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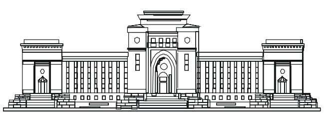

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PHISYCO-CHEMICAL ANALYSIS OF A TIN-CONTAINING ELECTROLYTIC SLIME

Abstract. This article is devoted to the problems of formation and of determination of the tin-containing slime composition formed during the electrolytic tinning. Slimeformed in acidic or subacidelectrolytes is a product of hydrolysis containing hydrated salts and metal hydroxides, which is shown in this work on the example of tin.In alkaline electrolytes, along with specified tin compounds and impurity metals, sulphate compounds of metals can be present in the slime, since sodium sulfate is added to stabilize and increase the ionic strength of the electrolyte.

To assess the quality of tin-containing slime proposed for the research by Kasting LLP (Almaty), elemental, X-ray fluorescent, X-ray phase and IR spectroscopic analysis methods were used using the appropriate modern analytical equipment.

From the presented analysis results, it follows that the main components of the slime are tin, lead, copper and iron, with tin accounted for more than 11%, which implies that this slime could be considered as a rich secondary source of tin.Copper, lead and tin are present in the slime as Sn_3O_4, SnO_2, $CaSn(OH)_6$, Na_2SnO_3, $Na_4(Sn(OH)_3)_2(Sn_2O(OH)_4)$, $Na_2Sn(OOH)_6$, $CaCu(C_2H_3O_2)_4(H_2O)_6$, $Cu_3(SO_3)_2(H_2O)_2$, PbO_2, $NaPb_2(CO_3)_2OH$. Regarding other important metals, it is worth mentioning the presence of gold and silver in the slime.

It was additionally confirmed by IR-spectroscopic analysis that the slime contains the following groups: $[OH]^-$, $[SO_3]^{2-}$,$[HSO_3]^-$and $[SO_4]^{2-}$.

Keywords. Tin, tin-containing slime, hydrolysis, physico-chemical analysis, secondary raw materials.

Introduction. Nowadays, the production and consumption of tin in the world reaches significant proportions.Tin consumption data of 171 companies over the world, with a total refined tin consumption share of more than 47% in 2009, is shown in the table 1(source : ITRI – International Tin Research Institute).

Table 1 – Data on the consumption of primary refined tin in the world[1]

Product name	Refined tin consumption by year, thousand tons						
	2004	2005	2006	2007	2008	2009	2010
Solders	157.3	168.5	197.2	203.4	182.3	172.0	194.3
Tinplate	60.5	59.7	59.6	58.1	57.2	53.8	58.8
Chemicals	49.7	48.7	50.0	52.5	47.8	42.5	51.0
Brass and bronze	20.2	20.0	21.5	21.1	20.1	18.2	19.5
Glass	6.6	6.8	6.7	7.7	6.5	7.5	7.0
Other	33.5	31.9	32.7	30.0	34.5	26.2	29.7
Total	**327.7**	**335.5**	**367.7**	**372.7**	**348.4**	**320.2**	**360.3**

As follows from Table 1, more than 50% of tin consumption is accounted for by solders, and a significant amount - by tinplate, chemicals and alloys production. Moreover, it should be noted, that the consumption of solders tends to increase.

It is known that there is a shortage of tin in the world, so the return of secondary tin to production is of particular importance. Secondary tin is used in industry mainly in the form of alloys (bronze, solders, babbits), but some of it comes back in the form of pure metal during regeneration from various wastes.

Classification of tin-containing industrial wastes [2, 3], processed for the production of secondary alloys or pure tin, is given in Table 2.

Table2 – Classification of the main types of tin-containing waste

Type of tin waste	Content of tin, %
1. Bell bronze scrap	$\geq$6.0
2. Cannon bronze scrap	$\geq$6.0
3. Shavings and scrap of high-tin bronzes	$\geq$6.0
4. Paper factories grid	$\geq$2.0
5. Shavings and scrap of tin bronzes	$\geq$2.0
6. Tin foil	$\geq$ 98.5
7. Scrap and shavings of high-tin babbits	83.0
8. Scrap of typographic tin foams, babbits, die cutting, shavings of lead-tin babbit	$\geq$ 5.0
9. Tin scraps, used cans and other	1-2
10. Lead-antimonic-tin solders (depending on the brand of solder)	1-95
11. Sludge, electrolytic sludge, etc.	$\geq$1.0

The processing of tin-containing scrap and solders is most often done by melting them and by producing another tin-containing product (solders, bronze, pure tin). Tin scraps and bronze are often recycled using electrochemical technologies [2, 4-8].

A number of authors [9-11] propose to make the recycling of ash and electrolytic slimes,containing lead along with tin, in two main stages.

The first stage is soda-reduction melting, the main technological parameters of which are the following: temperature - 1523 + 50 K, consumption of reducing agent - 8.5%, consumption of soda - 3%. At this stage, the yield of the alloy reaches 48-52%, and the extraction of lead and tin into the alloy reaches 98 and 95%, respectively. At the second stage, it is proposed to conduct electrolysis using fluorosilicic and fluoboricelectrolytes and the following process conditions :current density - 200 A / m2, cathode sediment build-up time - 96 hours, electrolyte circulation - 0.9 m3 / h, electrolyte adjustment for lead.An alloy with a content of 60-65% Pb and 34-39% Sn, which is useful as analloy for the preparation of solders [9-11], is produced on the cathode.

Such types of waste always attract the attention of scientists and researchers. The electrolytic slime, which was chosen as the object of this research, is of particular interest, in view of its changing composition depending on the type of electrolyte.

In acid and subacid sulfuric acid electrolytes, tin-containing slime is mostly formed as a result of the hydrolysis proceeding through the following reactions in two stages [12, 13].

The first stage of the hydrolysis:

$SnSO_4 \rightleftarrows Sn^{2+} + SO_4^{2-}$ (dissociation of salt); (1)

$Sn^{2+} + HOH \rightleftarrows SnOH^+ + H^+$ (cation hydrolysis); (2)

$Sn^{2+} + SO_4^{2-} + HOH \rightleftarrows SnOH^+ + SO_4^{2-} + H^+$ (3)
(ionic equation);

$2SnSO_4 + 2H_2O \rightleftarrows [Sn(OH)]_2SO_4 + H_2SO_4$ (4)
(molecular equation).

The second stage of the hydrolysis:

$[Sn(OH)]_2SO_4 \rightleftarrows 2SnOH^+ + SO_4^{2-}$ (dissociation of salt); (5)

$SnOH^+ + HOH \rightleftarrows Sn(OH)_2\downarrow + H^+$ (cation hydrolysis); (6)

$SnOH^+ + SO_4^{2-} + HOH \rightleftarrows Sn(OH)_2 + SO_4^{2-} + H^+$ (7)
(ionic equation);

$[Sn(OH)]_2SO_4 + 2H_2O \rightleftarrows 2Sn(OH)_2 + H_2SO_4$ (8)
(molecular equation).

In the case of muriatic tin-containing electrolytes, that are used less often than sulfuric acid electrolytes, hydrolysis is also proceeded in two stages [12, 13].

The first stage of the hydrolysis :

$SnCl_2 + HOH \rightleftarrows SnOHCl + HCl$ (molecular equation); (9)

$Sn^{2+} + 2Cl^- + HOH \rightleftarrows SnOH^+ + Cl^- + H^+ + Cl^-$ (10)
(complete ionic equation);

$Sn^{2+} + HOH \rightleftarrows SnOH^+ + H^+$ (net ionic equation). (11)

The second stage of the hydrolysis:

$SnOHCl + H_2O \rightleftarrows Sn(OH)_2 + HCl$ (12)
(molecular equation);

$SnOH^+ + Cl^- + H_2O \rightleftarrows Sn(OH)_2 + H^+ + Cl^-$ (complete ionic equation); (13)

$SnOH^+ + H_2O \rightleftarrows Sn(OH)_2 + H^+$ (net ionic equation). (14)

When dissolving tin salts, so-called combined hydrolysis may occur, which occurs in two cases: by dissolving a tin salt, the anion of which is an anion of a weak acid, and by mixing solutions of two salts that contain hydrolyzablecations and anions. Often hydrolysis of this type is an almost irreversible reaction.

Such situation can be considered using the following example of tin silicate (II): the dissociation of the tin salt when in contact with water proceeds according to the scheme of cation and anion hydrolysis in two stages.

$SnSiO_3 \rightleftarrows Sn^{+2} + SiO_3^{2-}$, (15)

The first stage of the hydrolysis:

$Sn^{+2} + H_2O \rightleftarrows SnOH^+ + H^+$ (cation hydrolysis); (16)

$SiO_3^{2-} + H_2O \rightleftarrows HSiO_3^- + OH^-$ (anion hydrolysis); (17)

$Sn^{2+} + SiO_3^{2-} + H_2O \rightleftarrows SnOH^+ + HSiO_3^-$ (18)
(general hydrolysis equation).

The second stage of the hydrolysis:

$SnOH^+ + H_2O \rightleftarrows Sn(OH)_2 + H^+$ (cation hydrolysis); (19)

$HSiO_3^- + H_2O \rightleftarrows H_2SiO_3 + OH^-$(anion hydrolysis); (20)

$SnOH^+ + HSiO_3^- + H_2O \rightleftarrows Sn(OH)_2 + H_2SiO_3$ (21)
(general hydrolysis equation).

Thus, in the electrolyte slime obtained by tinning from acidic and neutral electrolytes, both basic sulfur and chlorine-containing tin salts and tin hydroxides can be detected.

During electrolytic tinning, among alkali electrolytes, along with impurity metals and hydroxides and basic salts of tin, the following could be present in the slime: carbonates sulfates, sulfites, and spongy tin deposit on the cathode.The presence of sulphate and sulphite compounds in the slime is due to the fact that sodium sulfate and sulfite are added to stabilize and increase the ionic strength of the electrolyte.

As a result of the oxidation of tin compounds and sponge tin, tin oxides can be detected in a certain amount - another possible component of electrolytic slimes formed by tinning from alkaline electrolytes [11, 15-16].

The method of tin-containing slimes processing depends primarily on their nature and composition, therefore the purpose of the present work is to determine the composition of the sulfate tin electrolyte slime proposed for its composition study by Casting LLP.

The relevance of the study is justified by the fact that there is currently no industrial production of tin in the Republic of Kazakhstan,thoughthe tinning technology is established, but the resulting electrolytic slimes are not processed, despite the high content of tin.

Results and discussion. Physico-chemical analysis of electrolyte tin-containing slime was done using several methods, each of the analyzes was performed in three parallel experiments. Therefore, the chemical composition of the slime is an average value obtained during the research, and the X-ray diffraction pattern, micrograph and IR spectrum are chosen to be the most representative and consistent with the chemical composition.

Analysis of the slime sample was performed using a Phenom XL scanning electron microscope of the company Phenom-World (Thermo ScientificTM, The Netherlands) under FOV conditions: 50.7 µm, Mode: 15kV-Point, Detector: BSD Full. It showed that both the structure (Figure 1) and the composition (Table 3) of the slime are heterogeneous.

When examining the microphotography of the in the slime, it can be seen that there are phases of different nature and size. In the composition of the slime were found copper, lead, antimony, aluminum and iron in addition to tin. It means that, the slime is multicomponent and may be difficult to process.

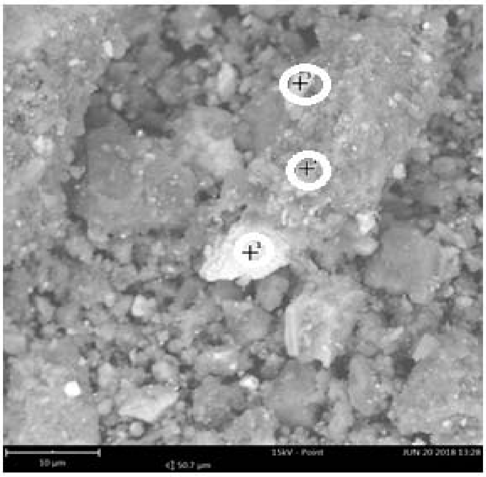

Figure 1 - Micrograph of electrolytic tin-containing slime indicating the points at which its elemental analysis was performed

Table 3 - Elemental analysis of tin-containing slime obtained using the Phenom XL electron microscope

Element	Content					
	at. %	wt. %	at. %	wt. %	at. %	wt. %
	Point on the microphotography (Figure 1)					
	1		2		3	
O	59.40	32.70	68.96	50.08	65.56	44.90
Sb	7.52	31.49	-	-	-	-
Na	25.56	20.22	21.71	22.65	20.87	20.54
Pb	1.58	11.30	1.13	10.65	0.98	8.66
Sn	-	-	1.41	7.61	1.44	7.33
Si	1.37	1.33	2.10	2.67	1.54	1.86
Ca	-	-	1.09	1.98	0.52	0.88
S	0.98	1.08	0.91	1.33	1.18	1.62
Al	0.77	0.72	1.03	1.27	0.60	0.70
Fe	-	-	0.44	1.10	1.34	3.19
C	2.81	1.16	1.22	0.66	2.69	1.38
Cu	-	-	-	-	3.28	8.92

In order to carry out further research, a more thorough averaging of the slime by the methods of quarting and rolling is done. A preliminary elemental analysis of the given slime is then carried out. The results of the analysis are shown in Table 4. The Fe, Se, Ag, and Au contents were determined by the neutron activation analysis method, the Cu, Ni, Sn, Pb content - by inductively coupled plasma atomic emission spectrometry (ICP-AES). The accuracy of elements identifying is given below. It was not possible to determine silicon in the slime composition by these methods.

Table 4 - Results of preliminary elemental analysis of sulfate electrolytic tin-containing slime

Sample	Contents							
	wt. %							ppm
	Fe	Cu	Ni	Se	Ag	Sn	Pb	Au
1	2.13±0.22	3.2±1.0	0.33±0.11	0.107±0.012	0.189±0.019	11.0±3.3	9.6±2.9	43.5±0.5
2	2.02±0.20	3.3±1.0	0.33±0.11	0.108±0.012	0.188±0.019	11.5±3.5	9.7±2.9	46.0±0.05

From the presented analysis results, it follows that the main components of the slime are tin, lead, copper and iron, with tin accounted for 8-11%, which implies that this slime could be considered as a rich secondary source of tin. Regarding other important metals, it is worth mentioning the presence of gold and silver in the slime.

To refine the elemental composition of the slime, X-ray fluorescence analysis was also performed using Venus 200 AxiosPANalyical B.V. wave diffuser spectrometer. (Holland). The results are presented in Table 5.

Table 5 – The results of X-ray fluorescence analysis of electrolyte slime

Element	Contents, %	Element	Contents, %	Element	Contents, %
O	46.711	Sb	1,103	Se	0,099
S	3.673	As	0,431	P	0,036
Si	3.309	Cl	0,148	Cr	0,131
Na	18.776	Ca	0,718	Ti	0,117
Sn	10.450	Zn	0,550	K	0,115
Pb	6.244	Mg	0,376	Mn	0,040
Cu	3.047	Ba	0,328	Nb	0,026
Fe	1.963	Ni	0,210	Sr	0,011
Al	1.242	Ag	0,144	Other	0,002
Total					100.0

As it follows from the results (Table 5), the Fe, Cu, Ni, Se, Ag, Sn, and Pb contents is within the limits of the preliminary elemental analysis (Table 3), i.e. tin, lead, copper and silver may be significant for the processing of this slime.

The presence of a large amount of oxygen, sulfur and silicon in the samples of the slime (Tables 3-5) suggests that sulfates, oxides, hydroxides, basic metal salts, possibly silicates or silica of various modifications can be detected during the phase analysis.

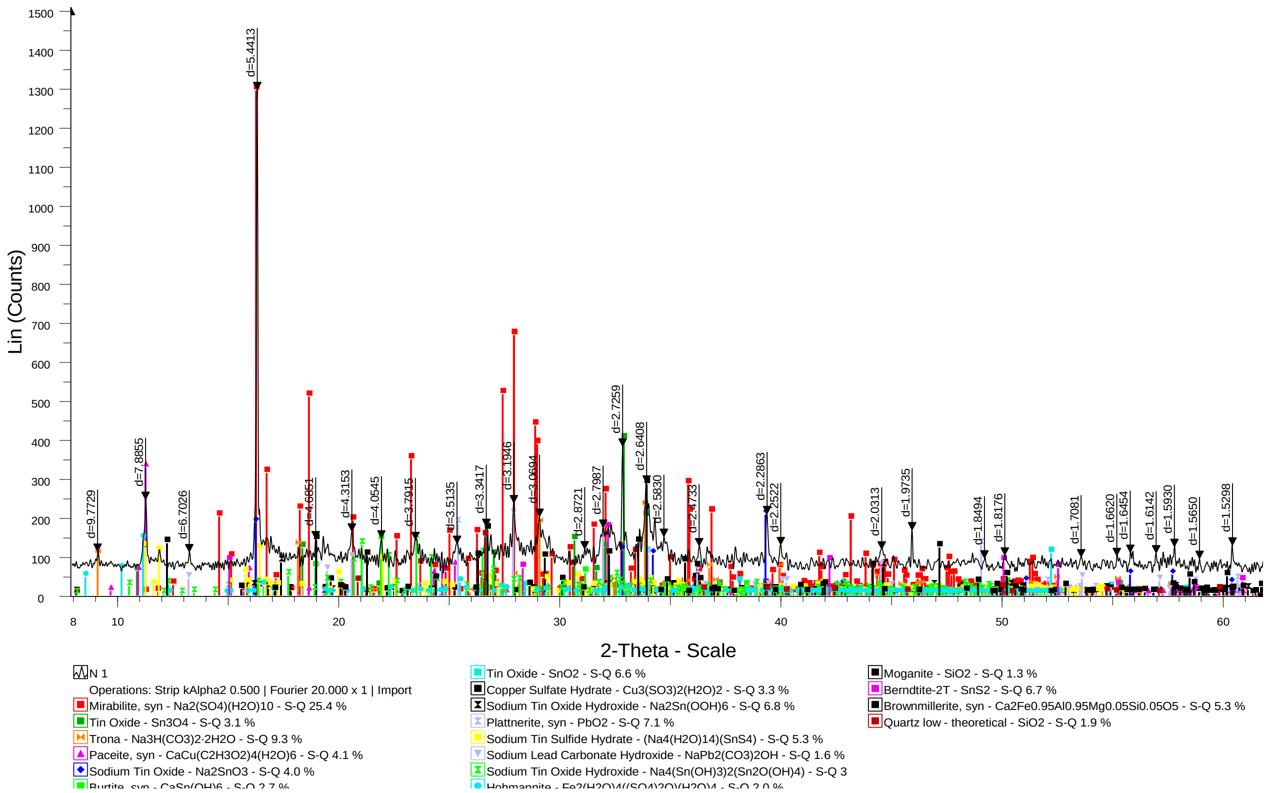

Figure 2 - Diffraction pattern of electrolyte tin-containing slime
(diffractometer D8 Advance, *a*-Cu radiation)

When X-ray diffraction analysis was carried out using the diffractometer D8 Advance (BRUKER), it was found that because of diffraction reflexes overlapping and non-stoichiometric phase composition, it is not possible to clearly select and determine the concentration of phases present in the sample. In addition, there are unidentified peaks in the sample. The results of qualitative X-ray diffraction analysis of tin-containing slime are presented in Figure 2 and Table 6.

Table 6 - Results of qualitative X-ray phase analysis of electrolyte tin-containing slime

Phase	Formula	Phase	Formula
Tin-containing compounds			
TinOxide	Sn_3O_4	Burtite, syn	$CaSn(OH)_6$
TinOxide	SnO_2	SodiumTinOxideHydroxide	$Na_2Sn(OOH)_6$
Berndtite-2T	SnS_2	SodiumTinOxideHydroxide	$Na_4(Sn(OH)_3)_2(Sn_2O(OH)_4)$
SodiumTinOxide	Na_2SnO_3	SodiumTinSulfideHydrate	$(Na_4(H_2O)_{14})(SnS_4)$
Compounds containing ferrous and non-ferrous metals			
CopperSulfateHydrate	$Cu_3(SO_3)_2(H_2O)_2$	Paceite, syn	$CaCu(C_2H_3O_2)_4 \cdot 6H_2O$
Plattnerite, syn	PbO_2	SodiumLeadCarbonateHydroxide	$NaPb_2(CO_3)_2OH$
Brownmillerite, syn	$Ca_2(Fe,Al)_2O_5$	Hohmannite	$Fe_2(SO_4)_2(OH)_2 \cdot 7H_2O$
Other phases			
Quartzlow - theoretical	SiO_2	Trona	$Na_3H(CO_3)_2 \cdot 2H_2O$
Moganite	SiO_2	Mirabilite, syn	$Na_2(SO_4) \cdot 10H_2O$

As follows from the presented results (Fig. 2, Table 6), oxidized compounds-oxides, hydroxides, carbonates and hydrated compounds prevail in the composition of tin-containing slime. The main phases containing tin are the following compounds : Sn_3O_4, SnO_2, SnS_2, Na_2SnO_3, $CaSn(OH)_6$, $Na_2Sn(OOH)_6$, $Na_4(Sn(OH)_3)_2(Sn_2O(OH)_4)$ and $(Na_4(H_2O)_{14})(SnS_4)$. Of the phases containing non-ferrous metals, it should be noted copper-containing phases($Cu_3(SO_3)_2(H_2O)_2$ and$CaCu(C_2H_3O_2)_4 \cdot 6H_2O$), as well aslead-containing compounds (PbO_2 and $NaPb_2(CO_3)_2OH$).The accuracy of the X-ray phase analysis did not allow us to establish the phases in which silver and gold are included.

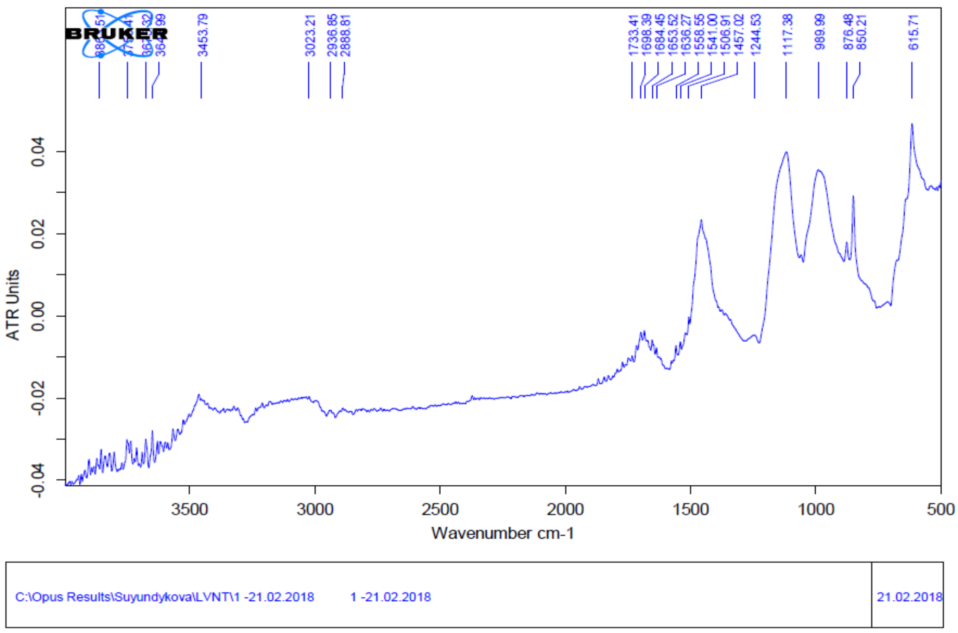

Figure 3 - IR spectrum of tin-containing slime

Silicon dioxide in the form of phases "Quartzlow - theoretical" and "Moganite" was detected in the slime. It may negatively affect the processing of the investigated slime when using both mineral acids and bases as reagents. Crystalline hydrates - sodium sulphate and hydrogen carbonate - can influence the acid processing of slime and electrolysis of the resulting tin-containing solutions. Therefore, when selecting reagents and calculating the thermodynamic characteristics of slime leaching, the presence of these compounds should be taken into account.

In order to clarify the picture of the phase analysis of the tin-containing slime, its group analysis was carried out using an infrared spectrophotometer BrukerAlpha in the wave number range from 4000 to 500 cm^{-1}, the spectrogram of which is shown in Figure 3.

The IR spectroscopic analysis (Figure 3) shows that in the compounds present in the slime, the main functional groups are the following: $[OH]^{-}$, $[CO_3]^{2-}$, $[HSO_3]^{-}$, $[SO_3]^{2-}$ и $[SO_4]^{2-}$. Thus, absorption bands with wave numbers of 3454 and 3023 cm^{-1}, related to the valence vibration of the [OH] group, are observed in the high-frequency region of the spectrum. The absorption bands with a wave number of 1698 cm^{-1} correspond to the deformation vibrations of the [OH] - group.

The absorption bands with a wavenumber of 1457 cm^{-1}correspond to the stretching vibrations of $[CO_3]^{2-}$ groups. The ones with wavenumbers of 1117 cm^{-1} and 990 cm^{-1}correspond to $[HSO_3]^{-}$ and $[SO_3]^{-}$ groups respectively. The ones with wavenumbers of 876 and 850 cm^{-1}also correspond to the stretching vibrations of $[HSO_3]^{-}$ and $[SO_3]^{-}$ groups. An absorption band with a wave number of 617 cm^{-1} corresponding to the stretching vibrations of the groups $[SO_4]^{2-}$ was also detected.

Thus, using the IR spectroscopic analysis method, the presence of hydroxides, carbonates, sulfates and sulfites in the electrolytic tin-containing slime is confirmed. In addition, the presence of hydrosulfite is possible in the slime.

Заключение. During the study of elemental, phase and group composition of tin-containing slime, it was found that both the structure and the composition of the slime are heterogeneous. The main components of the slime are tin, lead, copper and iron,with tin accounted for 8-14 %.

According to X-ray phase analysis, oxidized compounds (oxides, hydroxides, carbonates and hydrated compounds) prevail in the composition of tin-containing slime. Using IR spectroscopic analysis, the presence of hydroxides, carbonates, sulfates and sulfites in the electrolytic tin-containing slime was confirmed and the possible presence of hydrosulfites was detected.

The present research allows to conclude that tin-containing electrolytic slime can be a rich source of secondary tin. This findingis relevant for the Republic of Kazakhstan, which does not have its own industrial production of tin.

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ҚҰРАМЫНДА ҚАЛАЙЫ БАР ЭЛЕКТРОЛИТТІ ҚОЖДЫ ФИЗИКО-ХИМИЯЛЫҚ ТАЛДАУ

Аннотация. Бұл мақалада электролитті тазалау кезінде туындайтынқұрамында қалайы бар қождардың қалыптасуы мен құрамы қарастрылды. Қалайы алу мақаласының мысалында көрсетілгендей қышқылды және әлсіз қышқылды электролиттен, электролитті қож - гидролиз өнімі болып табылады. Сілтілі электролиттерде берілген қалайы қосылыстары мен металл қосылыстарынан бөлек, қожда сульфатты қосылыстар болуы мүмкін, себебі, иондық күшті жоғарылату және тұрақтандыру үшін электролитке натрий сульфатын қосады.

«Кастинг» ЖШС-нен (Алматы қаласы)құрамында қалайы бар қождың құрамын анықтау мақсатында заманауи құрылғылар қолданылатын электронды микроскопия, элементті, рентгенофлуоресцентті, рентгенофазалалы және ИҚ-спектроскопическиялық талдау әдістері қолданылады.

Берілген талдау нәтижелеріненқождың негізгі компоненттері болып мыс, қорғасын және қалайы екені анықталды, соның өзінде қалайының мөлшері 11 % асады, бұл берілген қождың екіншілік қалайыға бай екенін көрсетеді. Мыс, қорғасын және қалайы қож құрамында Sn_3O_4, SnO_2, $CaSn(OH)_6$, Na_2SnO_3, $Na_4(Sn(OH)_3)_2(Sn_2O(OH)_4)$, $Na_2Sn(OOH)_6$, $CaCu(C_2H_3O_2)_4(H_2O)_6$, $Cu_3(SO_3)_2(H_2O)_2$, PbO_2, $NaPb_2(CO_3)_2OH$ қоысылыстар түрінде кездесетіні анықталды. Қож құрамындағы $[OH]^-$, $[SO_3]^{2-}$,$[HSO_3]^-$ және $[SO_4]^{2-}$ топтары ИҚ-спектроскопияәдісімен байқалған.

Түйін сөздер. Қалайы, қалайы құрамды қож, гидролиз, физико-химиялық талдау, екіншілік шикізат**.**

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ФИЗИКО-ХИМИЧЕСКИЙ АНАЛИЗ ЭЛЕКТРОЛИТИЧЕСКОГО ОЛОВОСОДЕРЖАЩЕГО ШЛАМА

Аннотация. Настоящая статья посвящена вопросам образования и определения состава оловосодержащего шлама, образующегося в ходе электролитического лужения. Шлам, образующийся в кислых и слабокислых электролитах – продукт гидролиза, содержащий гидратированные соли и гидроксиды металлов, что показано на примере олова. В щелочных электролитах, наряду с указанными соединениями олова и примесных металлов, в шламе могут присутствовать сульфатные соединения металлов, поскольку для стабилизации и увеличения ионной силы электролита добавляют сульфат натрия.

Для оценки качества оловосодержащего шлама, предложенного для изучения состава ТОО «Кастинг» (г. Алматы), применяли метод электронной микроскопии, элементный, рентгенофлуоресцентный, рентгенофазовый и ИК-спектроскопический методы анализа с использованием соответствующего современного аналитического оборудования.

Из представленных результатов анализа следует, что основными компонентами шлама являются олово, свинец, медь и железо, причем на олово приходится более 11 %, что позволяет считать данный шлам богатым вторичным источником олова. Медь, свинец и олово присутствуют в шламе в виде соединений Sn_3O_4, SnO_2, $CaSn(OH)_6$, Na_2SnO_3, $Na_4(Sn(OH)_3)_2(Sn_2O(OH)_4)$, $Na_2Sn(OOH)_6$, $CaCu(C_2H_3O_2)_4(H_2O)_6$, $Cu_3(SO_3)_2(H_2O)_2$, PbO_2, $NaPb_2(CO_3)_2OH$. Из других значимых металлов следует отметить наличие в шламе золота и серебра. Наличие в составе шлама групп: $[OH]^-$, $[SO_3]^{2-}$,$[HSO_3]^-$ и $[SO_4]^{2-}$ дополнительно подтверждено ИК-спектроскопическим методом анализа.

Ключевые слова. Олово, оловосодержащий шлам, гидролиз, физико-химический анализ, вторичное сырье.

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