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Pilot tests of processing technologies of process solutions of copper production by ozonation

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ABSTRACT

At the copper-smelting plant of JSC Almalyk MMC, technological solutions are formed containing valuable components in copper production. This article presents the pilot tests of the technology for processing technical solutions of copper production using ozonation. As a result of studies on the oxidation of impurities in wastewater, the process parameters were determined - ozone consumption, process duration, pH, and process temperature, which made it possible to purify and return wastewater to production also receive a sludge of metal compounds. Based on the results obtained, a technology for ozonation of copper production wastewater has been developed. The developed technology was recommended for implementation at JSC Almalyk MMC.
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1. Introduction

The pyrometallurgical method for copper production is associated with the formation of many slags, cakes, gases, and metal-containing waste solutions, the volume of which is much larger than the importance of the obtained metal itself. Metal-containing solutions are formed during the processing of process gases, copper sulfate production, and copper electrolysis. These solutions are sulfate and contain a certain amount of metals characterized by a complex variable composition and high toxicity. For such solutions, an integrated waste-free technology of its processing is economically profitable since it ensures the profitability of production and solves the environmental problems of the coexistence of production and the environment.

The problem of technological processing solutions of copper production on-site has been one of the most critical issues in heavy metals pyrometallurgy. The choice of a method for disinfecting wastewater of metallurgical output is carried out based on the flow rate and quality of treated wastewater, conditions of delivery, and reagents storage. Also, the choice of a disinfection method is determined by technical and economic indicators and environmental requirements [1].

To treat wastewater from metallurgical production used mechanical, chemical, physicochemical, and electrochemical methods. Most of them are energy-consuming, involved in execution, and are guided by imported equipment and scarce reagents. Therefore, in current economic conditions, priority should be given to effective cleaning methods, inexpensive local raw materials, and industrial waste.

2. Metodology

With the gradual decrease in copper ores being processed, copper concentrates have become more involved with higher impurity and gangue content. This trend has had a detrimental effect on smelters. They have to increase throughput to maintain copper metal production while increasing operating costs due to processing the increased amounts of secondary products (slag, acid) and stabilizing waste streams. This paper discusses impacts from the increased complexity of resources from mine to smelters, highlighting the need for an integrated processing approach to achieve sustainable and competitive multi-metal recovery [2].

In polymetallic concentrates, a more robust approach to increase metal recovery and multi-metal production has been to

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develop multi-metal recovery facilities. These facilities' main goals are to achieve maximum metal recoveries, optimum waste, and effluent management by exchanging metal flows between the different metallurgical circuits. This principle, shown in [2], is applied in integrated metallurgical plants, such as the Kazzinc Ust-Kamenogorsk Metallurgical Complex in Kazakhstan, the Boliden Rönnskär Smelter, or multi-integrated sites in Japan, Germany and Korea among others [2].

This article [4] discusses the importance of innovation for the mining industry and describes the mechanisms by which it is carried out. It includes a review of the drivers and actors involved and current trends. The digital transformation process that the industry is going through is analyzed and other relevant trends that are likely to shape the future's mining. Additionally, a case study is presented to illustrate the technical and economic implications of developing a disruptive innovation project. An investigation on the recovery of nickel from spent electroless plating solutions has been performed using the electrowinning method. For this aim, nickel in paid electroless plating solutions was separated as nickel hydroxide through the addition of caustic soda. Nickel hydroxide was dissolved entirely with sulfuric acid, and electrolysis was performed to elect nickel from nickel solutions. As a result, it was found that more than 99% of nickel in spent electroless plating solutions could be precipitated as nickel hydroxide above pH 10 with the addition of caustic soda. As far as the current efficiency in the nickel's electrowinning was concerned, it was decreased with an increase in the current density [3].

An attempt to recover copper from electroless plating wastewater has been made through evaporation, followed by the electrowinning method. From the determination of each element in electroless plating wastewater, the Cu's content was 582 mg/l, and a small amount of Fe was also contained in it. Moreover, the COD and TOC content, which resulted from the addition of Rochell salt, was found to be 9,560 and 13,100 mg/l, respectively. The content of formic acid generated by the oxidation of formaldehyde was determined to be 7.73%. As a result, current efficiency was decreased with an increase in current density, and therefore current density less than should be maintained to obtain current efficiency more than 80%. Fe's content in Cu obtained by electrowinning was found to be 0.021 and 0.01% at the concentration of sulfuric acid of 2 and 10 vol%, respectively [5–9].

The problem of preventing harmful effects from heavy metal ions (HMI) having toxicity at low concentrations in environmental objects has acquired a global scale, and cleaning from pollutants is currently relevant for the whole world. The problem of wastewater treatment from HMI exists in many industries. It is especially suitable now when the world community is on the brink of an environmental crisis. Penalties for discharging ITM into natural water resources are tightened [10–11].

In [12], a brief review of the use of sorbents from secondary raw materials for the extraction of heavy metal ions from wastewater was carried out. Comparative characteristics of cellulose-containing sorbents in comparison with activated carbon and cation-exchange resins are presented. The study of the sorption of heavy metals by natural materials from renewable raw materials of a polysaccharide nature is of great interest. This is because wastes and by-products of the agro-industrial complex are promising and economically profitable sorbents for removing HMI from aqueous solutions of various compositions, from industrial wastewater to natural waters and food systems.

Many industrial enterprises' activities often lead to environmental pollution by wastewater, which contains harmful substances in its composition, which, in small quantities, have a rather severe negative impact on human health and the state of the biosphere as a whole [13]. HMIs and their compounds are considered to be hazardous toxins in wastewater [14–15]. They are contained in the wastewater of galvanic workshops, machine-building and metalworking enterprises, ore and mine production, ferrous and nonferrous metallurgy, chemical and petrochemical industries, and other industries [16].

The harm of heavy metals to a living organism is due to their ability to bioaccumulate and concentrate when moving along the trophic chain, which leads to disruption of the functioning of organ systems. The difficulty in removing HMIs from the body is because they form strong bonds with proteins and other components of cellular structures [17]. In this regard, industrial wastewater treatment should be carried out until the almost complete removal of heavy metals. But using only traditional methods, this isn't easy to achieve.

Sorption and ion-exchange methods are one of the most widespread forms of wastewater treatment from HMI. In this regard, the need to obtain cheaper sorbents with improved physicochemical and operational characteristics is growing. Sorbents used in industry are developed based on activated carbons. Sorbents made from recycled materials are also of interest. Such materials can solve, in addition to water purification, another problem, namely, waste disposal [18].

The creation of sufficiently effective and environmentally safe sorbents and technologies is feasible due to the formation of new functional groups on polysaccharide polymers that firmly bind heavy metal ions [19]. The so-called biopolymer sorbents are obtained similarly. The raw materials used for sorption of HMI are represented by a complete set of various materials of plant origin - cones, sawdust, leaves, seeds, fruits and stems of different plants, the bark of deciduous and coniferous trees, oilcake and meal, walnut shells, husks, beet pulp, fruit peels, straw, herbaceous and aquatic plants, peat. Besides silt seaweed, bacterial biomass, yeast, mushrooms, etc. [20–22]. It should be noted here that an important direction in creating biopolymer sorbents is modifying the structure of natural cellulose-containing raw materials, which causes the immobilization of new sorption-active centers on the cellulose matrix, which are fragments of complex ones. This increases their selectivity, sorption capacity and decreases the sorption time [19].

Thus, studying the processes of sorption of heavy metals by natural materials from renewable raw materials of polysaccharide nature is of great interest. This is because wastes and by-products of the agro-industrial complex are promising and economically profitable sorbents for removing HMI from aqueous solutions of various compositions, from industrial wastewater to natural waters and food systems.

In [23], the possibility of creating a technological scheme for the purification of household and related wastewater with a minimum amount of excess sludge formed by using bioreactors with attached biomass is considered, and a decrease in the volume of wet sludge is achieved by intensifying the process of aerobic stabilization. Based on the production experiment's data and the experimental operation of the modernized aerobic stabilizer, aerobic stabilizers were designed and built for treatment facilities in the Moscow region with a capacity of 600 ... 10000 m3/day.

The socio-ecological aspect is due to the constant expansion of areas for storing sediments [24], pollution of soils and the aquatic environment with heavy metal ions, and deterioration of the sanitary and parasitological situation in the area of organized and unorganized sediment disposal [25–29].

The flotation process of metal ions is used to remove insoluble impurities from aqueous solutions, which spontaneously settle poorly [30–35]. The method allows for the collective recovery of metals, but separate recovery of each metal is required.

Acid-base properties, solubility, and chemical stability of ethyl 2-aryl(methyl)sulfonylamino-4,5,6,7-tetrahydro benzothiophene-3-carboxylates have been studied. Complexes of Cu(II), Co(II), and

Ni(II) with one of the ligands have been prepared and isolated; their solubility products were determined [36–37].

The paper [38–40] studies sulfonyl aminothiophene derivatives as potential collecting agents in ion flotation of non-ferrous metals. The study determines optimal flotation conditions of Cu(II), Co(II), Ni(II), Zn(II), and Cd(II): the range of pH values, process time, and amount of reagent. It demonstrates the effectiveness of compounds as collecting agents for non-ferrous metals in standardized test solutions using ion flotation.

The new approach to reagents for flotation extraction of non-ferrous metals cations based on quantum-chemical calculations and the Person principle has been developed. The possibility of using the reactivity index of organic compounds obtained as a result of quantum-chemical calculations as a criterion for estimating their flotation activity has been substantiated. The new class of prospecting collector reagents as a compound ether of terephthalic acid has been taught [41–43].

Purification of process wastewater from copper production from organic contaminants and heavy non-ferrous metals is currently an urgent task. This issue is acute in regions where the industry is developing, and technological and human-made water resources pollution is increasing. The applied methods do not meet the requirement; therefore, removing heavy metal ions and organic compounds can be achieved by ozonating technological wastewater.

3. Objects and method of research

To carry out the research, waste solutions of copper production were selected: acid effluents from the vitriol shop and a washing solution (after washing process gases) of the sulfuric acid shop of the Copper Smelter of JSC Almalyk MMC. Until now, these solutions have not been disposed of due to the lack of adequate technology for the extraction of metals from solutions with high sulfuric acid content. Losses of molybdenum with waste mother liquor reach 4–8, nickel 140–150, copper 40–45, zinc 30–35 t/year.

From the results of chemical analysis (acidic effluents, g/dm3: H2SO4 – 4, Cu – 0.035, Zn – 0.001, Fe – 0.02, Ni – 12.5, Mo – 0.002, washing solution, g/dm^3: H_2SO_4 – 48 , Cu – 0.026, Zn – 0.012, Fe – 0.019, Ni – 0.002, Mo – 0.0012) it can be seen that copper, zinc, iron, nickel and molybdenum are of industrial interest for the extraction of metals. The complexity of processing the wash solution is due to the high acid content.

The technical solutions to be purified, passing through the accelerator (injector), enter a certain amount of gas-air ozone into the water, creating a rarefaction in it. Forced stirring in the accelerator atomizes ozone into small bubbles ranging in size from hundreds of micrometers to 1 mm with a large contact surface, thereby increasing the rate of O3 dissolution in water. The scheme of the installation and the principle of operation of the process of ozonization of copper production technological solutions is shown in Fig. 1.

4. Results

Reactions of ozone with components in wastewater are poorly studied; based on this, a necessary stage in developing wastewater ozonation technology is to conduct preliminary technological studies. The ozone formation process consists of several locations and more than 50 reactions associated with the formation of ozone and its decomposition, without which it is impossible to obtain ozone. Based on the above responses, the ozonation process can remove several heavy metals from wastewater: sulfates, metal carbonates, etc [44].

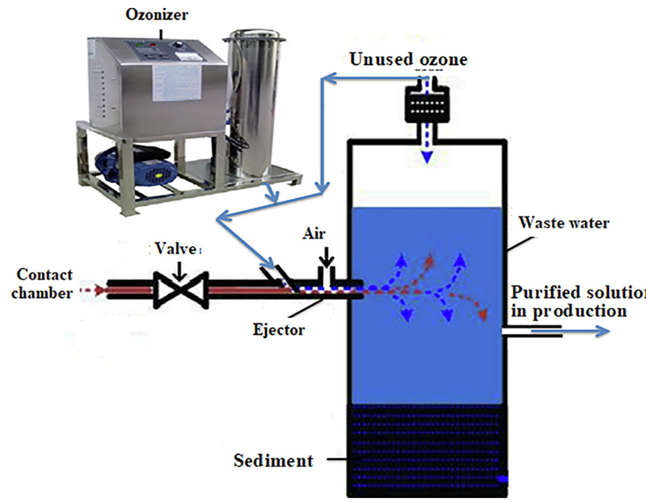

Fig. 1. Scheme of the ozonation plant.

The development and use of ozone technologies are based on the high reactivity of ozone and its ability to decompose without forming by-products that quickly pollute the environment. The only drawback is the relatively high cost of ozone. However, with the development of new energy-saving ozonizers and the tightening of environmental requirements about the traditionally used chemical reagents, ozonation methods have become competitive [45].

The ozone concentration in water depends on the following processes: a constant supply of ozone to the volume of purified water, consumption for the oxidation reaction, and self-decay of ozone. As a result of these processes, the ozone concentration in water changes [47–50]. Experiments to study the effect of the medium's initial pH on the intensity of treatment of process wastewater have shown that the oxidation of impurities, both ozone-air mixtures and air oxygen, proceeds intensively for the first time for 15 min in almost all cases. Then the oxidation rate decreases. Experiments used 1–1.5 g of ozone per 10 L of process solution.

The maximum wastewater treatment in an acidic environment can be explained by a chemical process accompanied by the enlargement of ion molecules (reactions of hydroxyl and peroxide radicals with a high oxidation potential) and the formation of a sludge, which is confirmed by the amount of the formed delicate solid phase [45].

The intensity of wastewater oxidation increases with increasing temperature (Fig. 2), but with a duration of 70–80 min, the degree of wastewater treatment, regardless of the temperature rise, is the same. In ten to twenty minutes, ozone interacts with easily oxidizable substances in wastewater. However, as shown in Fig. 3, the impurities' content decreases markedly after an hour of treatment, then stabilizes. It can be assumed that in the process of ozonation, not only surface oxidation of metals occurs, but the formation of peroxides, which, by diffusion, penetrate deep into the particles, cause secondary oxidative processes [45].

The UV (Fig. 3) spectra analysis showed that during ozonation in the solution under study, the accumulation of oxygen-containing functional groups (OH–, C=O, S=O) occurs (Fig. 2). As a result of ozone treatment in the component composition of wastewater, the total compounds' ratio changes. The metals' content, which makes up the bulk of metal-containing components, decreases with ozonation to 99%. The amount of co-deposition of two or more different metal ions at specific pH results in better results than the deposition of each metal separately. This is due to the formation of mixed sediments in the sediment and the

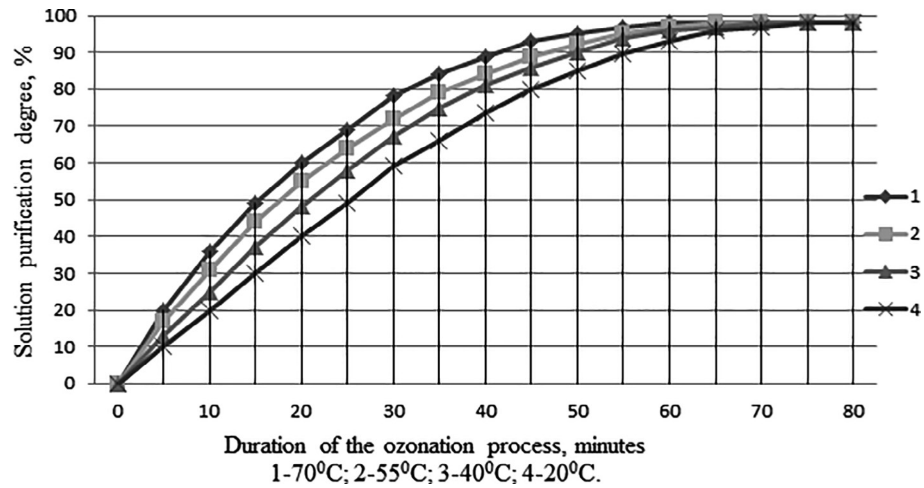

Fig. 2. Dependence of the change in the degree of purification on the ozonation time and temperature.

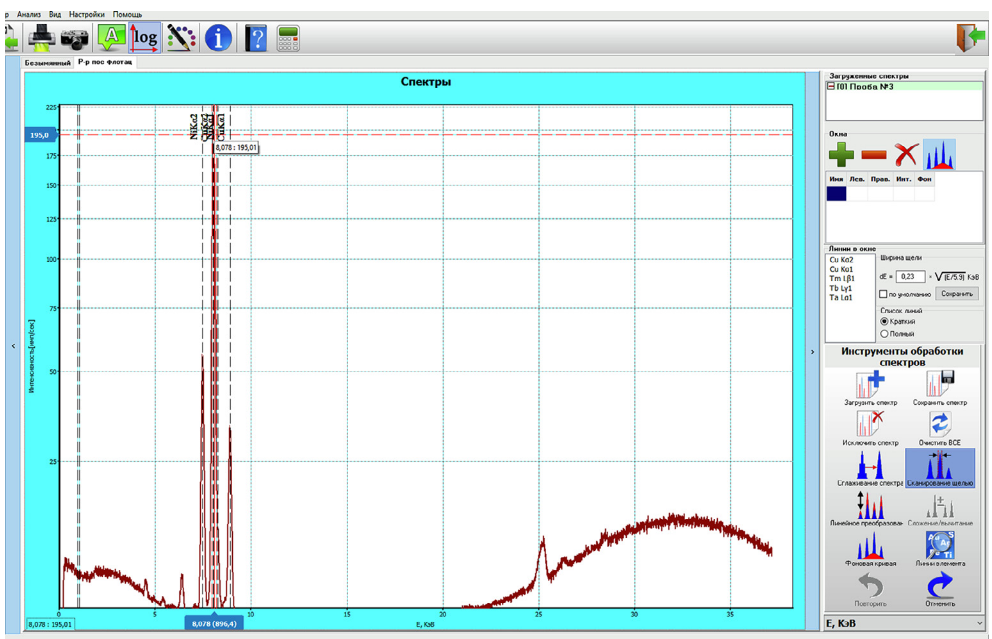

Fig. 3. Spectra of qualitative analysis of the solution.

absorption of metal ions on the solid phase's surface. Hence, it becomes necessary to control water quality after ozonation for several chemical and sanitary-hygienic indicators [45].

During the experiments, the ozone-air mixture was fed into the installation at a rate of 2.5 g/l. The initial pH values of the medium are within 2–5. The temperature of the technological solutions was kept continuously at 20 °C. The change in the concentration of metal ions depending on the pH of the process solution medium is presented in the table. 1. The experimental studies show that the maximum extraction of metals into the sediment is observed at the beginning of the process. The degree of purification of metals is up to 99% [45].

Thus, ozonation can be used as an effective method for neutralizing wastewater from metallurgical production. The resulting sediment after the ozonation process is subjected to chemical analysis and, according to the sediment's composition, is sent to the extraction of metals according to the traditional scheme of JSC Almalyk MMC. Treated water is used for technological needs.

A schematic flow diagram of semi-industrial tests for the purification of wastewater (wastewater) of copper production is shown in Fig. 4 [44–50]. A bubble column with a height of 1 m was used as a reactor (installation). The reactor is equipped with a fine-pore filter with a diameter of 70 ... 100 μm for dispersing the ozone-air mixture, removing waste gas, and periodically sampling purified water. The reactor operated in a flow-through mode, non-flowing in the liquid phase.

The efficiency of the process largely depends on the pH of the solution being treated. In the entire considered range of pH values of the resolution, the precipitation of metals occurs. Deposition as the whole proceeds in similar environments corresponding to metals in the form of hydroxides. It was found that for 1 h of treatment, the concentration of metals (Fe, Zn, Cu) decreased to a level of < 0.01 mg/l, which is an order of magnitude lower than the MPC of metals in water and also reduces environmental damage to the environment.

Table 1
The results of purification of acidic effluents and washing solutions with ozone.

Content in washing solutions, mg/dm^3		solution pH	Content in the solution after ion flotation of washing solutions, mg/dm^3					Wastewater treatment degree, %				
			Mo	Cu	Zn	Fe^{+2}	Fe^{+3}	Mo	Cu	Zn	Fe^{+2}	Fe^{+3}
Mo	0,27	1	0,11	2,2	17,2	1,8	0,06	59,26	88,42	64,9	28	96,25
Cu	19	2	0,09	0,76	12,2	1,6	0,04	66,67	96	75,1	36	97,5
Zn	49	3	0,02	0,2	3	0,9	0,03	92,59	98,95	93,88	64	98,13
Fe^{+2}	2,5	4	0,01	0,2	0,5	0,2	0,03	96,3	98,95	98,98	92	98,13
Fe^{+3}	1,6	5	0,01	0,2	0,5	0,1	0,03	96,3	98,95	98,98	96	98,13

*Experimental conditions: ozone concentration 6 mg/l, ozone consumption 0.1–0.15 g/l, solution temperature 200C, cleaning time 60 min.

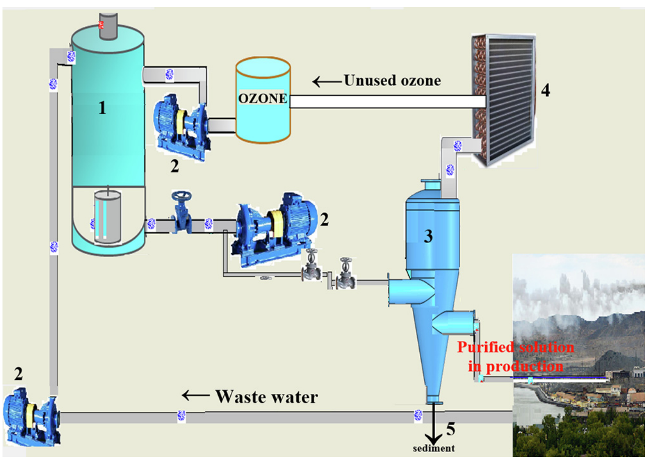

Fig. 4. Basic technological scheme of the ozone wastewater treatment plant. 1-mixer; 2-pump; 3-bubble absorber; 4-apparatus for collecting waste gases and unused ozone; 5-sediment is sent for metal extraction.

5. Conclusion

Based on the research results, the following conclusions can be drawn:

- as a result of the research, the degree of purification of waste solutions of copper production was determined, which depends on the pH value of the resolution, while almost complete precipitation of metal ions in the form of a precipitate is achieved;
- ozonation can be used to remove salts of several heavy metals from wastewater: sulfates, metal carbonates, etc.
- the developed technology is recommended for implementation in JSC Almalyk MMC.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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