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ЕРІТІНДІЛЕРДІ КӘДЕГЕ ЖАРАТУ КЕЗІНДЕ ӨНЕРКӘСІП ӨНІМДЕРІН ҚАЙТА ӨҢДЕУ МЫСТЫ ЭЛЕКТРЛІК ТАЗАРТУ

Аннотация: екінші мыс шикізатын қайта өңдеу кәсіпорындарында пайдаланылған электролиттен мысты электроэкстракция әдісімен алу әдетке айналған. Электроэкстракциядан кейін ерітінді құрамында гипс бар никель бар кек (сүзінді) алу үшін әк сүтімен бейтараптандырылады немесе құрамында металл сульфаты бар (Cu, Ni, Zn, Fe) бар кек бөлініп, буланады, ол тауарлық өнім ретінде сатылады. Мысты электрлік тазарту ерітінділерін қалпына келтіру және толық жою кезінде қайталама түсті металдарды селективті бөлудің тиімді технологиялық шешімдерін іздеу өзекті болып табылады. Жұмыста Қазақстанның мыс балқыту зауытында түсті металл сынықтарын қайта өңдеуден пайдаланылған мыс электролитін қайта өңдеуді зерттеу келтірілген. Пайдаланылған электролитті өңдеу үшін мырыш возгондары мен калий көмегімен кезең-кезеңмен бейтараптандыру жүргізілді. РН 4,7 дейін мырыш айдаумен бейтараптандырудың бірінші сатысы нәтижесінде құрамында PbO 44,69 %; SnO_2 16,36% болатын тұнба алынды. Тұнбаны сілтілі ерітіндімен өңдегеннен, 90^0С температурада карбонизациялағаннан және балқытқаннан кейін құрамында SnO_2 16,36% бар металл қорғасын мен қалайы бар шлак алынды. Калий 7,1-ге дейін бейтараптандырудың екінші кезеңінің нәтижесінде CuO 76,45% болатын тұнба алынды. Калий 9,5-ке дейін бейтараптандырудың үшінші сатысынан кейін NIO 27,63% және ZnO 55,75% болатын тұнба алынды. Тұнбаны 100 г / дм^3 кон бар ерітіндімен өңдегеннен кейін құрамында ZnO 225,0 г/дм^3 және тұнба бар құрамында мырыш бар ерітінді алынды, оны қыздырғаннан кейін құрамында NiO 89,14% бар никель оксиді алынды.

Түйін сөздер: мыс электролиті, құрамында мырыш бар өнім, калий, құрамында мыс бар тұнба, металл қорғасын, шлак.

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ПЕРЕРАБОТКА ПРОМПРОДУКТОВ ПРИ УТИЛИЗАЦИИ РАСТВОРОВ ЭЛЕКТРОРАФИНИРОВАНИЯ МЕДИ

Аннотация: на предприятиях по переработке вторичного медного сырья из отработанного электролита медь принято извлекать методом электроэкстракции. После электроэкстракции раствор нейтрализуют известковым молоком с получением гипсового никельсодержащего кека или упаривают с выделением кека содержащего сульфаты металлов (Cu, Ni, Zn, Fe), который реализуется как товарный продукт. Поиск эффективных технологических решений селективного выделения вторичных цветных металлов при регенерации и полной утилизации растворов электрорафинирования меди носит актуальный характер. В работе приведены исследования переработки отработанного электролита меди от переработки лома цветных металлов на медеплавильном заводе Казахстана. Для переработки отработанного электролита проведена стадийная нейтрализация с использованием цинковых возгонов и поташа. В результате первой стадии нейтрализации цинковыми возгонами до рН 4,7 получен осадок с содержанием PbO 44,69 %; SnO_2 16,36 %. После обработки осадка щелочным раствором, карбонизации и плавки при температуре 900^{o}С получен металлический свинец и оловосодержащий шлак с содержанием SnO_2 16,36 %.В результате второй стадии нейтрализации поташом до рН 7,1 получен осадок с содержанием CuO 76,45 %. После третьей стадии нейтрализации поташом до рН 9,5 получили осадок с содержанием NiO 27,63 % и ZnO 55,75 %. После обработки осадка раствором, содержащим 100 г/дм^3 КОН, получили цинксодержащий раствор с содержанием ZnO 225,0 г/дм^{3}и осадок, после прокалки которого получили оксид никеля с содержанием NiO 89,14 %.

Ключевые слова: электролит меди, цинксодержащий продукт, поташ, медьсодержащий осадок, металлический свинец, шлак.

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