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Development of dephosphorization technology for iron ores with high phosphorus content

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Abstract. The main methods of removing phosphorus and sulfur from iron ores, as well as magnetite and hematite concentrates, were analyzed. For effective dephosphorization of hematite concentrates, it is necessary to use direct cationic flotation of apatite, with the help of which it is possible to remove more than 74% of phosphorus from the concentrate. As a result of the analysis of the conducted research and the synthesis of the obtained scientific results, a technology was developed for removing phosphorus from iron concentrates. This technology allows for the reduction of the phosphorus content from 0.14% to 0.04%. Chemical methods – alkaline or acid leaching with separation of the leached concentrate – are the most common ways to dephosphorylate metal-containing ores. Research on chemical methods of cleaning ores and iron ore concentrates is carried out in the following directions: leaching with alkali solutions; acid leaching at different temperatures; and leaching of impurities in autoclaves. The conditions of leaching of poor hydrogethite high-phosphorus concentrates at high and low temperatures were investigated. The proposed technology of leaching samples of poor hydrogethite high-phosphorus concentrates at high and low temperatures allows us to claim that the parameters of the firing process allow the recrystallization of iron and therefore contribute to the access of mineral acid to particles containing phosphorus.

1. Introduction

Currently, Ukraine is the seventh largest producer of iron ore raw materials in the world with a state balance of ores in the amount of about 30 billion tons, which are concentrated in 52 deposits, 24 of which are currently being developed [1]. The analysis of the practical experience of beneficiation of magnetite ores at mining and beneficiation factories in Ukraine (more than 70 years) made it possible to establish a clear relationship between the beneficiation indicators of non-oxidized quartzites and their composition by comparing the results of beneficiation of quartzites according to their material composition and structural [2].

Nowadays, 98.5% of iron ore products are used for the needs of ferrous metallurgy, so the requirements for the quality of the mineral-raw material base come from the requirements for steel, and cast iron, as well as from the technological features of steelmaking production [3]. Since the blast furnace is the main technological process in Ukraine and Eastern Europe, the requirements for the quality of iron ore products are primarily focused on it. It should be noted that the requirements for the quality of iron ore products exported to Western Europe are higher since the metallurgical production of industrialized countries is focused primarily on



the direct recovery of iron, bypassing the blast furnace process. Therefore, the main task is to provide the iron mining industry with high-quality mineral raw materials suitable for high-quality metallurgy [4, 5]. This is possible due to the improvement of the technology in the development of operational objects [6, 7].

Phosphorus is a harmful impurity in metal, because, being in it, it gives it brittleness and greatly increases the tendency to brittle fracture. Phosphorus also increases the cold-brittleness threshold, that is, the temperature at which the impact viscosity drops sharply. Since phosphorus cannot be easily removed from the flux or melt, the maximum allowable content of phosphorus in steel is limited to a rather low limit, which, taking into account the requirements for the metal and the possibilities of dephosphorization, is up to 0.02-0.04%.

A large amount of damage in the smelting of iron and ferromanganese is caused by an excess of silica. When smelting iron, increasing the silica content by 1% increases the consumption of coke by approximately 3%, fluxes by 4%, and reduces the productivity of the blast furnace by 2-7%. When smelting ferromanganese, silica leads to the formation of strong manganese silicates in the lower part of the furnace, which turns into slag, which, in turn, sharply reduces the extraction of manganese.

In the recovery process of either the ore or the production concentrate, the empty rock contained in the metal pellets is transformed into the final product. In addition, the presence of empty rock in metalized raw materials causes a large slag formation in the steel smelting process. Under these conditions, the consumption of electricity and fluxes increases significantly, and the duration of melting increases. In addition, slag corrodes the lining of furnaces. Therefore, in the metalized raw materials intended for steel smelting, they strive to reduce the content of empty rock as much as possible and to bring the content of acidic rocks (SiO_2) to 4%.

Therefore, the creation of a fairly simple and effective technology for the preparation of magnetite and hematite concentrate, which ensures a reduction in the content of harmful impurities, is a very urgent practical task.

2. Literature review

Depending on the composition of iron ores, technologies for their enrichment and further preparation for metallurgical redistribution are being developed. Preparation of iron ore concentrates for metallurgical redistribution includes calcination, cooling, leaching with mineral acid, separation of the liquid phase from the solid phase, and so on. All this leads to an improvement in the quality of iron ore concentrates, because at the same time unwanted impurities contained in the concentrates, primarily phosphorus, sulfur, and arsenic, are eliminated. Metallurgical methods of phosphorus removal from concentrates are mainly used.

So, the firing process is carried out at a temperature of 800-1000°C. At the same time, the structure of hydroxide (FeOOH) is destroyed, and the recrystallization of iron and displacement of phosphorus from grains at the interface of phases (crystals) occurs.

At a temperature below 800°C, the process proceeds with insufficient completeness. At temperatures above 1000°C, particles sinter into agglomerates and acid access to phosphorus-containing mineral particles deteriorates, as a result of which the degree of phosphorus extraction decreases.

The firing time within 1 hour ensures a high extraction of phosphorus into the solution during further leaching. An increase in the firing time, of more than one hour, worsens the technical and economic indicators of the entire dephosphorization process. A process combining oxidative calcination-gas reduction and magnetic separation has been developed for iron dephosphorylation at a lower reduction temperature [8].

Studies based on the possibility of redistribution of phosphorus from ore components to slag-forming ones in the process of agglomerate firing have been conducted [8].

Disadvantages of metallurgical methods of dephosphorization include their uneconomical nature, large losses of iron (up to 20%), and environmental problems.

The problem of developing a technology for removing phosphorus by non-metallurgical means and obtaining a phosphorus-conditioned concentrate from these ores is of great practical interest and is of great importance for many countries of the world, such as Australia, Spain, Colombia, the USA, Sweden, France, and others. Part of phosphorus is part of the cement mass, which binds finely dispersed magnetite grains. Therefore, it is impossible to remove it by mechanical means to a mass fraction that meets the requirements of metallurgical redistribution. Fairly limited removal of phosphorus is achieved by grinding and magnetic separation.

The focus of current research and development in the field of flotation is cationic and anionic flotation methods from the point of view of reagent regimes, pulp chemical composition, and particle size, as well as the efficiency of removing major impurities such as silica, alumina, phosphorus, and sulfur [9]. Anionic reagents are usually used to remove phosphorus by flotation methods. Thus, the Aqua Nobel company developed a cationic reagent based on the Lilaflo reagent, the use of which during flotation at a flow rate of about 70 g/t made it possible to remove 90-95% of the phosphorus contained in this concentrate from the magnetite concentrate. At the same time, the loss of iron was about 2%.

A method of direct flotation of phosphate minerals from magnetite concentrates has been developed, in which the pulp is treated with an alkaline agent to pH from 7 to 11 and a depressor of carbonate and silicate minerals. A collector type of succinic acid derivatives is introduced in the amount from 20 to 2000 g/t of the initial product and a foaming agent [10].

Periodic flotation tests of an effective combination of reagents to reduce the content of silica and phosphorus in iron concentrate were carried out. The study aimed to study the influence of dosage, sodium silicate module, and the addition of Ca⁺⁺ ions on the selectivity of separation of phosphorous and siliceous vein minerals from iron oxides [11].

The disadvantage of flotation methods for cleaning iron ores from phosphorus is the impossibility of their removal from finely dispersed growths. Therefore, in this case, hydrometallurgical methods are used.

Consider the use of hydrometallurgical methods for removing phosphorus from iron ores.

The method of pre-treatment of ore to reduce the content of sulfur and phosphorus in it by washing the ore (essentially iron oxides) with aqueous solutions of soda with a gradual increase in temperature, includes mixing an inorganic base with oxide iron ore, which mainly contains iron oxides at a ratio of approximately 0.1÷1.5 – bases to the weight of iron ore; heating the mixture to 300°C and rinsing the mixture with hot water.

The disadvantages of this method include the fact that the degree of reduction of phosphorus impurities in the ore remains low and, therefore, does not allow obtaining conditioned iron ore concentrates.

Recently, the most common methods of dephosphorylation of metal-containing ores are chemical methods – alkaline or acid leaching with separation of the leached concentrate [12]. In the case of hematite ores, heat treatment is necessary before alkaline or acid leaching to make phosphorus available for chemical separation [9, 13]. The amount of phosphorus removed increases with heating temperature up to 1300°C. Silica, alumina and sulfur are usually removed along with phosphorus. Chemical methods of cleaning ores and iron ore concentrates are used in different versions. Research is mainly carried out in the following areas:

- leaching with alkali solutions;
- acid leaching at different temperatures;
- leaching of impurities in autoclaves.

When choosing a method of chemical enrichment, it is necessary to be guided by the availability of reagents, profitability, and a sufficient degree of purification.

Removal of silicon compounds is possible with alkali solutions. Acids are used to remove calcium and magnesium.

There is a known method of removing phosphorus from Lorraine iron ore, which contains 30% Fe, 20% SiO₂, 7% Al₂O₃ and 1.7% P₂O₅, which involves treating the ore with a 40-50% alkali solution at a temperature of 125-140°C, duration from 30 min to 3 hours and the amount of solids in the pulp from 50 to 200 g/l. At the same time, up to 60-80% of phosphorus, silica, and other ore minerals were extracted into the solution, while more than 93-95% of iron was extracted into the concentrate.

In this method, the most common is sodium hydroxide. But when it is used, there are non-technological solutions that are poorly defended and filtered. In addition, the discovered optimal leaching regime has several serious drawbacks: a complex scheme of alkali regeneration and significant water consumption for its washing.

A complex method of beneficiation of iron ore using NaOH and magnetic separation has been tested in the USA. It includes the following stages:

- (i) mixing ore or ordinary concentrate with a small volume of diluted NaOH solution;
- (ii) treatment of the mixture with superheated steam at a temperature of 260-400°C.

Such processing leads to the fact that the bonds between SiO₂ crystals, as well as between SiO₂ and iron minerals are weakened. There is other evidence that chemical treatment (alkaline or acid) of iron ore concentrates containing quartz and iron silicates alters the surface properties of the minerals, which improves flotation and magnetic re-separation performance. When ordinary hematite concentrates obtained from ores of the Kryvyorizhye deposit were treated with NaOH solution, the silicon content decreased from 8.62 to 0.76%. Phosphorus, sulfur and arsenic were leached to hundredths of a percent. The content of calcium and magnesium increases after alkaline treatment. At the same time, sulfur goes into solution in the form of sulfates according to the equation: $\text{FeS}_2 + 16\text{NaOH} + 15\text{O}_2 \rightarrow 4\text{Fe}(\text{OH})_3 + 8\text{Na}_2\text{SO}_4 + 2\text{H}_2\text{O}$.

Acid treatment of iron ores is carried out in order to separate impurities of phosphorus, arsenic, sulfur, calcium, and magnesium. According to research, any mineral acid at a temperature of 70°C removes arsenic from ores from the above-mentioned impurities, such acids as sulfate H₂SO₄ and chlorine HCl are used.

Many studies have been conducted on the purification of iron ores with nitric acid. Most of the works were carried out by Japanese researchers. A method of separating impurities from iron ore with concentrated nitric acid was patented in Japan, which ensured an increase in the iron content from 55 to 65% due to the complete leaching of phosphorus and arsenic and partially – calcium, magnesium, manganese, and sulfur.

In further studies, the concentration of nitric acid was reduced without reducing the leaching efficiency by increasing the temperature adding sulfuric acid to nitric acid, or conducting the leaching process at 200°C in an autoclave.

Acids, as you know, interact with iron compounds, which leads to additional consumption of acids and a decrease in the yield of the concentrate. But in all patents, these disadvantages of acid enrichment are ignored. It should also be noted that acid regeneration is impossible. After leaching, the alkali can be regenerated by adding lime.

The method of oxidative leaching in an acidic environment of complex iron ores consists of processing the material under such conditions as to promote the oxidation of iron and sulfur, to oxidize sulfur sulfide, at least partially, into sulfate [14,15]. Oxidized iron and impurities go into the solution in the leaching process, which is carried out over a certain period until more than 80% of the sulfide contained in the material is oxidized. At the same time, leaching sediment is formed, which is suitable for the thermometallurgical recovery of iron. This method gives a positive result for sulfur removal but does not provide dephosphorization of iron ore.

The method of leaching phosphorus with sulfuric acid at a temperature of 95-100°C is also known. The consumption of sulfuric acid to dissolve phosphorus contained in concentrates is 30 kg per 1 kg of leached phosphorus, i.e. much higher than stoichiometry. To avoid re-precipitation of ferric sulfate, it is necessary to operate with pH values in the range of 1.6-1.4, depending on the content of solid particles in the pulp. The ratio of solid to liquid (S÷L) should be 1÷2-1÷3, and the total leaching time should be 50 minutes. Further processing of the concentrate is carried out by the method of pelleting and firing of the obtained pellets. At the same time, the content of sulfur decreases by 95%, and phosphorus – by approximately 80% [15].

The disadvantage of this method is the complexity of the technological design of the leaching process, the high temperature of the process, and the large losses of iron with solutions.

Hydrochloric acid can also be used as a mineral acid during leaching. The essence of the method is that iron ore is first agglomerated, and then leached using hydrochloric acid or gaseous hydrogen chloride. The leaching temperature is 90-105°C.

The method of selective acid leaching was used to remove phosphorus from iron ores with a high phosphorus content. Hydroxyapatite in iron ores with a high phosphorus content was converted to soluble phosphate in the process of HCl leaching. The effect of reaction time, particle size, concentration of hydrochloric acid, reaction temperature, liquid-solid ratio, and stirring power on the degree of dephosphorylation was studied [16].

The disadvantage of this method is that it uses volatile hydrochloric acid, which is very harmful to service personnel and leads to equipment corrosion due to the presence of hydrogen chloride.

It should be noted that chemical leaching is successful only if it is preceded by heat treatment, which causes recrystallization of iron minerals in L-Fe₂O₃ and concentrated phosphorus between hematite grains. Heat treatment consists of burning the concentrate at a temperature of 500-600°C for 1-1.5 hours, and for leaching, sulfuric acid is used in an amount of at least 110-150% of the stoichiometric about phosphorus, at a temperature of 60-80°C, the ratio S÷L = 1÷3-1÷5. The leaching time is 2-3 hours.

A known method of cleaning iron ore from arsenic and phosphorus, in which the ore, crushed to 0.05-0.50 mm, was treated with a 0.5-2% solution of sulfuric acid at a high ratio of liquid to solid phases for 10-20 hours with subsequent ion exchange removal of impurities from the solution.

The disadvantages of this method are the long duration of the process (25 hours) and a significant amount of liquid phase, which requires a large volume of equipment.

The speed of all physicochemical heterogeneous processes increases with increasing temperature. When the leaching of impurities is carried out in autoclaves. At the same time, alkali solutions with concentrations of 40-50% are used in autoclaves at a temperature of 124-140°C, or sulfuric acid with a concentration of 60-70% at 95-100°C. The use of autoclaves allows you to maintain the temperature of the process above the boiling point of the solution. This leads to more efficient leaching.

3. Research objectives and approach

The primary objective of this research was to develop effective technologies for dephosphorization of various iron-containing concentrates, including magnetite, hematite, and hydrogethite, to reduce phosphorus content to levels meeting global quality requirements for iron ore products.

Specific aims of the study included:

1. Characterize the mineralogical composition and phosphorus distribution in magnetite-hematite ore samples and concentrates.
2. Evaluate desliming as a method to reduce phosphorus in magnetite concentrates.

3. Investigate direct flotation techniques, both anionic and cationic, for phosphorus removal from hematite concentrates.
4. Examine high and low temperature acid leaching processes for dephosphorization of hydrogethite concentrates.
5. Develop an integrated dephosphorization technology applicable to different types of iron ore concentrates.

The research approach involved detailed mineralogical analysis, laboratory-scale beneficiation and flotation experiments, and leaching tests under various conditions. Multiple dephosphorization techniques were evaluated to determine the most effective methods for different ore types. The ultimate goal was to achieve phosphorus reduction to below 0.05% in the final iron ore products, meeting stringent international quality standards.

4. Results

At the first stage of the research, a sample of magnetite-hematite ore with a mass fraction of iron of 53.8% was received. As a result of magnetic separation, two concentrates were obtained: magnetite (yield 47.7%, Fe_{total} mass fraction 69.509%) and hematite (yield 15.27%, Fe_{total} mass fraction 60.48%). The yield of tails was 37.03%. Iron losses by magnetic technology amounted to 21.2%. As you can see, the concentrates meet the requirements of the metallurgical industry in terms of the mass fraction of iron. However, it should be noted that they have a high mass fraction of phosphorus – 0.071 and 0.343%, respectively, in magnetite and hematite concentrates. Therefore, research on dephosphorylation was carried out precisely on samples of these concentrates. Note that a larger mass fraction of phosphorus is characteristic of hematite concentrate, because it has a smaller size compared to magnetite.

As a result of the study of the mineralogical composition of the concentrates, it was established that calcite is present in all the studied products – a violent reaction with acid. Non-ore minerals are observed in the form of both free fragments and in the form of inclusions with ore minerals. A characteristic feature of the studied magnetite and hematite concentrate is also a sharp dispersion of the dimensions of the fragments. The smallest fragments are mainly represented by iron hydroxides, calcite, and apatite. Apatite is represented by elongated as well as short-prismatic individuals. It most often spatially tends to calcite, but it is emphasized in the form of small inclusions in magnetite. The size varies widely from units of micrometers to tenths of a millimeter (figure 1).

On the basis of the analysis of the results of the study of the material composition, chemical and mineralogical properties of magnetite and hematite concentrates and the properties of apatite, we came to the conclusion about the feasibility of descaling magnetite concentrates and direct flotation of apatite from hematite concentrates.

Descaling was carried out on a sample of magnetite concentrate. The purpose of desliming was to reduce the mass fraction of phosphorus in the sample due to the removal of the minus 0.02 mm grade, where finely divided apatite is mainly concentrated. The results of magnetite concentrate de-slagging are shown in figure 2.

The obtained data confirm the expediency of the de-sludging operation in the technological scheme of beneficiation of rich hematite-magnetite ores. Thus, it was possible to reduce the mass fraction of phosphorus in the concentrate by 2.37 times – from 0.071 to 0.03%, which meets international standards for the quality of magnetite concentrate. which meets international standards for the quality of magnetite concentrate [17]. As a result of the descaling of the magnetite concentrate, the mass fraction of total iron in the concentrate also increased by 0.01%.

In the second stage of research, studies on direct anionic flotation of apatite were conducted on a sample of hematite concentrate. Distilled tall oil soap (DTOS) was used as an anionic

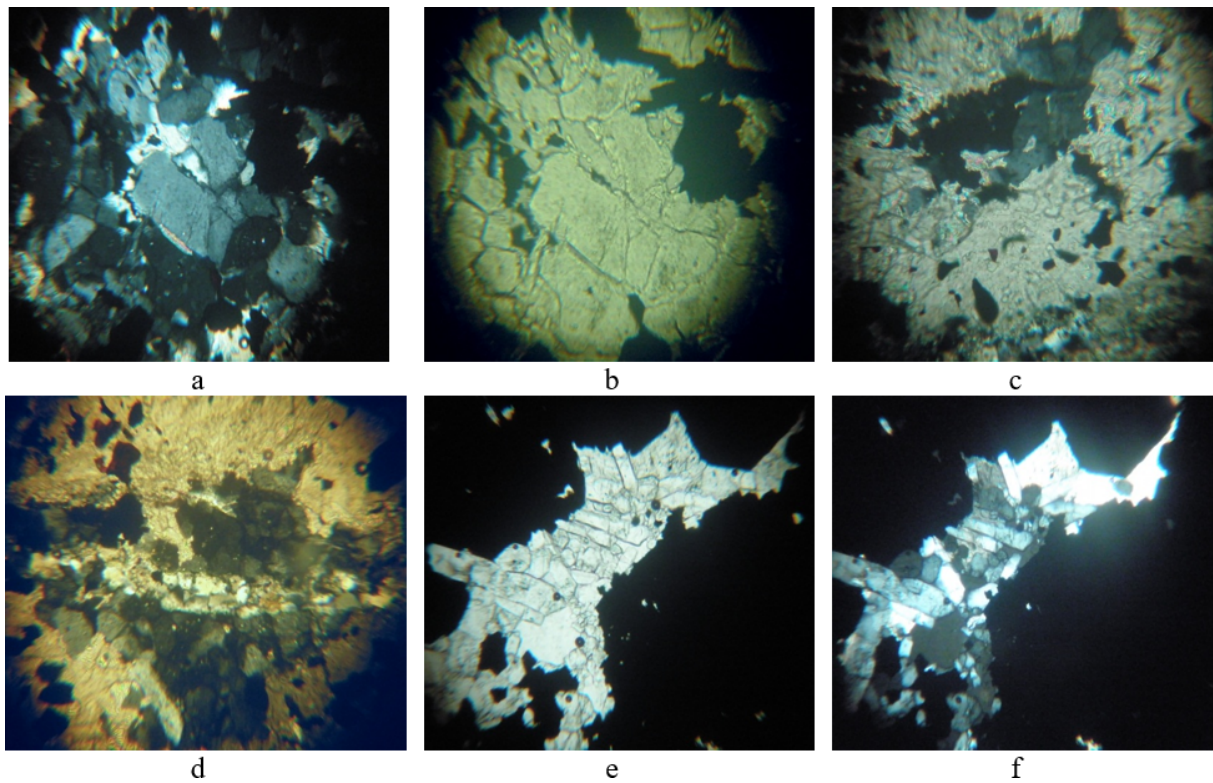


Figure 1. Iron ore raw materials in the light: a, b – nest-shaped aggregate of apatite; c – apatite in calcite mass; d – horizontally oriented quartz vein, apatite-calcite aggregate; e, f – apatite aggregate.

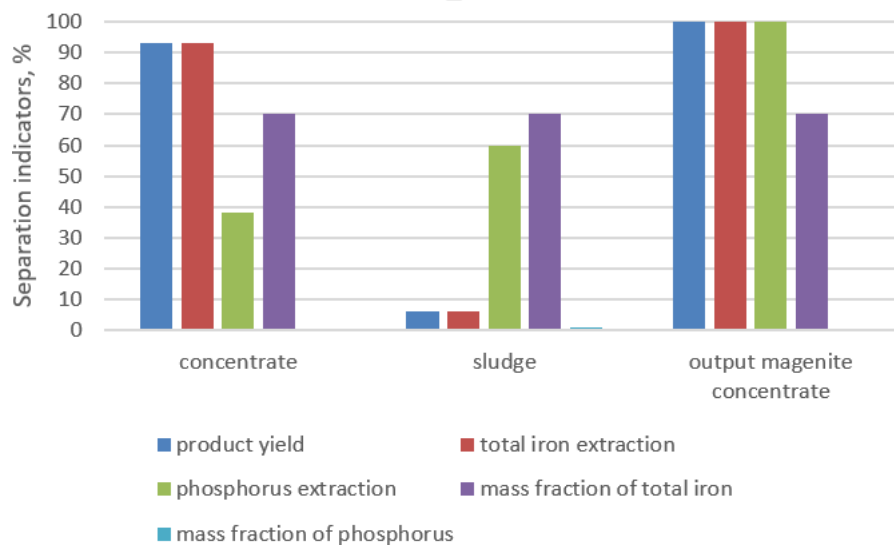


Figure 2. Indicators of magnetite concentrate de-sludging.

oxyhydroxyl collector during flotation studies. Saponified tallow oil is usually used in practice. DTOS is more selective compared to crude tall oil soap (CTOS). The main part of DTOS is salts (usually sodium) of inorganic fatty acids (oleic, linoleic, and linolenic), and the content

of resin acids in it is no more than 10-15%. The increased content of resin acids (up to 30% or more) in CTOS makes it less suitable for obtaining high-quality concentrates due to the deterioration of its collector properties and increased foaming. The choice of such a collector was made, mainly, only empirically and based on the availability and economy of the reagent.

To establish the regularities of the flotation process, several experiments were conducted using the Latin square method, where the variables were the consumption of the reagents – the collector, the depressor, the medium regulator, and the contact time with the reagents. The best indicators of dephosphorylation (according to the mass fraction of phosphorus in the concentrate) were obtained at the consumption of reagents (g/t): DTOS – 500 g/t, starch – 2500 g/t, soda – 1000 g/t.

It should be noted that during direct anionic flotation, a large part of hematite aggregates passes into the foam products, which leads to large losses of hematite with a foam product of 75-85%, which indicates the negative results of the conducted research.

Since the desired results were not achieved with direct anionic flotation of apatite, further studies were conducted on direct cationic flotation of apatite. In the study of direct cationic flotation of apatite, amines were used as collector reagents. Lilaflot reagent was used for flotation tests. These reagents are primary amines based on higher fatty acids with 17-21 carbon atoms in the radical. Lilaflot reagent is a mixture consisting of alkyloxypropylamine (60-80%) and alkyloxypropylamine acetate (20-40%). According to the parameters of high toxicity, the product can be classified as a moderately dangerous substance (hazard class 3). It should be noted that it decomposes completely into safe chemical compounds in the air within 24 hours

The use of cationic flotation is because, in comparison with anionic flotation, it does not require softening of water and large consumption of reagent. In addition, the duration of cationic flotation is 2-4 times shorter than that of anionic flotation.

The mechanism of action of amines consists in the hydrophobization of minerals during ion fixation, i.e. chemical interaction of ions and molecules of the collector, as well as their physical adsorption on the surface of the mineral, takes place. The reasons for the adsorption of a surface-active collector ion can be Coulomb attraction of the ion by the electrostatic field of the mineral surface, polarization of the adsorbent by the ion, electrostatic polarization of the ion by the surface field, and nonpolar Van der Waals forces. In turn, the adsorption of amine molecules, the polar group of which has a permanent dipole, depends on the possibility of dipole and dispersion interaction forces, the formation of a hydrogen bond.

A series of tests were conducted in laboratory conditions, which resulted in positive results: it was possible to reduce the mass fraction of phosphorus in the hematite concentrate by 2.87 times from 0.343 to 0.12%. The mass fraction of total iron in the hematite concentrate was 62.88%, which is 2.4% more than in the rough hematite concentrate.

In the third stage, the conditions of leaching of poor hydrogethite high phosphorus concentrate at high and low temperatures were investigated.

High concentration sulfuric acid was used in the research. The firing temperature was maintained at 800°C because at lower temperatures the process of destruction of the mineral structure is not carried out completely.

At a temperature of more than 1000°C, sintering of particles into agglomerates occurs, and access of the acid to the particles of the mineral, which contains phosphorus, deteriorates. As a result, the degree of phosphorus extraction decreases. Therefore, the optimal temperature is 900°C. This is the temperature at which the destruction of the structure of hydrogethite (FeOOH), recrystallization of iron, and displacement of phosphorus grains at the interface of phases (crystals) occurs. At the same time, artificial hematite $2\text{FeOOH} \rightarrow \text{Fe}_2\text{O}_3 + \text{H}_2\text{O}$ and phosphorus compounds are formed. Thus, access of mineral acid to phosphorus compounds and their transfer into solution is ensured. The firing time is 1 hour. This ensures a high level of phosphorus extraction into the solution during leaching. More time for firing is not required,

because it also leads to the sintering of particles into agglomerates.

A weight of iron ore concentrate weighing 100 g was fired in a muffle furnace at a temperature of 900°C for 1 hour. The cucumber was cooled to a temperature of 25°C. Leaching was carried out with sulfuric acid with a concentration of 49% for 1 hour. The ratio of the solid to liquid phase was $S:L = 1:1.5$. The suspension was stirred, and after leaching it was filtered. The sediment on the filter was washed with a volume of 50 cm³ of cold water. The sediment from the filter was dried at a temperature of 105°C in a drying cabinet for 6 hours to a constant mass, and the content of phosphorus and iron was determined. As a result, a concentrate with a mass fraction of total iron of 55.2% and 1.2% phosphorus was obtained from a concentrate with a mass fraction of total iron of 56.4% and phosphorus – 0.15%.

5. Conclusions

1. The research was conducted on a sample of magnetite-hematite ore with the production of magnetite and hematite concentrate, which have a high mass fraction of phosphorus. As a result of the mineralogical study of the magnetite and hematite concentrate, it was established that the main carrier of phosphorus in the concentrates is apatite, which is found in fine fractions less than 20 μm.
2. To extract phosphorus from magnetite concentrates, it is sufficient to use de-sludging operations in the enrichment technology of rich magnetite-hematite ores, which can remove up to 61% of phosphorus.
3. For effective dephosphorization of hematite concentrates, it is necessary to use direct cationic flotation of apatite, with the help of which it is possible to remove more than 74% of phosphorus from the concentrate.
4. As a result of the analysis of the conducted research and the synthesis of the obtained scientific results, a technology for removing phosphorus from magnetite and hematite concentrates was developed, which allows reducing the mass fraction of phosphorus in the total concentrate from 0.14 to 0.04%.
5. With further improvement of the regime of flotation of hematite concentrates, it is planned to reduce the mass fraction of phosphorus to 0.02%.
6. The proposed technology of leaching of samples of poor hydrogethite high-phosphorus concentrates at high and low temperatures allows us to state that the parameters of the firing process allow the recrystallization of iron and therefore contribute to the access of mineral acid to particles containing phosphorus.

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