

**PROCESSING OF SOLID ARSENIC-CONTAINING MATERIALS**

(analytical review)

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This article provides an analytical review of scientific and technical developments in the processing of solid arsenic-containing materials. Hydro-, pyrometallurgical and combined methods for extracting arsenic from dust, sublimes, and sludge from metallurgical production are considered. The most acceptable technologies seem to be those in which arsenic is removed from the process at the preliminary stage of processing in the most stable, non-toxic form - arsenic sulfides, which exist in nature as orpiment and realgar minerals. Studies conducted at the Zh. Abishev Chemical and Metallurgical Institute (Karaganda, Republic of Kazakhstan) and the Gidrotsvetmet Institute (Novosibirsk, Russian Federation) on products from a number of arsenic-containing raw material deposits for the removal of arsenic by oxidizing-sulfidizing roasting in a shaft-type furnace have shown the promise of this technology. The removal of arsenic from roasting products was 97-98.5%, simultaneously with the production of compact sulfide material with an arsenic content of 60-70% that does not require special disposal.

**Keywords:** metallurgical production, arsenic-containing materials, leaching, oxidation-sulfiding roasting, acid, autoclave, bacterial oxidation, arsenates, oxides and sulfides of arsenic.

**ҚҰРАМЫНДА МЫШЬЯК БАР ҚАТТЫ МАТЕРИАЛДАРДЫ ӨНДЕУ**

(аналитикалық шолу)

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Бұл мақалада мышьяк бар қатты материалдарды өндеудегі ғылыми-техникалық әзірлемелерге аналитикалық шолу берілген. Металлургиялық өндірістегі шандардан, сублиматтардан және шламдардан мышьяк алудың гидро-, пирометаллургиялық және аралас әдістері қарастырылған. Табиғатта аурипигмент және реальгар минералдары түрінде болатын мышьяк сульфидтері - мышьяк ең тұрақты, улы емес түрінде өндеудің алдын ала сатысында процестен шығарылатын технологиялар ең қолайлы болып табылады. Әбішев атындағы Химия-металлургия институтында (Қарағанды, Қазақстан Республикасы) және Гидроцветмет институтында (Новосибирск, Ресей Федерациясы) мышьяқты жою бойынша бірқатар кен орындарының өнімдеріне жүргізілген зерттеулер. шахталық пеште тотықтырғыш-сульфидтендіргіш күйдіру осы технологияның болашағын көрсетті. Қуырылған өнімдерден мышьяқты кетіру 97-98,5% құрады, арнайы көмуді қажет етпейтін мышьяк мөлшері 60-70% болатын ықшам сульфидті материалды алумен бір мезгілде.

**Түйін сөздер:** металлургиялық өндіріс, құрамында мышьяк бар материалдар, сілтілеу, тотықтырғыш-сульфидтендіргіш күйдіру, қышқыл, автоклав, бактериялық тотығу, арсенаттар, мышьяк оксидтері және сульфидтер.

## ПЕРЕРАБОТКА ТВЕРДЫХ МЫШЬЯКСОДЕРЖАЩИХ МАТЕРИАЛОВ

(аналитический обзор)

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В данной статье приведен аналитический обзор научно-технических работ по переработке твердых мышьяксодержащих материалов. Рассмотрены гидро-, пирометаллургические и комбинированные методы извлечения мышьяка из пылей, возгонов, шламов металлургического производства. Наиболее приемлемыми представляются технологии, по которым мышьяк на предварительной стадии передела выводится из процесса в наиболее устойчивой, нетоксичной форме – сульфидов мышьяка, существующих в природе в виде минералов аурипигмента и реалгара. Исследования, проведенные в Химико-металлургическом институте имени Ж.Абишева (г. Караганда, Республика Казахстан) и институте «Гидроцветмет» (г. Новосибирск, Российская Федерация) на продуктах целого ряда месторождений мышьяксодержащего сырья по выводу мышьяка окислительно-сульфидизирующим обжигом в печи шахтного типа, показали перспективность данной технологии. Вывод мышьяка из продуктов обжига составил 97-98,5 %, одновременно с получением компактного, не требующего специального захоронения сульфидного материала с содержанием мышьяка 60-70%.

**Ключевые слова:** металлургическое производство, мышьяксодержащие материалы, выщелачивание, окислительно-сульфидизирующий обжиг, кислотное, автоклавное, бактериальное окисление, арсенаты, оксиды и сульфиды мышьяка.

**Introduction.** One of the problematic objects in the production of heavy non-ferrous metals is arsenic, which is present in various concentrations in ores and concentrates of non-ferrous and noble metals. During their processing, arsenic leads to various technological difficulties that worsen the quality of the base metal and other by-products. The process is accompanied by environmental pollution with arsenic, its accumulation in intermediate products, mandatory neutralization and storage of arsenic-containing materials.

Metallurgical arsenic-containing industrial objects are conventionally classified into four classes:

- natural raw materials, where arsenic phases

are represented by arsenopyrite, iron arsenides, thioarsenates and arsenides of non-ferrous metals;

- products of metallurgical oxidation processes, where the form of arsenic is arsenates, its oxides;

- products of reduction processes, where arsenic is elemental, arsenides;

- sulfur and phosphorus (chalcogenides) of technical grades, where arsenic is in the form of sulfides and monophosphide.

Products of arsenic purification of gases (dust, sublimes) and waste water (cakes, sediments) in addition to arsenic contain significant amounts of other valuable metals, as shown in Table 1.

The disposal of these materials is a complex technical problem and includes either their

comprehensive processing or safe disposal. The degree of arsenic sublimation is determined by the composition and temperature of the slag and matte, the temperature and composition of the exhaust gases, as well as the partial pressure of oxygen in the gas phase and a number of other factors [1, 2]. Most of the arsenic in the dusts is in the form of arsenates (70%), approximately 15% of the total content is oxides. The zinc and lead content reaches 9.7 and 36.8%, respectively.

During the processing of copper raw materials

and copper-containing semi-finished products of lead production, lead, arsenic and other metals are concentrated in converter dusts. Up to 90% of lead, more than 90% of arsenic, up to 95% of cadmium and more than 65% of rhenium and selenium from the amounts contained in the matte pass into them. Fine converter dusts contain, %: 42-50 lead, 0.3-0.7 cadmium, 15-28 arsenic, 0.3-0.5 bismuth, 0.2-1.0 selenium and 30-70 g / t rhenium. The transfer of these dusts to lead production is one of the main factors in the intra- and inter-plant circulation of arsenic.

**Table 1-Chemical composition of dust and sublimates from metallurgical production, %**

Arsenic containing materials	As	Sn	Pb	Zn	Cu	Fe	S
Fuming sublimates of tin production	7,4	40,8	13,8	4,1	0,05	0,5	3,7
1.	1,6	13,2	11,7	47,8	0,17	-	4,5
2.	2,4	66,2	0,4	2,7	0,03	-	0,8
3.							
Fuming sublimates of lead-zinc production	0,92	-	12,2	58,6	0,75	0,6	1,4
Dust from electric smelting of tin raw materials	1,1	27,6	1,2	28,4	0,03	-	1,1
Polymetallic raw material electric smelting dust	2,7	12,6	14,5	34,6	0,13	-	5
Electric smelting dust from tin refining	1,5	34,2	0,2	0,8	0,16	-	0,4
Electric smelting dust from refining of Pb-Sn alloys	2,6	18,5	27,4	6,9	1,5	0,4	2,5
Dust from agglomeration and shaft smelting of lead concentrates	5,1	-	54,8	11,8	0,22	-	-
Dust from roasting zinc concentrates	2,1	-	31,7	14	0,74	2,1	13,6
Dust from the smelting of copper sulphide concentrates	1	-	8,3	7	13	5,4	28,5
Copper production converter dust	5,9	-	32,2	18,9	3,3	-	-

Copper-containing cakes are mainly added to the smelting furnace charge. This is the simplest and least expensive method, but it seems the least rational, since arsenic is returned to the copper smelting production, accumulates in the electrolyte, thereby worsening the indicators and ecology of the process.

The variety of methods for processing arsenic-containing dusts, sublimates, and sludge from metallurgical production can be conditionally

divided into hydro-, pyrometallurgical, and combined methods.

*Hydrometallurgical methods.* A large set of reagents has been proposed for extracting arsenic from dusts and sublimates: sulfuric, hydrochloric and nitric acids, solutions of sodium and ammonium carbonates, caustic soda, sodium sulfide and others. However, most of them are not selective: heavy non-ferrous metals also pass into the solution, which complicates their subsequent processing [1].

Nevertheless, there are developments that meet the conditions of selectivity.

For example, in work [2], arsenic from arsenic-containing materials was transferred into a solution with sodium hydroxide or sulfide. Experiments have shown that under optimal conditions, the degree of arsenic extraction into the solution in both cases reaches 95-98%. In the case of materials containing arsenic in the form of oxides, good results are achieved using hot water leaching. Extraction from tin production roasting furnace dust (44%  $\text{As}_2\text{O}_3$ ) into solution is 93% As. Satisfactory selectivity of arsenic extraction (88-93%) is achieved by leaching converter dust from lead production with sodium sulfide solutions (concentration 80-100  $\text{g/dm}^3$ , 90-95  $^\circ\text{C}$ , L:S = 6:1, 1 h). With three-stage leaching, arsenic extraction reaches 99%.

Selective extraction of arsenic into an alkaline solution can be achieved by electrolytic leaching of dusts. Optimum process conditions:  $C_{\text{NaOH}} = 90-100 \text{ g/dm}^3$ , L:S = (3-4):1.5, 50-75  $^\circ\text{C}$ ,  $D_k = 1000-3000 \text{ A/cm}^2$ . Lead is released at the cathode in the form of a spongy sediment. To bind and precipitate lead and zinc from the solution during leaching of arsenic with strong hot solutions of caustic soda (100-250  $\text{g/dm}^3$ ), an additional 10-20  $\text{g/dm}^3$  of sodium sulfide is added to the alkaline solution [3].

It is proposed to leach copper smelting converter dusts with 1.3-5.0% As and high contents of non-ferrous metals with sodium sulfide and precipitate arsenic with various reagents, such as calcium oxide, copper or iron sulfate, a mixture of phosphoric acid with calcium hydroxide [4-6]. The scheme provides for the possibility of further processing of sediments to obtain antiseptics for wood preservation or their safe storage. When processing converter dusts with sodium sulfide solutions (80  $\text{g/dm}^3 \text{ Na}_2\text{S}$ , 90  $^\circ\text{C}$ , L:S = 1:6), 92.9% of arsenic was extracted into the solution in the form of thiosalt after 1 hour. With three-stage leaching - up to 99%.

For selective and deep (92-96%) extraction of arsenic from tin production fuming sublimes, alkaline leaching at a NaOH concentration of 100-150  $\text{g/dm}^3$ , L:S = 1:5-10, 80-100  $^\circ\text{C}$ , duration of 1-2 h, stoichiometric  $\text{Na}_2\text{S}$  consumption for binding lead and zinc into sulfides is proposed.

The most common method for extracting arsenic from copper-containing cakes is the alkaline leaching scheme and subsequent precipitation of calcium arsenate. The cake is treated with an alkali solution (70-90  $\text{g/dm}^3 \text{ NaOH}$ ) at 70-90  $^\circ\text{C}$ , a solid:liquid ratio of 5, and a time of 1-3 hours. Extraction into the solution is 90-98% As and no more than 1.0-1.5% Cu. The copper-containing residue can be returned to the batch preparation department of the smelting shop. By introducing lime pulp (with a CaO:As ratio > 1.5) into the solution at 80-90  $^\circ\text{C}$  in 1.5-3 hours, it is possible to precipitate a product containing 90-96% As. This achieves alkali regeneration, and the filtrate is returned to the alkaline leaching stage. As a result, more than 90-92% of arsenic passes into the burial product, and the alkali consumption, taking into account regeneration, is 0.05 kg/kg As.

To extract copper and arsenic from copper electrolyte production middlings, copper-arsenic cake is leached at a mass ratio of  $\text{H}_2\text{SO}_4/\text{Cu} = (1.53-1.54):1$ . The leaching solution is cooled to 20-80  $^\circ\text{C}$ , the copper-containing product is isolated, which is returned to the main production. The mother liquor is evaporated to an As concentration of 600-800  $\text{g/dm}^3$  and used to prepare antiseptic compositions.

Complex processing of lead production dust is complicated by its high content of harmful impurities. The method of granulation with strong sulfuric acid and subsequent heat treatment of the resulting granules in a FB (fluidized bed) furnace allows removing up to 80-85% of arsenic in the "head" of the process and then extracting valuable components from the resulting product [7]. Sublimates are captured in a wet gas cleaning system, where the arsenic content in the solution reaches 20-30  $\text{g/dm}^3$ , and in the sludge up to 40%. The sludge is leached and arsenic is precipitated from the combined solution with lime milk in the form of calcium arsenate (10-12% As).

There are developments of autoclave technologies for extracting arsenic from dusts from processing copper and lead concentrates. Depending on the composition of the dusts, solutions of sulfuric, nitric or hydrochloric acids and a number of other reagents are used for their leaching and removal of arsenic.

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The process of arsenic dissolution in an alkaline solution can be combined with the electrolytic extraction of heavy non-ferrous metals on the cathode. In this case, alkali regeneration occurs, due to which the equilibrium in the system shifts towards arsenic dissolution. Alkaline-electrolytic treatment of converter dust ensures almost complete extraction of arsenic (96-98%) into the solution and lead (91%) into the cathode sponge. Zinc, cadmium, mercury, tellurium are concentrated in an insoluble precipitate. In this case, there is a risk of arsine formation. To prevent this during electrolysis, the original dust is pre-treated with a 4% NaOH solution. The alkaline solution containing arsenic is fortified with alkali to 120 g/dm<sup>3</sup>, cooled to 18-20 °C and, after separating the sodium arsenate crystals, sent for leaching of a new portion of dust. Sodium arsenate is dissolved in water at 60-70 °C and calcium arsenate is precipitated from the resulting solution (50-60 g/dm<sup>3</sup> As) with lime milk, which is sent for disposal [3].

*Pyrometallurgical and combined methods.* At non-ferrous metallurgy plants, up to 30% of arsenic is concentrated in dust and sublimates [8, 9].

When processing sulfide copper concentrates with a low arsenic content (up to 1%) in order to obtain arsenic-free gases during smelting, it was proposed [10] to roast the concentrate at 750 °C in a mixture with calcium hydroxide in an amount from 1.5 to 3 times greater than the stoichiometric amount required to bind arsenic into calcium arsenate. During subsequent smelting, the resulting calcium arsenate is converted into waste slag.

For relatively arsenic-rich materials (>5% As) that do not contain other subliming components, the main part of the arsenic (80-90%) can be distilled off in the form of sublimates containing up to 90-98% As<sub>2</sub>O<sub>3</sub> by firing at 550-700 °C in multiple-hearth, muffle or other furnaces. In order to prevent the formation of higher arsenic oxides and accelerate the distillation, up to 10% of fine coal and a sulfidizer (pyrite) are added to the batch [11]. Metallurgical sublimates and dusts are subjected to various types of smelting with concentration of valuable metals (lead, zinc and others) in the melt, and arsenic - in sublimates or in melts of

rough alloy, mattes, slags. Sodium hydroxide, soda, sodium sulfate, their mixtures are used as slag- and matte-forming fluxing components. The process is carried out in a controlled environment with the introduction of coke into the charge.

When smelting dusts using only sodium sulfate (22-30% of the dust mass), 75% of the arsenic passes into the matte-slag melt and 23% into the crude lead. 93% of the lead is concentrated in the lead melt. When using a mixture of sodium sulfate and soda (40-45% of the mixture in the charge), the arsenic is concentrated in the matte-slag melt, and with an excess of soda (about 60%), 75-80% of the arsenic passes into the crude lead and only 20% into the matte-slag melt. The extraction of lead into the melt, containing 9.3% As, was 93-94% of the content in the original dust. As a result, arsenic can be concentrated in either the crude lead or the matte-slag melt during the smelting process, if necessary. Black lead with a high arsenic content can be suitable for special alloys for various purposes (high-capacity batteries, bearings, etc.). Arsenic is leached from the stein-slag melt with water and then precipitated with lime.

Systematic research and technological developments on the removal of arsenic from metallurgical processes in the form of a non-toxic, storable product – sulfide – were carried out at the Zh. Abishev Chemical and Metallurgical Institute (Karaganda) [12-16]. A method was developed for obtaining arsenic sulfide sublimates from sulfide materials by roasting them at 650-700 °C in a mixture with pyrite in a ratio of arsenic to arsenic in the charge equal to (1.8-2):1, followed by melting the powdered sublimates and obtaining a compact alloy that is not oxidized in air and insoluble in water. Methods have also been proposed for converting arsenic dust into sulfide by sulfiding it with elemental sulfur at a temperature of 325-350 °C, followed by obtaining sublimates by roasting at 700-800 °C, as well as elemental sulfur at 325-350 °C and then leaching with a solution of sodium sulfide at a ratio of the latter to arsenic (3.4-3.5):1. The resulting arsenic sulfide is precipitated from the solution with sulfuric acid at pH = 2.

Work on the removal of arsenic into a poorly



soluble form during the processing of refractory gold-arsenic ores and concentrates deserves special attention. Until recently, about 75% of gold in Russia was mined from placers and 25% from ore raw materials. At the same time, in the near future, 75% of the predicted resources and 53% of the reserves of Russian gold are concentrated in ore raw materials. At the same time, gold mining in foreign countries has long been carried out mainly at

the expense of raw materials from primary deposits [17]. It follows that the development strategy of this sub-sector will be entirely determined by the processing of ore raw materials, a significant part of which is refractory, difficult to process arsenic-containing ores. The compositions of concentrates from some deposits in Russia and the CIS are given in Table 2.

**Table 2. Chemical composition of some gold-arsenic concentrates of Russia and the CIS, %**

Deposit	Au, g/t	Ag, g/t	As	Fe	S	C	Sb
Zodskoe	55,4	42,5	2,08	27,09	26,15	Not found	Not found
Kokpatasskoe	32,4	7,4	9,96	26,6	24,1	4,3-11,0	Not found
Bakyrchikskoe	10,8-34	15,4	5,7-9,6	10,0-13,8	10-17,8	7,6-19,2	Not found
Nezhdaninskoe	21-150	120-1300	4,8-5,6	14,7-19,2	15-19,5	1,8-7,7	0,1
Olimpiadinskoe	49-63,1	4	3,73	21,98	14,5-20,7	0,4-5,6	0,1
Majskoe	68,8	9,9	5,7	19,8	18,3	2,9	1,4
Kjuchusskoe	36,5	9,8	4,6	10,1	5,56	1,38	Not found
Zarmitan	35	239	16,4	30,17	29,47	0.15	Not found

This type of raw material, in which some of the gold, as studies have shown, is directly included in the crystal lattice of matrix minerals (arsenopyrite, pyrite), cannot be processed by traditional methods. Without preliminary opening of the minerals of the matrix base of concentrates, gold extraction is possible only in the range from 10-15 % to 60-70%, depending on the specific composition of the raw material [18]. At gold recovery plants, during the processing of such ore using the technology of converting arsenopyrite into tailings, all the bound gold goes into them, and when it is preserved in the concentrate, arsenic remains in it and enters the technological process. It pollutes the industrial products and must be removed from the process.

In order to open up the material for further gold extraction and to remove arsenic from the process, oxidative roasting is widely used using various types of furnace units and technologies: roasting in multi-hearth, rotary drum furnaces, roasting in a fluidized bed, and others.

Other methods of opening refractory ores and concentrates were also tested and mastered, where

oxidation of arsenopyrite and pyrite was carried out by acid, autoclave, bacterial oxidation, chlorinating roasting. The use of nitric acid as an oxidizer in acid opening of refractory gold-arsenic raw materials allows removing arsenic and iron in scorodite, which is poorly soluble in water. Carbon-containing arsenopyrite concentrates, rich in silver and gold, are proposed to be treated with sulfuric acid. In this case, arsenic and iron completely pass into solution, and precious metals remain in the silicate residue and are extracted as a free impurity (with a size of at least 50 µg) by cyanidation.

To intensify the process of acid opening and more complete extraction of precious metals from refractory sulfide-arsenide concentrates, it is proposed to carry out the process of hydrothermal decomposition in autoclaves ( $T = 120-225\text{ }^{\circ}\text{C}$ ,  $P > 3.5\text{ atm}$ ) [19, 20] using oxygen as an additional oxidizer. Sulfuric, nitric, hydrochloric acids, as well as chlorine-containing compounds, such as  $\text{CaCl}_2$ , are used as an acid agent.

Biological methods of decomposition and opening of sulphide and sulphoarsenide

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concentrates of heavy non-ferrous and precious metals belonging to the refractory group have been developed [3, 17]. This was preceded by many years of research on obtaining the necessary strains of bioculture and developing schemes and modes of the technology of bacterial opening of raw materials. The technology of bacterial opening has begun to be mastered in a number of gold mining countries: South Africa, the USA, Canada, Australia, and in Russia by the Polyus company at the Olimpiadinskoye and Nezhdaninskoye deposits.

A common disadvantage of all the above methods, as well as in non-ferrous metallurgy in general, is the unsolved environmental problem. During oxidative roasting, arsenic is distilled off in the form of trioxide (hazard class 1) with its subsequent conversion to calcium arsenate (hazard class 2). During acid, autoclave methods of opening and bioleaching, arsenic is converted into the form of calcium-iron salts of the scorodite type (hazard class 3). Thus, during bioleaching, the resulting sediments intended for storage in dumps contain from 8 to 23 % As in the form of complex salts related to the sulfoscorodite type. However, the possibility of interaction of these compounds with the environment and migration of arsenic in the hypergenesis zone with high dispersion of the dump material and changes in external storage conditions has been established. In order to prevent the environment from being contaminated with arsenic from drainage solutions, as well as from infiltration waters during floods and other natural disasters, it is necessary to carry out burial in special storage facilities or burial grounds, the construction of which requires significant expenditures and the alienation of large areas of land [21-23].

Therefore, work is being carried out [24] aimed at increasing the stability of arsenic sulfide compounds. Arsenic sulfides obtained during dearsenizing roasting are, in the main, a finely dispersed material with a developed surface, which

facilitates oxidation and leaching upon contact with the external environment. Methods have been developed for rendering harmless such sublimates by melting them into compact blocks or pressing them into briquettes with subsequent coating with sulfur or bitumen. These measures allow them to be stored in hazard class 3-4 in ordinary warehouses and abandoned mines.

**Conclusion.** Despite the large number of developments, none of the alternative technologies to oxidative roasting has surpassed it in terms of its level of use. For all these technologies, a number of key problems remain unresolved: the complexity of the equipment, machinery and process flow charts used; the toxicity of the resulting waste, the need for expensive disposal and constant environmental monitoring of the technical condition of temporary dumps and burial grounds and their impact on the environment.

The most acceptable technologies seem to be those in which arsenic is removed from the process at the preliminary stage of processing in the most stable, non-toxic form - arsenic sulfides, which exist in nature as orpiment and realgar minerals. Research conducted at the Zh. Abishev Chemical and Metallurgical Institute (Karaganda, Republic of Kazakhstan) and the Gidrosvetmet Institute (Novosibirsk, Russian Federation) on products from a number of arsenic-containing raw material deposits on the removal of arsenic by oxidizing-sulfidizing roasting in a shaft-type furnace showed the prospects of this technology. The removal of arsenic from the roasting products was 97-98.5 %, simultaneously with the production of a compact sulfide material with an arsenic content of 60-70% that does not require special disposal. In the cinder, which after roasting can be processed either by the traditional method or by smelting into a collector, the residual concentration of arsenic is less than 0.15-0.20 %.

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