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Metallurgy

Hydrothermal treatment of sinters containing thiosalts of non-ferrous metals

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ABSTRACT
Preliminary experiments have shown that the solution to the environmental problem of recycling copper-electrolyte smelting slags is by sulfidization followed by leaching and subsequent separation of selenium, tellurium, arsenic, and antimony from the solution. The first operation of this technology, which provides high selectivity, is sintering. The results obtained indicate the formation of metal thiosalts during sintering in the presence of sodium sulfate and carbonate and a reducing agent at a temperature of 800 ^{0}C. An increase in temperature leads to the melting of individual components of the charge and a slowdown in the process of sulfidization of slag components. At lower temperatures, a decrease in the activity of the charge components is observed. The optimal addition of Na_2SO_4 was 27 % of the slag weight, and Na_2CO_3 - 8.5 % of the slag weight. Reducing agent consumption is 27 % of the slag weight, sintering time is 2 hours. The optimal parameters for leaching the resulting cakes are temperature 90 ^{0}C, L:S ratio = 3:1, leaching duration 2 hours, and sodium sulfide concentration 2 mol/l. The best results for cake melting from cake leaching are temperature 1200 ^{0}C, heating rate 10 ^{0}C/min, and holding time 30 minutes. Charge composition: cake, 30 % soda ash by weight of cake, 11 % activated carbon. During the smelting, a metallized phase was obtained, consisting mainly of lead (90-91 %), and slag was obtained with a residual content of lead and copper of no more than 0.5 %.

Keywords: slag, sintering, sintering, leaching, cake, smelting.

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Introduction

The complex composition of artificial raw materials, comprising various heavy, minor, rare, and noble metals, presents substantial obstacles in devising technologies to transform them into viable products for the market. There is considerable focus on further processing copper smelting residues, including those derived from slag in copper smelting [[1], [2], [3], [4], [5], [6], [7]]. The wide possibilities for purposefully changing the properties of chemical compounds of non-ferrous metals to clarify fundamental issues of the relationship between the structure and properties of a substance, as well as to solve several applied problems, served as the basis for the development of a direction based on the metallurgy of thiosalts. One of the most promising is the improvement of functional properties, as well as the acquisition of new characteristics [[8], [9], [10]].

One of the ways to search for and develop new phases and materials using the method of directed synthesis is the study of phase equilibria. A large amount of research has been carried out in this direction. The phase diagrams of $CuInS_2$-$FeInS_2$, phase equilibria in the Cu_2S-La_2S_3-EuS system, and the polythermal section in the FeS-Sb_2S_3-Sm_2S_3 system were studied. Using DTA and X-ray diffraction methods, phase equilibria in the Cu_2S-Cu_3AsS_4-S system were studied, diagrams of the side quasi-binary systems Cu_2S-Cu_3AsS_4-S and Cu_3AsS_4-S were constructed, the fields of primary crystallization of phases, types and coordinates of non- and monovariant equilibria were determined [[11], [12], [13], [14]]. Phase equilibria in the Cu-Cu_2Se-As system have been studied [15]. The phase equilibria in the quasi-ternary system Ag_2S-SnS_2-Sb_2S_3 along the Ag_2SnS_3-$AgSbS_2$ cross section have been studied. It has been established that the Ag_2SnS_3-$AgSbS_2$ system is a quasi-binary cut of the eutectic type [16]. A phase magnetic diagram of solid solutions of the $CoCrS_4$-$Co_{0.5}Ga_{0.5}Cr_2S_4$ system has been constructed; the most common solid solutions on the diagram are those based on the ferrimagnetic semiconductor $CoCr_2S_4$, which exhibits properties in the field of magnetic ordering when measuring the temperature dependence of the dynamic susceptibility [17].

The electrical-physical properties of (Sb_2S_3)1-x$(PbCuSbS_3)$x solid solutions, which are p-type semiconductors, have been studied [18]. The influence of the ratio of starting substances on the phase composition and electrical-physical properties of $Cu_{1.85}ZnSnS_4$ and $Cu_{1.5}Zn_{1.15}Sn_{0.85}S_4$ solid solutions obtained from binary sulfides and sulfur in the melt has been studied. It has been shown that $Cu_{1.5}Zn_{1.15}Sn_{0.85}S_4$ and $Cu_{1.85}ZnSnS_4$ solid solutions are p-type semiconductors [19]. The crystal structures of $SrLnCuS_3$ (Ln = Er, Yb) compounds were defined using methods of minimizing the derivative difference in the anisotropic approximation for all atoms. The structure of $SrLnCuS_3$ compounds is described by two-dimensional $[LnCuS_3]$ layers formed by distorted CuS_4 tetrahedra and LnS_6 octahedra, between which Sr^{2+} ions are located [20]. The ternary system Nd_2S_3-Ga_2S_3-EuS has been studied. A projection of the liquidus surface was constructed and the boundaries of the glass formation region were determined. It has been established that during thermolysis in an inert atmosphere at a temperature of 1010 K, glass $(Ga_2S_3)_{0.7}(Nd_2S_3)_{0.25}(EuS)_{0.05}$ softens and then crystallizes at 1110 K [21].

The results of studies of the spectral and luminescent properties of non-stereometric Ag-In-S and Cu-Zn-S nanoparticles obtained in aqueous solutions are summarized. The possibilities of size-selective deposition of nanoparticles from colloidal systems with photoluminescence quantum yields have been demonstrated [22]. The FeS-PbS system was studied using thermal, X-ray diffraction and microstructural analysis methods, and microhardness was measured. The system is a quasi-binary section and belongs to the eutectic type [23]. The Cu_2GeS_3-Ag_2GeS_3 system was studied in the temperature range 300-380 K. Current-generating reactions were determined, with the help of which standard thermodynamic functions of formation and standard entropies of Cu_2GeS_3 compounds and Cu_2-xAgxGeS_3 solid solutions were calculated [24].

Due to the dramatically changed composition of the mineral raw materials of copper production, the amount of impurities that must be extracted and disposed of is increasing. Such impurities are lead, zinc, arsenic, and antimony, which negatively affect the main process of copper extraction, reducing technical and economic indicators. The use of previously developed technologies does not allow for achieving high selectivity, as a result which there is a need to create a new complex technology that allows the extraction of all valuable metals into commercial products and the disposal of toxic components.

Experimental part

The object of research is the smelting slag produced by the precious metal shop of the Balkhash copper smelter. Slag is an oxidized material, the chemical composition of which includes, wt. %: 49.5 Pb, 17.3 SiO2, 1.5 Cu, 3.75 Sb, 13.3 Ba, 0.65 As, 4.5 Na, 0.2 Se, 0.24 Te, Au 0.39 g/t, Ag 7.3 g/t, 14.66 others. Experiments to identify optimal conditions for processing slags were carried out in three stages.

Stage 1 - sintering of slag with sodium sulfate and carbonate in the presence of carbon. Sintering was carried out in alundum crucibles in similar furnaces NTS 08/16 Nabertherm GmbH and SNOL 12/16. The sintering temperature is 500-800 ^{0}C, the heating rate of the furnaces to the experimental temperature is 10 ^{0}C/min. The holding time of the charge at the experimental temperature is 2 hours. The crucible was cooled with the furnace. After the experiment, the crucible was broken, the resulting cake was weighed and stored in a desiccator in an inert atmosphere to prevent the decomposition of the resulting sulfides and thiosalts. The sulfidization of non-ferrous metals, including the formation of their thiosalts, was previously established using microprobe analysis of sintered samples.

The 2-nd stage of the experiments consisted of leaching the resulting sinters. Leaching was carried out using standard laboratory equipment with a mechanical stirrer and electrical heating. The sinter was crushed in an amount of 100 g and leached at a ratio of sinter and sodium sulfide solution of 3:1, and 4:1 at temperatures of 50, 75, and 90 ^{0}C, leaching duration 1-2 hours. The content of sodium sulfide in the solution was 0-2 mol/l. The solution after filtration was analyzed for the content of antimony, arsenic, selenium and tellurium; the cake was washed with hot distilled water, dried at 50 ^{0}C and weighed. The cake and the resulting solution were analyzed for the content of major metals.

The 3rd stage consisted of melting the resulting sinter to get lead and noble metals into a separate phase. Meltings were carried out in the same furnaces as sintering at the first stage. The temperature of the melts varied between 1150-1250 ^{0}C, the heating rate to the experimental temperature was 10 ^{0}C/min. The exposure time at the experimental temperature was 30 minutes for all experiments. The sinter was melted with soda ash and activated carbon in various ratios.

At all stages of the experimental work, the problem of identifying optimal conditions for sintering, leaching and melting of leach sinters was solved.

Results and Discussion

Experiments carried out to study the process of sintering precious metal slag with sodium sulfate and carbonate in the presence of carbon indicate the possibility of obtaining a sinter containing metal sulfides and thiosalts. At temperatures of 500-600 ^{0}C, sintering of the charge components practically did not occur, therefore, sintered sinters obtained at a temperature of 800 ^{0}C were used, in which the process of formation of thiosalts took place. A temperature of 800 ^{0}C should be considered critical since at a higher temperature the components begin to melt.

The optimal addition of Na_2SO_4 was 27 % of the slag weight, and Na_2CO_3 - 8.5 % of the slag weight. The consumption of the reducing agent in the experiments was 6, 7, 8 g or 20, 23, 27 % of the weight of the slag, the sintering time was 2 hours. Each experiment was repeated 3 times, and approximately the same results were obtained for the weight of the sinter. The main technological parameters are given in Table 1.

Table 1 – Indicators of the sintering process of slag with charge components

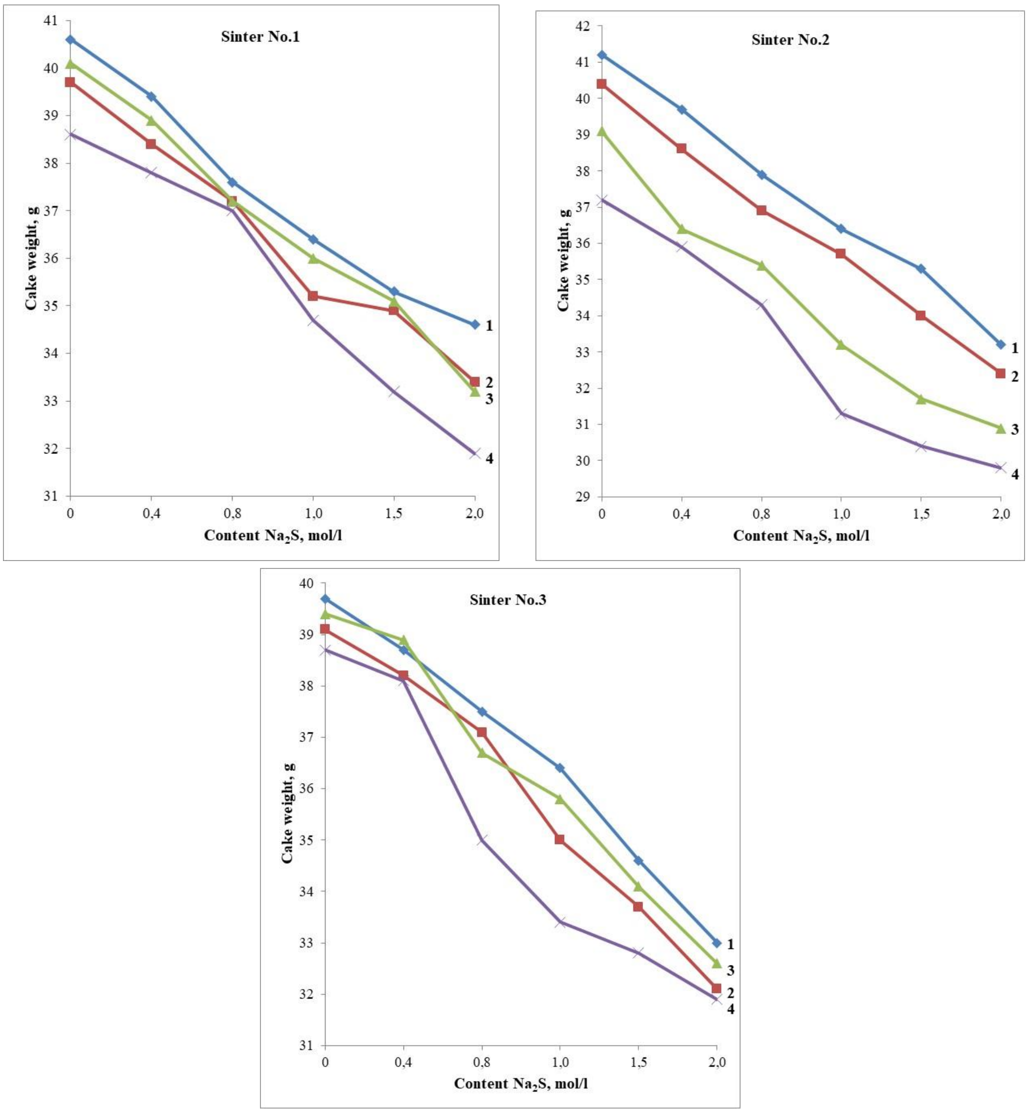
Experiment no.	Sintering conditions	Charge composition, g				Sinter weight, g
		slag	Na_2SO_4	Na_2CO_3	coal	
1	t – 800 ^{0}C τ – 2 h	30	7.65	2.55	6	38.3-39.1
2					7	39.3-39.5
3					8	39.8-39.9

For leaching, cakes No. 1-3 were used, obtained in the corresponding experiments at a temperature of 800 ^{0}C with the addition of carbonate, sodium sulfate and a reducing agent to the mixture. The cake was filtered, dried and analyzed for lead, gold, silver and copper content. The solutions were analyzed for content

The results of leaching of three cakes obtained at 90 ^{0}C are presented in table 2 and in figures 1 and 2.

Table 2 - Compositions of sinter leaching products at optimal leaching parameters (temperature 90 ^{0}C, L:S = 3:1, leaching duration 2 hours, sodium sulfide content in solution 2 mol/l)

Sinter sample number	Solution composition, g/l				Cake composition				
	As	Sb	Te	Se	Pb, %	Cu, %	Au g/t	Ag, g/t	SiO_2, %
1	0.21	3.60	0.16	0.11	63.8	1.8	0.24	4.3	21.0
2	0.19	3.60	0.14	0.09	68.6	1.9	0.19	3.9	19.5
3	0.17	3.51	0.14	0.13	69.3	1.7	0.20	3.8	19.8



Conditions for cake leaching: 1–1 h, L:S=3:1; 2 – 2 hours, L:S=3:1; 3 – 1 hour, L:S=4:1; 4 – 2 hours, L:S=4:1.

Figure 1 – Variation in cake weight as a function of Na_2S concentration, L:S ratio, and leaching duration at 90°C

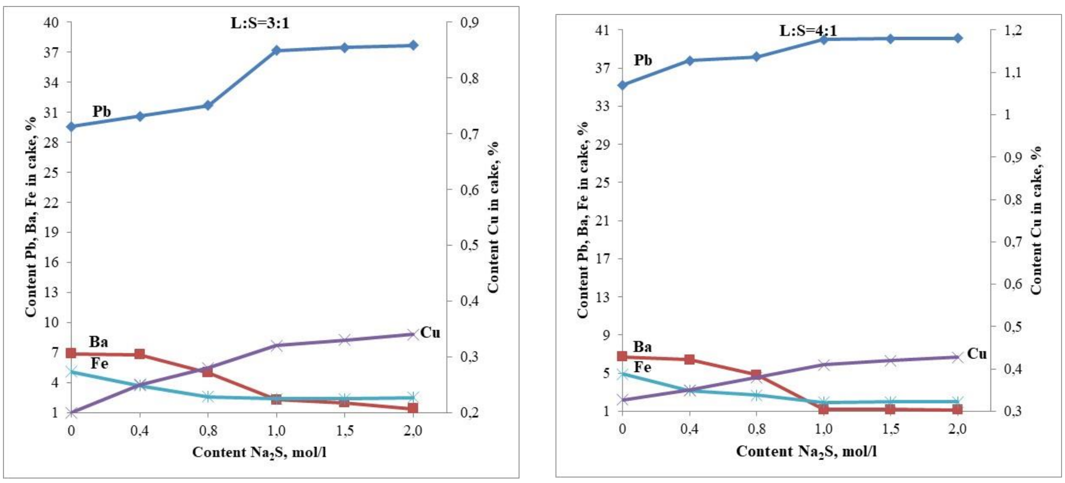


Figure 2 – Variations in metal content in leaching cake No. 2 based on sodium sulfide concentration at 90°C

The change in the weight of the cake remaining after leaching depending on the concentration of sodium sulfide in the solution and L:S is quite predictable. With an increase in the concentration of sodium sulfide in the solution, an increase in L:S and leaching time, the transition of sinter components into the solution increases. This is especially clearly visible when leaching sinter No. 2. Accordingly, during leaching in the residual cakes, the content of copper and lead increases and the content of arsenic and antimony decreases.

It has been established that the most complete leaching of the resulting cakes (slag, sulfate and sodium carbonate in the presence of a reducing agent) occurs at a temperature of 90 ^{0}C, L:S ratio = 3:1, leaching duration of 2 hours and sodium sulfide concentration in solution of 2 mol/l.

The 3rd stage of the experimental work consisted of melting the resulting cakes in order to get lead and noble metals into a separate phase. All previously obtained cakes were combined and averaged, thus, the average composition of the cakes was used for the melts. Meltings were carried out in the same furnaces as sintering at the first stage. The temperature of the melts varied between 1150-1250 ^{0}C, and the heating rate to the experimental temperature was 10 ^{0}C /min. The exposure time at the experimental temperature was 30 minutes for all experiments. The cake was melted with soda ash and activated carbon in various ratios.

Conditions for smelting:

- smelting No. 1: temperature 1150 ^{0}C, heating rate 10 ^{0}C /min, holding time 30 min; charge composition: 100 g cake, 25 g soda ash, 9 g activated carbon;
- smelting No. 2: temperature 1200 ^{0}C, heating rate 10 ^{0}C /min, holding time 30 min; charge composition: 100 g cake, 30 g soda ash, 11 g activated carbon;
- smelting No. 3: temperature 1200 ^{0}C, heating rate 10 ^{0}C /min, holding time 30 min; composition of the charge: 100 g cake, 30 g soda ash, 5 g activated carbon.

The results of the analysis of the compositions of the resulting products are shown in Table 3.

During the smelting, a metallized phase was obtained, consisting mainly of lead (90-91 %), and slag was obtained with a residual content of lead and copper of no more than 0.5 %. Thus, the optimal conditions for the processes of sintering slags with sodium sulfate and sodium carbonate in the presence of a reducing agent, leaching of cakes in a sodium sulfide solution and the process of melting leaching cakes with soda ash and activated carbon were determined.

Table 3 – Results of chemical analysis of cake leaching

Smelting no.	Content in metal			Content in slag, %		
	Pb, %	Au, g/t	Ag, g/t	Pb	Cu	Zn
1	90.81	0.8	14.4	0.2	0.3	0.02
2	91.79	0.9	14.1	0.5	0.4	0.02
3	91.85	0.85	13.8	0.3	0.3	0.01

Conclusions

The results obtained indicate the formation of metal thiosalts during sintering in the presence of sodium sulfate and carbonate and a reducing agent at a temperature of 800 ^{0}C. An increase in temperature leads to the melting of individual components of the charge and a slowdown in the process of sulfidization of slag components. At lower temperatures, a decrease in the activity of the charge components is observed. The best results were obtained with the addition of Na_2SO_4 – 27 % of the slag weight, and Na_2CO_3 – 8.5 % of the slag weight. Reducing agent consumption is 27 % of the slag weight, sintering time is 2 hours.

The optimal parameters for leaching the resulting cakes are temperature 90 ^{0}C, L:S ratio = 3:1, leaching duration 2 hours, sodium sulfide concentration 2 mol/l.

The best results for cake melting are temperature 1200 ^{0}C, heating rate 10 ^{0}C /min, and holding time 30 minutes. Charge composition: cake, 30 % soda ash by weight of cake, 11 % activated coal. During the smelting, a metallized phase was obtained, consisting mainly of lead (90-91 %), and slag was obtained with a residual content of lead and copper of no more than 0.5 %.

Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

CRediT author statement: **S. Kvyatkovskiy:** the idea of research; provided the support for the project and revised the draft; **S. Kozhakhmetov**: writing – review & editing; **A. Semenova**: performed the experiments and calculations; **M. Dyussebekova** and **A. Shakhalov**: data checking.

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Құрамында түсті металдардың тиотұздары бар күйежентекті гидротермиялық өңдеу

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ТҮЙІНДЕМЕ

Алдын ала жүргізілген тәжірибелер мыс-электролитті балқыту қождарын қайта өңдеудің экологиялық мәселесін шешу сульфидтеу, одан кейін шаймалау және одан кейін ерітіндіден селен, теллур, мышьяк, сурьманы бөліп алу арқылы жүзеге асыруға болатындығын көрсетті. Жоғары селективтілікті қамтамасыз ететін бұл технологияның бірінші операциясы күйежентектеу болып табылады. Алынған нәтижелер 800 ^{0}С температурада натрий сульфаты мен карбонатының және тотықсыздандырғыштың қатысуымен күйежентектеу кезінде металл тиотұздарының түзілетінін көрсетеді. Температураның жоғарылауы шикіқұрамның жеке құрамдас бөліктерінің балқуына және қож компоненттерінің сульфидтену процесінің баяулауына әкеледі. Төмен температурада шикіқұрам компоненттерінің белсенділігінің төмендеуі байқалады. Na_2SO_4 оңтайлы қосындысы қож салмағының 27 %, Na_2CO_3 – қож салмағының 8,5 % құрады. Тотықсыздандырғыштың шығыны қож салмағының 27 % құрайды, күйежентектеу уақыты 2 сағат. Алынған сүзінділерді шаймалаудың оңтайлы параметрлері: температура 90 ^{0}С, L:S қатынасы = 3:1, шаймалау ұзақтығы 2 сағат, натрий сульфидінің концентрациясы 2 моль/л. Күйежентекті шаймалаудан алынған сүзінділерді балқыту үшін ең жақсы нәтижелер: температура 1200 ^{0}С, қыздыру жылдамдығы 10 ^{0}С/мин, ұстау уақыты 30 минут кезінде болады. Шикіқұрамның құрамы: сүзінді, сүзіндінің салмағы бойынша 30% кальцинденген сода, 11% белсендірілген көмір. Балқыту кезінде негізінен қорғасыннан (90-91%) тұратын металданған фаза, ал қорғасын мен мыстың қалдық мөлшері 0,5 % аспайтын қож алынды.

Түйін сөздер: қож, күйежентектеу, күйежентек, шаймалау, сүзінді, балқыту.

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Гидротермальная переработка спеков содержащих тиосоли цветных металлов

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АННОТАЦИЯ
Предварительными экспериментами показано, что решение экологической проблемы утилизации шлаков плавки медеэлектролитных шлаков достигается путем сульфидизации с последующим выщелачиванием и последующим выделением из раствора селена, теллура, мышьяка, сурьмы. Первой операцией этой технологии, обеспечивающей высокую селективность, является спекание. Полученные результаты свидетельствуют о прохождении процесса образования тиосолей металлов при спекании в присутствии сульфата и карбоната натрия и восстановителя при температуре 800 ^{0}C. Повышение температуры приводит к оплавлению отдельных компонентов шихты и замедлению процесса сульфидизации компонентов шлака. При более низких температурах наблюдается снижение активности компонентов шихты. Оптимальная добавка Na_2SO_4 составляла 27 % от веса шлака, Na_2CO_3 - 8,5 % от веса шлака. Расход восстановителя – 27 % от веса шлака, время спекания – 2 часа.
Оптимальные параметры выщелачивания полученных кеков - температура 90 ^{0}C, соотношении Ж:Т = 3:1, продолжительность выщелачивания 2 ч., концентрация сульфида натрия 2 моль/л. Лучшие результаты плавки кека от выщелачивания спеков – температура 1200 ^{0}C, скорость нагрева 10 ^{0}C/мин, выдержка 30 мин. Состав шихты: кек, 30 % кальцинированной соды от веса кека, 11 % активированного угля. При плавках была получена металлизированная фаза, состоящая в основном, из свинца (90-91 %) и был получен шлак с остаточным содержанием свинца и меди не более 0,5 %.

Ключевые слова: шлак, спекание, спек, выщелачивание, кек, плавка.

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