



RESEARCH INTO MODERN TECHNOLOGIES OF HYDROMETALLURGICAL PROCESSING OF ZINC CAKES

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Annotation. This article explores several literature on hydrometallurgical processing of zinc cakes. Also, based on the chemical, mineralogical and fractional analysis of zinc cakes, the results of the experiment are presented, and on this basis, a conclusion had given on the hydrometallurgical processing of zinc cakes.

Key words: zinc cake, process, chlorine, fluorine, carbon, raw materials, metal extraction, solution, acid concentration, temperature

Аннотация. В этой статье исследуется несколько источников литературы по гидрометаллургической переработке цинковых кеков. Также на основе химического, минералогического и фракционного анализа цинковых кеков представлены результаты эксперимента, и на этой основе было дано заключение о гидрометаллургической переработке цинковых кеков.

Ключевые слова: цинковый кек, технологический процесс, хлор, фтор, углерод, сырье, извлечение металла, раствор, концентрация кислоты, температура

Introduction

Currently, the zinc cake produced at the zinc plant JSC “Almalyk MMC” is being processed in the process of velting in the vels oven by the pyrometallurgical method. As a disadvantage of this method, we can see the use of large amounts of cake in this process, the loss of gold, silver, copper, lead and other metals with clinker, the complexity of the launch. harmful additives in the resulting product: chlorine, fluorine, carbon, as well as technologies for processing zinc cakes in various hydrometallurgical methods were considered in the article. Analyzing the existing technologies among themselves, in place of the conclusion, we proposed a suitable method for the zinc plant owned by JSC “Almalyk MMC” [1].

This day, about 10 billion tons are mined annually in the world. tons of minerals. Scientific researches are carried out in the world aimed at using man-made waste of the industry, in the form of additional raw materials for the production of metals, due to their large volume, the mass amount of valuable metals exceeds the amount of metals in primary ores. In this regard, the processing of man-made waste will allow enterprises to significantly expand the raw material base, without capital costs for geological, mining and processing works [2-3].

Literature review

In world practice, the transition from pyrometallurgical to hydrometallurgical methods of processing zinc cakes is successfully carried out. As is known, due to ferrite formation during the firing of zinc concentrates during the leaching of the stub, part of the zinc remains in the cakes in the form of ferrite and other insoluble compounds. Together with zinc, lead, gold, silver, as well as up to 50-60% Cu and 30% Cd are almost completely transferred to the leaching residue [4-7].

The zinc content in the cakes and its distribution between the solutions and the leaching residue depend on the quality and nature of the raw materials. When processing



high-grade zinc concentrates with a low content of iron and lead, no more than 10-12% of Zn from the initial amount passes into zinc cakes. In the case of processing low-quality concentrates (with a high iron content), the degree of zinc transition to cakes increases to 20-25% [8].

Approximate composition of cakes, %: 18-23 Zn; 5-12 Pb; 0,1-2,3 Cu; 0,1-0,23 Cd; 0,1 Sn; 0,25 As; 0,8-1,2 CaO; 4-12 SiO₂; 1,0-5,0 Al₂O₃; 6,0 (including sulfur sulfate 5,5 and sulfur sulfide 0,5); 10-30 Fe; 150-425 g/t Ag. The cakes may contain Au, In, Tl, Ga and other elements. Cakes are an additional source of zinc and other components. Therefore, most foreign and all domestic plants process them. Only in some foreign factories (for example, Indian and Korean) they are stored without processing, which is often forced. The processing of leaching cakes with the extraction of valuable components from them significantly affects the economy and ecology of zinc production [9].

Japanese authors [10] proposed to treat a sulfuric acid solution with hydrochloric acid and extract zinc and iron by extraction. Trioctylphosphine oxide dissolved in benzene up to 0.1 mol/dm³ (TORO) has been proposed as an extracting reagent. Extraction during extraction is, %: 65,9 Zn and 95,5 Fe. The process does not require heating more than 100°C and is very efficient.

A method for leaching zinc cakes with spent electrolyte with purging of CO₂ pulp has been developed [11]. Extraction into solution was, %: 88,5 Zn; 88,6 Cu; 75,7 Cd; 65,0 In. During hydrolytic purification of the obtained solutions containing 32,5 g/dm³ of iron, metal losses in the hydrate cake were, under normal conditions, %: 7,0 Zn; 3,5 Cu and 1.8 Cd. Neutralization of solutions during hydrolytic purification was performed by ZnO and CaO.

A Japanese patent [12] claims a technology for processing zinc cakes by leaching in dilute sulfuric acid in a reducing atmosphere with purification of the resulting solutions from iron and other impurities harmful to electrolysis, and subsequent flotation of the insoluble residue to extract silver, copper and lead from it.

The US patent [13] proposed to process metals from cakes with sulfuric acid (20-50 g/dm³) at a temperature of 90-94 °C. The resulting secondary cake is sent for melting, the filtered solution is neutralized with the addition of ZnO to pH 5,0-5,3 to purify the solution from iron and the purified solution is sent to electrolysis, and the filtered Fe(OH)₃ precipitate is treated with a solution containing 8 g / dm³ H₂SO₄ at 90 °C to extract zinc into the solution.

Getskin L. S., Margulis E. V. et al. proposed a method [14] for processing zinc cakes by high-temperature leaching, followed by purification of zinc solutions from iron by precipitation in the form of a jarous cake. In order to increase the integrated use of raw materials, jarosite cake was leached in 2,5-3,2% aqueous ammonia solution at 70-100°C.

To extract zinc from cakes containing 22% zinc, of which 75-90% are bound to zinc ferrite, it was proposed [15] to treat cakes at atmospheric pressure with a solution containing 15-300 g/dm³ of sulfuric acid at ~ 60 °C and above (up to the boiling point of the solution). Na⁺, K⁺ or NH⁺ ions must be added to the resulting solution for the precipitation of iron in the form of double sulfates of the jarosite type at 60 °C.

The patent [16] proposed leaching of zinc cake and subsequent flotation in the presence of sodium hyposulfite and ammonium sulfate at their ratio of 1: (0,5-3,0), pulp pH 4-5,5 and temperature 30-60°C. Silver was isolated by cementation with zinc.

Experiments were carried out [17] on synthetic zinc ferrite with a ZnO ratio:Fe₂O₃ = 1:2. At the same time, optimal leaching conditions were determined: duration 3-4 hours, sulfuric acid content 4 mol/dm³, temperature 60-80°C. Liquid extraction was carried out with a solution of trioctylphosphine oxide reagent in benzene from a 4 mol sulfuric acid solution containing zinc and iron, with the addition of HC1 in an equivalent volume. Zinc



was concentrated in the organic phase, re-extracted with dilute sulfuric acid, followed by electrolysis. Extraction was, %: 75,0 Zn; < 2,5 Fe.

A method for processing zinc cakes by leaching with a solution of sulfuric acid at 108-113°C in the presence of zinc and iron sulfate is proposed [18].

The publication [19] presents the results of a study of a hydrometallurgical method for processing zinc cakes according to a scheme including: single-stage leaching of zinc cinder, high-temperature decomposition of zinc cakes, precipitation of iron from solutions in the form of complex insoluble salts, hydrolytic purification of solutions.

The issue [20] is devoted to various variants of goethite processes for the purification of zinc-containing sulfuric acid solutions from iron. The morphology of formed sediments based on goethite, ferrihydrite and jarosite depending on the regime is considered in detail. It is characterized by a positive effect of the addition of solutions at the stage of neutralization to pH = 3-3,5 of burnt iron-jarosite precipitation.

In [21], it was proposed to use the jarosite process for cakes from acid leaching. The resulting secondary cake does not contain copper, cadmium, contains little zinc and an increased amount of lead, silver and gold and can be sent to the charge for lead smelting. The most favorable conditions for the decomposition of zinc ferrite are: leaching at 95 ° C with the addition of sulfuric acid to the final pH < 1.5, then neutralization of the solution to 10 g/dm³ of sulfuric acid with the addition of ammonium or sodium salts to precipitate a corrosive iron precipitate.

In Japan [22], the issue of the need to introduce zinc production technology using iron deposition in the form of jarosite, developed in Australia, Norway and Spain on the basis of high-temperature leaching of zinc cinder (up to 100°C), followed by the addition of sodium and potassium salts to the solution and the removal of iron in the form of jarosite, was considered. At the same time, 95-98% of zinc is extracted.

A method has been patented [23] for removing iron from acidic solutions and ZnSO₄ pulps by repeated precipitation in the form of a jarositic compound, at <80°C by bringing the pH of the solution to 1,0-2,8 with an additive containing a zinc neutralizer. The resulting solid residue is separated, Na⁺, K⁺ or Ml⁴⁺ ions are added to the solution and the solution is heated above 80°C, precipitating the bulk of the iron in the form of a jarositic compound. A concentrate containing ZnO, ZnCO₃, Zn(OH)₂ is recommended as a neutralizer.

A method has been patented [24] for cleaning solutions from iron according to the scheme: precipitation of iron at pH < 4 in the form of basic sulfate KFe₃(SO₄)₂(OH)₆, jarosite or goethite, decantation of the resulting sludge and filtration of the bottom drain with washing the sediment with water. In this case, a fast and effective countercurrent washing into an aqueous solution of zinc and copper is provided before they pass (as a result of hydrolysis) into the insoluble form of the basic sulfate.

The issue [25] presents the results of studies of the deposition rate of iron from a solution with the following jarosite-forming additives: K₂SO₄, Na₂SO₄ and (NH₄)₂SO₄. jarosite-forming additives and elevated temperature improve the deposition of iron. The K₂SO₄ additive has the greatest positive effect.

The issue [26] presents the results of laboratory studies of the deposition of jarosite in 4 sequentially connected agitators. At the initial concentration of Fe(III) 25 g/dm³, its yield in the precipitate was 92,0-93,2%, the residual content is reduced to 1,7-2,0 g/dm³, meeting the technology requirement.

Objects of study

A sample of zinc cake from the Zinc Plant of JSC "Almalyk MMC" was selected for the research. The fractional composition, the results of the chemical and mineralogical



composition of zinc cake using spectral, chemical and mineralogical methods of analysis are given in Tables 1-2. Besides zinc, copper, lead, cadmium, iron, etc. are of industrial interest for the extraction of metals.

The spectral analysis of the zinc cake (Fig.1) shows that the composition of the zinc cake is more than 20% zinc, 2% copper and 15% iron. The main chemical compounds of zinc cake are sphalerite, zinc ferrite, copper ferrite, metal silicates, copper sulfate, zinc sulfate, gypsum and sphalerite, lead sulfate. The microstructure of the sample of the zinc cake sample is shown in Fig. 2 and Table 5.

Table 1

Fractional composition of zinc cake

Size class, mm	Fraction output	
	g	%
+40	38,5	1,925
-40+20	227	11,35
-20+10	607	30,35
-10+5	645	32,25
-5+2	416	20,8
-2+1	14	0,7
-1+0,315	15	0,75
-0,315+0,140	10	0,5
-0,140	27,5	1,375
Total:	2000	100

Table 2

Chemical composition of zinc cake, %

Product Name	Components, %								
	Zn _{tot}	Zn _w	Zn _c	C	S _{tot}	S _{SO4}	Pb	Fe	SiO ₂
Zinc cake	21,42	5,59	13,26	0,14	7,69	6,86	6,48	15,21	9,39
	Al ₂ O ₃	Cu	Cd	CaO	MnO	Mg	K	As	In
	1,42	2,32	0,21	2,67	0,85	0,49	0,28	0,35	0,006

Table 3

Mineralogical composition of zinc cake, %

Product Name	Components, %					
	Zn _{tot}	ZnSO ₄	ZnO	ZnO·SiO ₂	ZnS	ZnFe ₂ O ₄
Zinc cake	21,42	7,97	3,05	3,44	1,07	6,11
	CuFe ₂ O ₄	CuSO ₄ ·5H ₂ O	Cu ₂ S	CuO	CdO	CaSO ₄ ·2H ₂ O
	5,25	1,45	0,16	0,03	0,02	7,16
	PbSO ₄	FeS	Fe ₂ O ₃	CaCO ₃	MnS	MgO
	6,05	1,27	0,56	1,45	1,28	0,45

Table 4

Chemical composition of zinc cake by fractions

Size class, mm	Fraction output, %	Components, %							
		Zn	Cu	Pb	Cd	Fe	Al ₂ O ₃	SiO ₂	S
+40	1,925	21,89	3,27	6,48	0,22	17,23	4,11	9,23	7,77



-40+20	11,35	21,60	3,38	6,57	0,22	17,34	4,13	9,18	7,75
-20+10	30,35	21,08	2,35	6,52	0,20	16,48	4,08	9,14	7,66
-10+5	32,25	21,62	2,34	6,49	0,21	16,50	4,15	9,36	7,69
-5+2	20,8	21,66	2,23	6,37	0,20	16,66	4,12	9,29	7,71
-2+1	0,7	21,63	3,33	6,25	0,29	14,60	4,07	9,17	7,74
-1+0,315	0,75	21,95	3,37	6,32	0,29	13,75	4,08	9,19	7,65
-0,315+0,140	0,5	22,45	2,29	6,32	0,28	12,09	4,17	9,14	7,68
-0,140	1,375	21,66	2,08	6,39	0,28	12,07	4,18	9,16	7,73

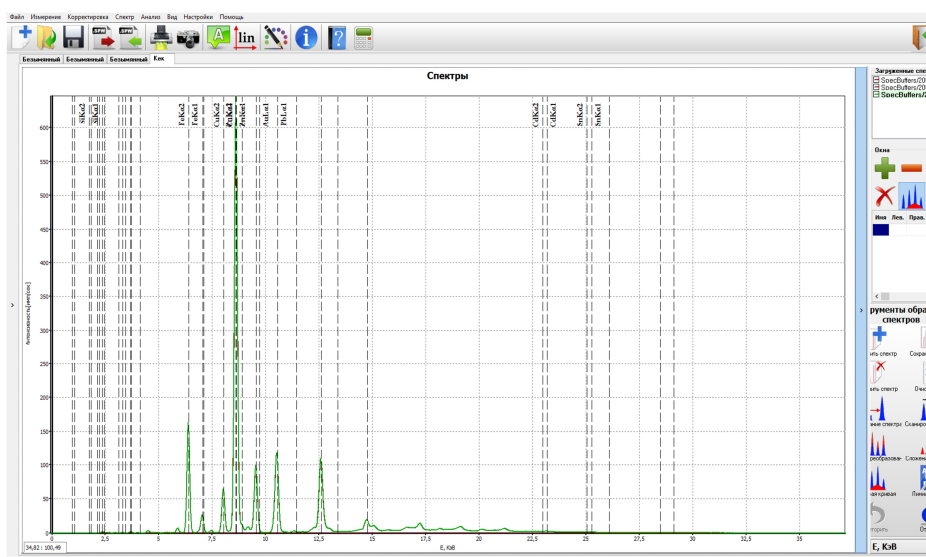
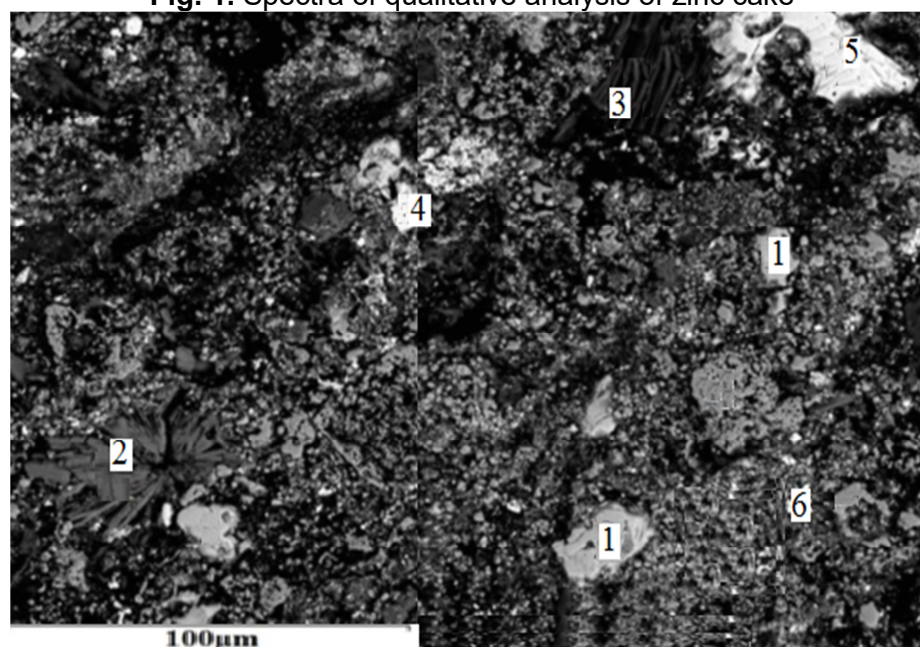


Fig. 1. Spectra of qualitative analysis of zinc cake

Fig. 2. Results of microstructural analysis of the sample
zinc cake

1-sphalerite; 2-ctenazite; 3-gypsum; 4-anglesite and barite; 5-barium sulfate; 6-zinc-copper ferrite

The results of studies on the microstructure of a sample of zinc cake have established that the cake consists mainly of nanoparticles (submicron particles, isolated solid-phase object, size more than 100 nm) in ferritic form. There are also individual



particles of zinc-copper ferrite, zinc-copper sulfate, zinc sulfide, lead sulfates, calcium sulfate, barium sulfate.

Table 5

Composition of the zinc cake sample (results of microstructural analysis)

Minerals	Zn	Cu	Fe	Pb	Cd	Ca	O	Al	Si	Sr	As	Ba	S
Sphalerite	63,4 8	0,50	2,53	-	0,5 2	-	-	-	-	-	-	-	32,9 8
Ktenazit	29,9 6	23,6 4	2,5	-	-	-	31,9 2	-	-	-	-	-	11,9 7
Zinc Ferrite	-	27,9 9	1,79	0,3 4	-	0,3 0	21,7 6	-	47,6 5	-	-	-	0,17
Anglesite and barite	2,96	0,37	2,73	43, 8	-	0,6 7	23,3 9	-	-	1,5 6	-	13,6 6	10,8 7
Gypsum	-	-	0,69	-	-	28, 65	47,0 3	-	-	-	-	-	23,6 3
Barit	-	-	0,25	-	-	-	28,7 3	-	-	3,1 2	-	53,2 8	14,6 2

Research methods

For the research, a sample of zinc cake from the Zinc Plant of JSC "Almalyk MMC" was selected with the following chemical composition, %: Zn_{tot} -21,42; Zn_w -5,59; Zn_a -13,26; S_{tot} -7,69; S_{SO_4} -6,86; Pb-6,48; Fe-15,21; SiO_2 -9,39; Al_2O_3 -1,42; Cu-2,32; Cd-0,21; CaO-2,67; MnO-0,85; Mg-0,49; K-0,28; As-0,35. The main chemical compounds of zinc cake are sphalerite, zinc ferrite, copper ferrite, metal silicates, copper sulfate, zinc sulfate, gypsum and sphalerite, lead sulfate.

To study the process of agitation high-temperature leaching of zinc cake, a special installation LR 1000 basic was used. The experiments were carried out as follows: the zinc cake was crushed to a fraction <0.1 . The crushed zinc cake was placed in a leaching plant, a solution of sulfuric acid was added, heated and kept for a certain time. The temperature was regulated through a microprocessor-based automatic controller. Pulp samples for analysis were filtered through a boom.

Under laboratory conditions [27], studies were conducted on the effect of temperature and acid concentration on the speed and completeness of zinc transition from cake to solution. The process was controlled by the decrease in the concentration of sulfuric acid in the solution and the content of zinc in it. The concentration of sulfuric acid and zinc in the solution was determined by titration according to the standard procedure. After the conducted studies, calculations were made and graphs of the dependence of zinc extraction into the solution on the temperature and the initial concentration of sulfuric acid were constructed. The study of the effect of the duration of the process on the leaching of zinc from the cake with a sulfuric acid solution with a concentration of 100-200 g/l at various temperatures shows that in the initial period (up to 60-90 minutes), the transition of zinc into the solution proceeds very intensively, and after 3-4 hours the dynamic equilibrium of the leaching process is established (Fig. 3-4).

To achieve maximum zinc extraction with minimal transition to a solution of impurities, the duration of the process is chosen 4 hours. Studies have been conducted to study changes in the material composition of high-temperature leaching products obtained under optimal conditions (Table 6). The degree of oxidation of sulfides was 98-99 %.

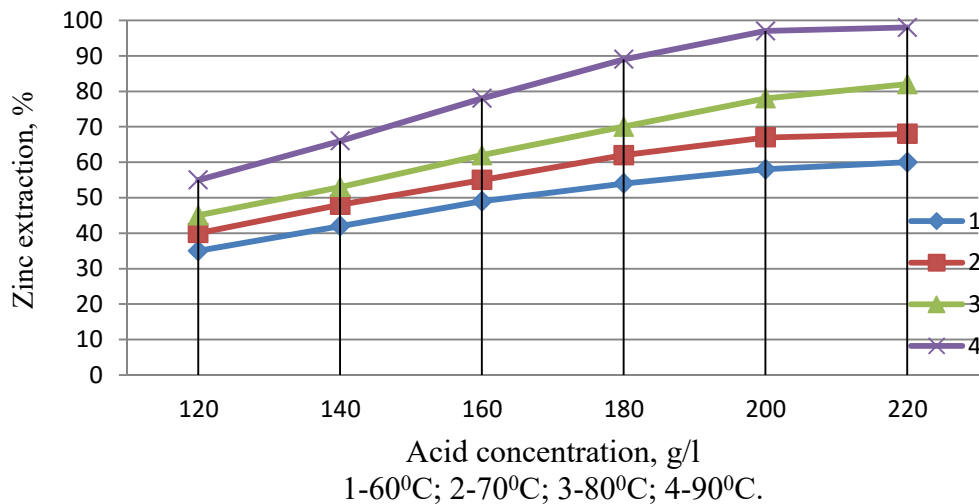


Fig. 3. Dependence of zinc extraction into solution on the initial acid concentration at different temperatures

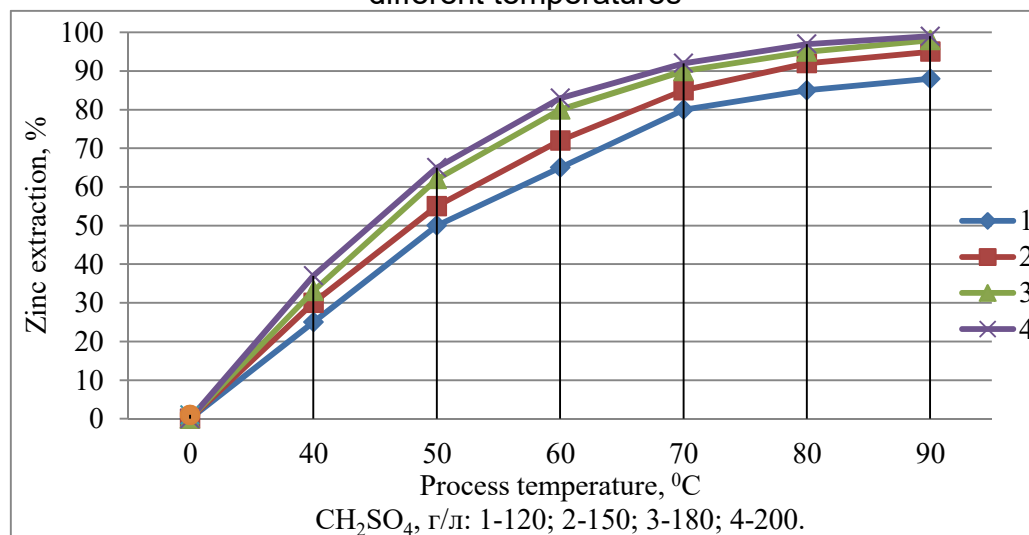


Fig. 4. Dependence of zinc extraction into solution on duration at different concentrations of sulfuric acid

Table 6

Results of experiments on high-temperature leaching of zinc cake, solution and cake composition

Elements	Content in the initial cake, %	Extraction into solution, %	Composition of the solution, g/l	Content in the leaching cake, %
Zn	21,42	98,5	53,4	0,61
Pb	6,48	1,2	0,48	16,4
Fe	15,21	75,2	14,87	6,72
Cu	2,32	92,8	4,28	0,32
Cd	0,21	79,2	0,78	0,2
SiO ₂	9,39	1,2		18,6
Al ₂ O ₃	1,42	0,2	0,2	3,1

An increase in the concentration of sulfuric acid over 200 g/l does not significantly increase the degree of zinc conversion into solution, while the transition of impurities into solution begins to increase. With an increase in temperature, there is a fairly intensive



increase in the concentration of sulfide in the solution. However, at a process temperature of 90°C, with an increase in the duration of the process, a more intensive increase in the degree of zinc extraction occurs. This is due to the fact that sulfates are formed faster at elevated temperatures.

With an increase in temperature in the future, the rate of dissolution will increase. But at the same time, it must be borne in mind that an increase in temperature has a negligible effect on the dissolution of the useful component, while the transition to a solution of impurities increases greatly. When the temperature rises to 100°C, the boiling of the solution begins, and the formation of acid vapors. The necessary hydrodynamic regime to achieve a homogeneous pulp density is provided using a mechanical mixing device.

Conclusion

High-temperature sulfuric acid leaching has proven itself well in the processing of zinc cake, provides selectivity and complexity of processing. The optimal leaching duration is 4 hours with sulfuric acid concentrations of 180-190 g/l, and the leaching temperature is 90-95°C. The end-to-end extraction of zinc into the solution is 97-98,5%, copper 96% with a cake yield of 45-46%. The resulting sulfuric acid solution contains (g / l): zinc – 53,4; copper – 4,28; iron – 14,87. The concentration of zinc in the solution is sufficient for the electrolysis of zinc. However, due to the content of impurities, the solution must be cleaned by hydrolytic methods, which is used by the zinc plant of JSC “Almalyk MMC”.

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