Код МРНТИ 53.37.13

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IMPROVING THE TECHNOLOGY OF WAELZATION OF ZINC CAKES

Abstract. Today, about 90% of zinc in the world is produced by the hydrometallurgical method. In zinc plant of Almalyk Mining and Metallurgical Complex a mixed technology is used for zinc production: roasting – leaching – purification from impurities – electrolysis, the extraction of zinc from concentrates is 92.5-94%. The waelz process makes it possible to achieve high rates of zinc extraction, however, it has the following disadvantages: an increase in the content of chlorine, fluorine and reducing agent leads to contamination of solutions with impurities. Experiments were carried out to remove process impurities by water-alkaline washing and calcination of waelz-oxides. Washing of secondary sublimates after preliminary calcination of waelz-oxides allows to effectively solve the problem of reducing the content of reducing agent, chlorine and fluorine from the technological cycle of zinc production.

Key words: *zinc cake, waelz-oxide, charge, washing, waelzation, sublimates, chemical composition, fractional composition, temperature, extraction.*

Мырыш кектерін вельцирлеу технологиясын жетілдіру

Аңдатпа. Қазіргі таңда дүние жүзінде мырыштың 90%-ға жуығы гидрометаллургиялық әдіспен өндіріледі. «Алмалық ТМК» АҚ мырыш өндірісінде аралас технология қолданылады: күйдіру – шаймалау – қоспалардан тазарту – электролиз, концентраттардан мырыш алу 92,5-94% құрайды. Вельц процесі мырыш экстракциясының жоғары көрсеткіштеріне қол жеткізуге мүмкіндік береді, бірақ оның келесі кемшіліктері бар: хлордың, фтордың және қалпына келтіргіштің мөлшерінің жоғарылауы ерітінділердің қоспалармен ластануына әкеледі. Вельц оксидтерін су-сілтімен жуу және күйдіру арқылы технологиялық қоспаларды жою үшін сынақтар жүргізілді. Вельц оксидтерін алдын ала күйдіруден кейін қайталама сублиматты жуу мырыш өндірісінің технологиялық циклінен тотықсыздандырғыштың, хлор мен фтордың құрамын азайту мәселесін тиімді шешуге мүмкіндік береді.

Түйінді сөздер: *мырыш торты, вельц-оксиді, шихта, жуу, вельцтеу, сублиматтар, химиялық құрамы, фракциялық құрамы, температура, экстракция.*

Усовершенствование технологии вельцевания цинковых кеков

Аннотация. На сегодняшний день около 90% цинка в мире производят гидрометаллургическим методом. В цинковом производстве АО «Алмалыкский ГМК» применяется смешенная технология: обжиг – выщелачивание – очистка от примесей – электролиз. Извлечение цинка из концентратов составляет 92,5-94%. Вельц-процесс позволяет достигнуть высоких показателей по извлечению цинка, однако имеет следующие недостатки: увеличение содержания хлора, фтора и восстановителя приводит загрязнению растворов примесями. Проведены испытания по удалению примесей процесса водно-щелочной отмывкой и прокаливанием вельц-окислов. Отмывка вторичных возгонов после предварительной прокалки вельц-окислов позволяет эффективно решить задачу уменьшения содержания восстановителя, хлора и фтора из технологического цикла цинкового производства.

Ключевые слова: *цинковый кек, вельц-окись, шихта, отмывка, вельцевание, возгоны, химический состав, фракционный состав, температура, извлечение.*

Introduction

Today, about 90% of zinc in the world is produced by the hydrometallurgical method. According to this method, the resulting calcine after oxidative roasting of sulfide zinc concentrates is subjected to leaching with sulfuric acid, after which leaching precipitates (zinc cake) are formed with a high content of zinc (18-24%) and copper (0,3-1,5%). Zinc and copper in cakes are in the form of sparingly soluble ferrites. The main method of cake processing is the waelz process, based on reductive-distillation pyrometallurgical processing of cake at temperatures of 1200-1300°C, resulting in sublimates (waelz zinc oxide) and clinker. Waelz-oxide undergoes leaching and further electrolytic extraction of zinc. Clinker is processed to extract copper.

At present, one of the main indicators of any production is its competitiveness, which means high technical and economic efficiency and solution of environmental issues. From these positions, the task of improving the technology of zinc production at the stages of processing intermediate products is relevant. In the zinc production of Almalyk Mining and Metallurgical Complex (AMMC), the main share of the cost of extracted metals is the cost of purchased raw materials. The price of zinc in sulfide zinc flotation concentrates is more than half of the cost of the metal.

The extraction of metal, in turn, is predetermined by the existing technology. Although at present up to 92,5-94% of zinc is extracted into metal from concentrates, the main part of the losses falls on tail products, the extraction of which is not economically feasible. Therefore, the task of reducing the loss of zinc in the main technological process upon receipt is topical.

Research methods and results

In the production of zinc by the hydrometallurgical method, after leaching the calcined zinc concentrate, zinc cakes remain, which contain lead, zinc, silver, and some other valuable metals. To extract lead, zinc and valuable metals from cakes by pyrometallurgical method, zinc cakes are usually subjected to waelz process, during which almost all zinc, most of lead, cadmium and indium, partly copper, precious metals and some other components pass into waelz sublimates. At the same time, silver, the content of which in zinc cake is usually 200-400 g/t, is transferred by 50% or more to the residue of waelz process – copper clinker (3-4% copper), which is processed together with copper concentrate in the copper smelter of AMMC. Having gone through all the stages of copper smelting and electrolyte production, noble metals are concentrated in copper electrolyte slime, which is processed according to a separate scheme [1].

The aim of the work is to conduct research on technological processes that allow increasing the extraction of metals from raw materials due to its separation from zinc cakes by washing and changing the temperature during calcination, waelz process, the study of subsequent processing processes, obtaining more valuable and high-quality commercial products, to the extraction of zinc and its return to technological cycle. To achieve this goal,

Table 1

Chemical composition of zinc cake, %

Кесте 1

Мырыш тортының химиялық құрамы, %

Таблица 1

Химический состав цинкового кека, %

Zn_{total}	Zn_{water}	Zn_{acid}	Cl	F	C	S_{total}	S_{SO_4}	Pb	Fe	SiO_2
21,42	5,59	13,26	0,039	0,0024	0,14	7,69	6,86	6,48	15,21	9,39
Al_2O_3	Cu	Cd	reducing agent	Au, g/t	CaO	MnO	Mg	K	As	In
1,42	2,32	0,21	4,01	1,22	2,67	0,85	0,49	0,28	0,35	0,006

Table 2

Mineralogical composition of zinc cake, %

Кесте 2

Мырыш тортының минералогиялық құрамы, %

Таблица 2

Минералогический состав цинкового кека, %

Product name	Compounds, %					
	Zn_{total}	$ZnSO_4$	ZnO	$ZnO{\cdot}SiO_2$	ZnS	$ZnFe_2O_4$
Zinc cake	21,42	7,97	3,05	3,44	1,07	6,11
	$CuFe_2O_4$	$CuSO_4{\cdot}5H_2O$	Cu_2S	CuO	CdO	$CaSO_4{\cdot}2H_2O$
	5,25	1,45	0,16	0,03	0,02	7,16
	$PbSO_4$	FeS	Fe_2O_3	$CaCO_3$	MnS	MgO
	6,05	1,27	0,56	1,45	1,28	0,45

a technology has been developed to extract zinc and other valuable metals into marketable products.

For research, a sample of zinc cake was taken from zinc plant of AMMC. The results of the chemical and mineralogical composition of zinc cake using the methods of spectral analysis are given in table 1-2. Of industrial interest are the extraction of metals zinc, copper, lead, cadmium, iron, etc. From the spectral analysis of zinc cake [2], it can be seen that zinc cake contains more than 20% zinc, 2% copper and 15% iron. The main chemical compounds of zinc cake are sphalerite, zinc ferrite, copper ferrite, copper sulfate, zinc sulfate, gypsum, lead sulfate and metal silicates.

As shown by the feasibility study of modern methods of zinc cake processing, the waelz process is still one of the effective technologies. The process is popular because of its versatility, ease of maintenance, and other benefits. The advantage of the waelz process is the possibility of recycling zinc-containing waste: lead smelting slag, sludge from wastewater treatment containing zinc, waste from metallurgical and chemical enterprises, tailings of processing plants, etc. [3]. The waelz process makes it possible to achieve high rates of zinc extraction (more than 90%), however, it has the following disadvantages[1] [4]:

- high consumption of imported expensive coke (462 kg of carbonaceous material is consumed per 1 ton of cake);
- high temperature of the waelz process;
- huge consumption of energy – natural gas up to 500 m^3/hour;
- the problem of extracting other valuable components of the cake – copper, iron, gold, silver, lead, etc., due to the lack of cost-effective technology for processing zinc clinker;
- pollution of the environment by products of processing (sulphur-containing exhaust gases, hard-to-recycle clinker);
- expensive, due to the high consumption of coke.

At zinc plants with sulfide raw materials, oxidized zinc-containing materials (waelz-oxides and slag sublimes) are also processed, containing a significant (1-2 orders of magnitude higher than in sulfide raw materials) amount of impurities harmful to hydrometallurgical production (*As*, *Sb*, *Cl*, *F*, etc.). If arsenic and antimony are sufficiently completely precipitated during the hydrolytic purification of sulfate zinc solutions, then the methods used for precipitating chlorine from solutions in the form of copper semichloride by means of copper cake and in the form of silver chloride have significant drawbacks: contamination of solutions with impurities, especially antimony, and the high cost of the reagent.

A large amount of chlorine in neutral solutions fed into the electrolysis process leads to the release of elemental chlorine at the anodes and, as a result, to the formation of compounds with lead (since the anode is made of lead). Due to the fact that these compounds

[1]*Kholikulov D.B. Development of technology for processing technological solutions and cakes of copper, zinc production in order to extract valuable metals. / Abstract of the dissertation of the Doctor of Technical Sciences (DSc): specialty 05.02.01: Materials science in mechanical engineering. Foundry production. Heat treatment and pressure treatment of metals. Metallurgy of ferrous, non-ferrous and rare metals. – Tashkent, 2020. – 208 p. (in Russian)*

are readily soluble in sulfate, they lead to accelerated anodic corrosion and deterioration of the quality of the cathode deposit (zinc released at the cathode).

As a result of the presence of fluorine ions in a neutral solution, the decomposition of the oxide film on the surface of aluminum cathodes is observed. As a result, aluminum begins to dissolve and hydrogen is released. In addition, part of the zinc metal deposited on the surface of aluminum cathodes becomes more rigid than usual and is difficult to bear.

The reducing agent (mainly carbon in waelz-oxides) slows down the electrolysis process and sharply reduces the current efficiency factor. If the amount exceeds the prescribed limit, the aluminum cathodes redissolve the zinc on the surface (reverse dissolution).

At Chelyabinsk Zinc Plant, a complex technology has been introduced, including waelz process, calcination of the obtained waelz sublimates in a large-sized tube furnace and leaching of the calcined products with the extraction of zinc, cadmium and indium into solutions, and lead into lead concentrate [5]. The paper presents the currently accepted three-stage scheme for the hydrometallurgical processing of calcined waelz-oxide, the optimal technological parameters of the process under these conditions, the compositions of the starting material for processing, as well as the resulting lead concentrate. Compared to the processing of a non-calcined product (with pre-washing from *Cl* and *F*), the technology of hydrometallurgical processing of calcined waelz oxide made it possible to increase the productivity of the existing capacities of the leaching department of the hydrometallurgical shop by 33-36% due to an increase in the bulk density of the product (from ~0,92 to 2,0 t/m^3) and reduce losses with commercial lead concentrate by 2,7% zinc, 4,6% cadmium by increasing the solubility of zinc (by 3,8%) and cadmium (by 35,3%).

The invention[2] allows increasing the yield of suitable sinter, improving the quality of the sinter and reducing fuel and energy costs for its production. The mixture contains, wt. %: circulating products of agglomeration 30-40; products of distillation production of zinc 15-25; oxidized zinc raw materials – the rest, and as products of the distillation production of zinc, rimming, dump clinker from rimming waelz or a mixture thereof is used.

The work [6] is devoted to increasing the degree of use of the waelz furnace and reducing fuel consumption, including the operations of mixing, pelletizing and drying zinc cake together with a solid carbonaceous reducing agent and waelz of the rolled material. A refractory calcium-magnesium-containing material, a solid carbonaceous reducing agent, recycled waelz and drying dusts are fed to the stage of mixing and pelletizing. The amount of recycled drying and waling dusts is 15-30% for the essential waelz process. With an increase in coke consumption from 40% to 45%, the waelz treatment of zinc cakes improves.

The method[3] for obtaining zinc oxide by using waelz process of highly basic oxidized zinc ore mixed with quartz-containing material (ferrosilicon production waste slag in the amount of 5-15% by weight of the charge or a mixture of quartz sand and ferrosilicon production waste slag, taken respectively in the amount of 2-5% and 5-10% of the mass of the charge) and a solid carbonaceous reducing agent, reduces the consumption of the reducing agent, increases the degree of extraction of zinc and improves the quality of sublimates. The consumption of the reducing agent is in the amount of 10-30% by weight of the charge. The disadvantages of these methods are the low utilization rate of the waelz furnace and the high fuel consumption.

The method[4] of pyrometallurgical processing of zinc-containing materials includes the preparation of a mixture of zinc-containing material and a solid carbon-containing reducing agent, agglomeration of the mixture and subsequent processing of the well of the agglomerated mixture together with a solid carbon-containing reducing agent. The mixture also contains fine fractions of limestone in the amount of 2-10% by weight of the zinc-containing material, the agglomeration of the mixture is carried out by pressing it, and before the mixing operation or simultaneously with it, the zinc-containing material is crushed, an increase in the zinc content in weltz oxide is provided, a decrease in the consumption of a carbonaceous reducing agent and simplification technologies for processing zinc-containing materials. The disadvantage of this method is the low strength of the pellets, which leads to their destruction during transportation to the furnace for waelz processing.

The method[5] is carried out by treating zinc cakes in waelz furnace with a solid carbonaceous reducing agent and supplying the chlorine-containing material with high-pressure air from the lower cone of the furnace to the one heated to a temperature of 1050-1150°C. The waferable material reduces losses with clinker and reduces the chlorine content in waelz-oxide.

The analysis of the reviewed literature and the practice of operating enterprises on the problem of the technology of waelz process of zinc production cakes allows us to draw the following conclusions.

1. Various technological schemes are used for cake processing, and they are quite complex, many stages, expensive and, most importantly, complete extraction of metals is not achieved due to the strong association of minerals and ions. The production of a high-quality product by the «roasting – leaching with various

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[2]*Chaptykov P.G., Maisky O.V., Loginov N.V., Schneider I.G., Rudko N.A. Charge for agglomeration of zinc-containing materials. / Description of the invention to the copyright certificate SU 1346688 A1. Application number: 4057857. Registration date: 04.18.1986. Date of publication: 10.23.1987. – 4 p. (in Russian)*

[3]*Ospanov Zh.O., Onaev I.A., Najmanbaev M.A., Golev G.D., Sejdaliev M.T., Makhsutov Zh.M., Rakhmanov U.R. Process for producing zinc oxide. / Description of the invention to the copyright certificate SU 1068524 A1. Application number: 3550920. Registration date: 11.23.1982. Date of publication: 23.01.1984. (in Russian)*

[4]*Shashmurin P.I., Posokhov M.Ju., Stepin M.B., Demin A.P., Stukov M.I., Zagajnov V.S. Method of pyrometallurgical processing of zinc-containing materials. / Patent for invention RU 2244034 C1. Application number: 2003127667/02. Registration date: 11.09.2003. Date of publication: 10.01.2005. – 9 p. (in Russian)*

[5]*Kazanbaev L.A., Kozlov P.A., Kolesnikov A.V., Reshetnikov Ju.V. Method of rolling zinc cakes. / Patent for invention RU 2150525 C1. Application number: 98118215/02. Registration date: 05.10.1998. Date of publication: 10.06.2000. (in Russian)*

solvents» scheme is ensured by the use of effective, but rather complex hydrometallurgical schemes. Obviously, the hydrometallurgical processing should be preceded by oxidative roasting, which converts metals into a soluble state. At zinc plant of AMMC, for the processing of zinc cakes, a distillation method is used, which is based on the solid-phase reduction of zinc from cakes (waelz process). As a result of the waelz treatment of zinc cakes, sublimates and lead-zinc oxides are formed, contaminated with iron, antimony, arsenic, chlorine and fluorine, and the solid residue is carbon clinker containing copper, gold, silver and other metals [5].

Methods for processing cakes with the melting of the charge are based on the same principle – melting of cakes in gas-generating furnaces and shaft furnaces. Cakes are melted in a mixture with fluxes without adding other metal-containing materials to obtain lead-zinc fumes contaminated with impurities, copper-iron matte and slag [7].

Due to a significant increase in the volume of processing of oxidized raw materials at AMMC, the issue of removing chlorine, fluorine, and especially organic compounds from sublimates is becoming increasingly important. Industrial tests of the process of water-alkaline washing of waelz-oxides were carried out, the dependence of the parameters of the washing process on the pH of the medium was studied using soda (Na_2CO_3) as a neutralizing agent, under the following conditions – the duration of agitation is 0,5...2 h, t = 25...60°C; W:T = (3...5):1 (figure 1). The results obtained indicate the possibility of sufficiently satisfactory washing of the waelz-oxides from *Cl* (by 80%) and *F* (by 70%) at a pulp pH of 8,0-8,5. The consumption of soda is 20-25 kg/t of waelz-oxides. Increasing the duration of agitation to 2 hours and the temperature of the washing water to 50°C does not have a significant effect on the degree of washing off chlorine and fluorine from the waelz-oxides. With an increase in the ratio W:S from 5:1 to (8...10), the degree of washing off impurities increases by 5-7%. Based on the results of the studies carried out, the following mode is recommended for water-alkaline washing of waelz-oxides from chlorine and fluorine: pH = 8,0...8,5; τ = 1,5 h; t = 20-30°C; W:T = (4...5):1.

The water-alkaline washing process, despite its certain advantages, causes an additional input of water into the process due to the moisture of the washed sublimates and does not allow a significant reduction in the content of inorganic and organic reducing agents. Experiments were carried out to remove impurities by calcining the waelz-oxides. The research results showed that with increasing temperature, a multi-stage decomposition of complex sulfides of copper, iron and other metals were observed, occurring in a wide temperature range – 700-850°C. The change in the content of waelz-oxide impurities depends on the temperature and duration of the process (figure 2-3).

With an increase in the calcination temperature from 700°C to 900°C, the zinc content in the calcined waelz-oxides increases to 62,1%, and the content of the reducing agent, chlorine and fluorine decreases to 0,47%; 0,044% and 0,0009%, respectively.

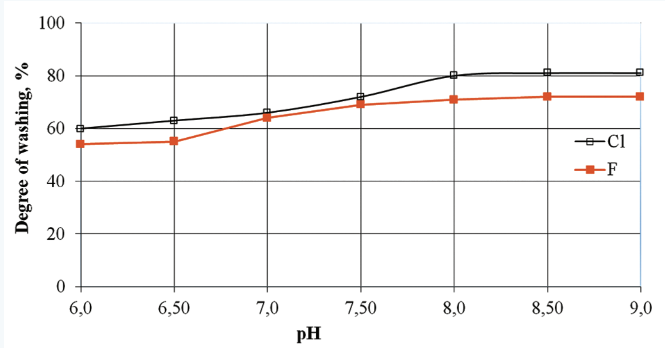

Figure 1. The results of washing waelz oxide with soda depending on the degree washing *Cl* and *F* from pH.
Сурет 1. Дәрежесіне байланысты вельц-оксидін содамен жуу нәтижелері pH деңгейінен *Cl* және *F* жуу.
Рис. 1. Результаты отмывки вельц-оксида содой в зависимости от степени отмывки *Cl* и *F* от pH.

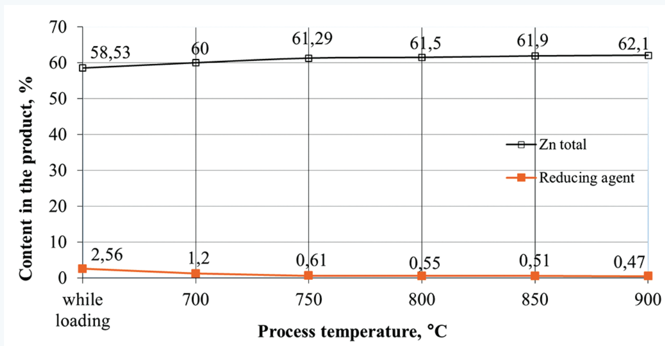

Figure 2. Change the content of impurities (Zn_{total} and reducing agent) waelz-oxide depending on temperature.
Сурет 2. Қоспалар құрамының өзгеруі ($Zn_{жалпы}$ және қалпына келтіргіш) температураға байланысты вельц-оксиді.
Рис. 2. Изменение содержания примесей ($Zn_{общ}$ и восстановитель) вельц-оксида в зависимости от температуры.

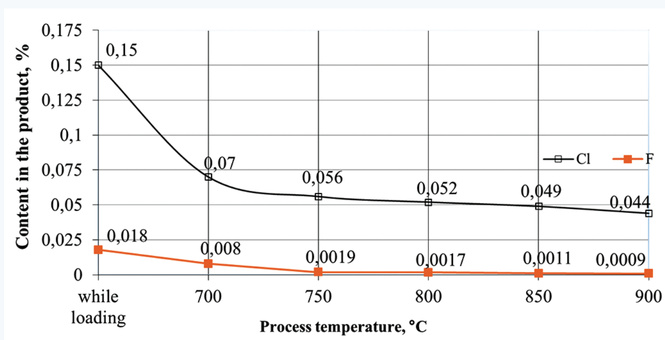

Figure 3. Change in the content of impurities (chlorine and fluorine) of waelz-oxide depending on temperature.
Сурет 3. Температураға байланысты вельц-оксидіндегі қоспалар (хлор және фтор) құрамының өзгеруі.
Рис. 3. Изменение содержания примесей (хлора и фтора) вельц-оксида в зависимости от температуры.

In this case, the forming secondary waelz-oxide (in the amount of 2-3% of the volume of the loaded waelz-oxide) is captured on the bag filter and fluorine, chlorine and the reducing agent accumulate in it. Secondary waelz-oxide (content, %: Zn_{total} – 43,33; Zn_{acid} – 41,05; Zn_{water} – 2,07; Cl – 4,37; S_{total} – 7,6; S_{SO_4} – 7,05; Cd – 2,12; SiO_2 – 1,2; reducing agent – 2,45; F – 0,16; Pb – 10; Cu – 0,86; Fe – 0,72; Au – 0,78; Ag – 232,42) is sent to the roasting process , instead of zinc sulfide concentrate.

Conclusion

Thus, the washing of secondary sublimates with preliminary calcination of waelz-oxides makes it possible to effectively solve the problem of reducing the content of the reducing agent, chlorine and fluorine from the technological cycle of zinc production.

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