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REVIEW

Methods of iron ore desulfurization

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ABSTRACT

The article presents information on raw material resources and iron ore production worldwide. Kazakhstan is among the world's top ten iron ore-producing states. As high-grade iron reserves become exhausted, ores with low iron content and high impurity levels, including sulfur, are increasingly being processed. Various methods of iron ore desulfurization are discussed: flotation, magnetic, and combined approaches based on pyro- or hydrometallurgical processes that alter the chemical composition of the feedstock. The review provides information on methods for beneficiating low-grade iron ore and their significance for the development of iron and steel metallurgical production.

Keywords: iron ore; raw material; desulfurization; flotation; magnetic separation

1. IRON ORE RAW MATERIALS RESOURCES

World iron ore reserves are 190 billion tonnes (USGS data 2023). Four countries among them share 72.2% of the world's iron ore resources: Australia (31.0%), Brazil (17.2%), Russia (16.1%) and China (7.9%) [1].

The biggest iron ore resource base - the Australian one - was estimated to be 58 billion tonnes (including 27 billion tonnes of iron) in 2023. Brazil has 34 billion tonnes of iron ore reserves, with an iron content of 15 billion tonnes. Russia has 29 billion tonnes of ore (14 billion tonnes of iron) in reserves. China's resource base is 20 billion tonnes of ore containing 6.9 billion tonnes of iron. India's reserves were estimated to be 5.5 billion tonnes of iron ore, containing 3.4 billion tonnes of iron. Iron ore is mined commercially in over 40 countries. The main producers are Australia, Brazil, China and India, which together account for 80.6% of world production [2].

Australia is responsible for more than a third of the world's iron ore, 590 million tonnes of iron content, of a global total of 1.5 billion tonnes (39.3%). Australian ores are predominantly hematite with an iron content of up to 65%. After extraction, the ore is processed by relatively simple crushing and screening to produce direct shipping ore (DSO) suitable for metallurgical processing without additional beneficiation. Brazil is the world's second-largest iron ore producer, mining 280 million tonnes of iron ore and holding an 18.7% share of the world market. A characteristic of Brazilian iron ores is their low content of harmful impurities - silica, alumina and sulfur. Minas Gerais, with four large open-pit mines, is the home of enriched hematite-martite ores (63-69% iron).

China's iron ore output was 170 million tonnes of iron (11.3% of global output) in 2023, while also holding its position as the world's largest consumer of the raw material. The country is the world's largest steel producer and processes over half the world's mined ore. But the quality of Chinese iron ore is poor, with low iron content (no more than 35%) due to high impurity levels.

The world's iron ore output is 170 million tonnes of iron, and India produces 11.3% of it. Hematite is the main component of some 75% of the iron ore; the rest is magnetite.

In 2023, Russia produced 58 million tonnes of iron (3.9% of the world volume). More than half of the iron ore raw material production was provided by enterprises developing deposits of the Kursk Magnetic Anomaly, with iron content exceeding 60%. Western Europe lacks rich iron ore deposits, with the exception of Sweden, where deposits are characterized by high iron content (60-65%). As a result, many European countries have ceased developing their own deposits.

In Eastern Europe, the average iron content in mined ores is approximately 40%. In the United States, the richest deposits have been exhausted, and ores with up to 50% iron are now being processed. Canada and Mexico have rich ores with 61-63% iron. Among Asian countries, only India mines iron ore with up to 63% useful component.

Kazakhstan possesses significant iron ore resources totalling 2.5 billion tonnes with an iron content of 0.9 billion tonnes; 90% of these reserves are concentrated in the northern regions of the country [1]. It is also one of the top ten producers of iron ore in the world, with 8.8 million tonnes of iron produced in 2023.

The Sokolovskoye, Sarbayskoye, and Kacharskoye magnetite deposits, as well as the brown iron ore deposits Ayatskoye and Lisakovskoye, are in industrial development. A key advantage of Kazakhstani iron ore is its relatively high quality: 6.9% are ores requiring no beneficiation, 73.1% are easily beneficiable, and 20% are difficult to beneficiate.

More than 50% of mined raw materials are exported; domestic consumers include the Karaganda Metallurgical Plant, the Yermakovsky and Aktyubinsky ferroalloy plants [3]. Studies on the use of briquetted mono-charge in ferrochrome smelting and on the beneficiation of refractory iron-manganese ores from Kazakhstani deposits have been conducted in support of the development of domestic ferroalloy production [4, 5].

2. TECHNOLOGICAL TYPES OF IRON ORES

Various types of iron ores exist magnetite, hematite, oxidized, and ferruginous quartzites, which determines the choice of different technologies for producing iron ore concentrates [6-9].

Magnetite has a high iron content (up to 72.4%) and exhibits high magnetic susceptibility, enabling its separation from gangue and the production of a high-quality concentrate (above 70%) with very few impurities. Hematite has a slightly lower iron content than magnetite (up to 70%), often occurs together with magnetite and limonite, and is more easily reduced than magnetite. Limonite is a mixture of hydrated iron oxide minerals, primarily consisting of goethite. Siderite is iron carbonate, free of sulfur and phosphorus, a mineral analogous to calcite in which calcium is replaced by iron.

The iron content in ores can vary significantly: ores with more than 50% iron are considered rich, those with 25-50% are average-grade, and those with up to 25% are lean. Rich ores, comprising approximately 20% of global reserves, often require no beneficiation; simple beneficiation schemes are applicable to the majority of ores (2/3); and 20% of lean ores must be processed using complex, combined ore preparation schemes.

In magnetite ores, magnetite dominates (over 70%), and secondary minerals include martite, hematite, pyrrhotite, pyrite, chalcopyrite, cobaltite, sphalerite, galena, marcasite, and arsenopyrite. Semi-oxidized ores are characterized by the presence of magnetite and martite (a variety of hematite formed by the oxidation of magnetite), while oxidized ores are represented by martite or brown iron ores.

Magnetite ores have a variable chemical composition, with an average iron content of 30-53%. In addition to iron, the ores contain sulfur, phosphorus, and other metals. Magnetite ores typically have high basicity and low silica content. The variable chemical composition of magnetite is due to isomorphic substitution

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in the mineral's crystal lattice by magnesium, aluminum, and other elements, altering the iron content in magnetite. Oxidised ores in deposits are represented by small amounts of martite and brown limestone ores.

Among non-metallic minerals, quartz, pyroxenes, garnet, calcite, serpentine, chlorite, and apatite are most commonly found, as well as amphiboles, spinel, biotite, muscovite, epidote, phlogopite, feldspar, sphene, and tourmaline. Differences in ore composition between individual deposits are due to variations in the content and ratio of ore and non-ore minerals. Mined iron ore often has a complex structure and high levels of undesirable impurities, such as quartz and sulfur. To raise the quality of iron ore concentrates intended for metallurgical processing, the content of these impurities must be reduced.

The growing demand for iron ore is associated with its high consumption in the metallurgical industry. Due to the depletion of high-quality iron ores, complex ultra-fine iron ores with high impurity contents, including sulfur, are being processed [10-12]. Pyrrhotite (Fe1-xS) represents a common sulfide mineral found in magnetite ore as an associated component.

The processing of high-sulfur magnetite is insufficiently efficient, as it tends to associate with sulfide minerals such as pyrite (FeS_2) and pyrrhotite ($Fe_{1-x}S$) [13]. Separating pyrrhotite from a magnetite matrix is complicated by their comparable magnetic behaviour and by magnetic flocculation (the clustering of fine magnetic particles in a liquid medium under a magnetic field) between pyrrhotite and magnetite.

Contamination of magnetite concentrates with sulfur released from pyrrhotite presents a problem in the iron ore smelting process. Steel containing sulfur loses strength at high temperatures. Sulfur affects the microstructure of steel by forming sulfide inclusions that may initiate cracks when unevenly distributed. Pyrite, pyrrhotite, and sulfate constitute the chief sources of sulfur in ores. Modern ore desulfurization methods include flotation, magnetic separation, and combined beneficiation.

3. METHODS OF IRON ORE DESULFURIZATION

Modern ore desulfurization methods include flotation, magnetic separation, and combined beneficiation [14]. Flotation beneficiation is based on differences in the physicochemical properties of mineral surfaces, which manifest as unequal water wettability and selective particle attachment at the interphase boundary.

Combined beneficiation schemes include pyro- or hydrometallurgical stages that ensure targeted changes in the chemical composition of the initial raw material. Pyrometallurgical operations include roasting, smelting, and converting; hydrometallurgical operations include leaching, precipitation, extraction, and sorption. Roasting is used to adjust the magnetic characteristics of weakly magnetic iron-bearing minerals: at temperatures of 600-800 °C, hematite (Fe_2O_3) is reduced to magnetite (Fe_3O_4) under the action of gaseous or solid reducing agents. The ore obtained after roasting is separated using low-intensity magnetic separators.

Magnetic beneficiation is based on differences in the magnetic properties of the minerals being separated and has found the widest application in the production of magnetite concentrates, as well as in the beneficiation of ferrous metal ores in general. The process is based on the separation of mineral particles in a magnetic field under the combined action of magnetic and mechanical forces - either by extracting magnetic components or by retaining them.

The quantitative characteristic of magnetic properties is specific magnetic susceptibility, by the value of which all natural minerals are divided into three groups: strongly magnetic, weakly magnetic, and non-magnetic.

Strongly magnetic (ferromagnetic) minerals are predominantly iron bearing – magnetite, pyrrhotite, and similar; their separation is carried out on low-field separators with a field intensity $H < 1500$ Oe (oersteds). The extensive group of weakly magnetic minerals includes, in particular, hematite, ilmenite, and garnet; their recovery requires high-intensity separators with a field of 10,000-20,000 Oe (800-1600 kA/m). Non-magnetic minerals – quartz, apatite, and others - cannot be recovered by any method of magnetic separation using modern equipment with field intensities up to 20,000 Oe.

Pyrrhotite ($Fe_{1-x}S$) is known to occur in two crystalline modifications - monoclinic and hexagonal. The monoclinic variety has a higher sulfur content than the hexagonal one, yet, owing to the ordered distribution of iron vacancies within the lattice, it exhibits high magnetic susceptibility. Hexagonal pyrrhotite cannot recover with a low-intensity magnetic separator, whereas the monoclinic form is recovered to a varying extent.

Research on the desulfurization of sulfur-bearing iron ore has developed in response to increasing demand for iron and its alloys in industry [15-19]. Studies have been conducted on iron ore desulfurization via reduction roasting followed by magnetic separation [20-24].

To extract iron from high-sulfur iron ore, reduction with coal in the presence of lime was carried out, achieving a total iron content and metallization degree of 80% and 93%, respectively [25]. Subsequent magnetic separation achieved an iron recovery rate of 75%. The roasting process is conducted to generate calcium sulfide rather than SO2, thereby avoiding environmental problems.

Using a mineral phase transformation via hydrogen reduction, a high-quality iron ore concentrate was obtained by reducing the sulfur content in refractory iron ore [26]. It was found that in the oxidative heating stage, sulfur in the ore is oxidised to SO_2. At the reduction roasting stage at 520 °C, hematite in the iron ore powder is almost completely converted into magnetite. The iron-bearing minerals can then be recovered efficiently by grinding and low-intensity magnetic separation. Under these conditions, the iron content of the magnetic concentrate increased to 66.40%, iron recovery increased to 92.44%, and the sulfur content decreased from 0.547% to 0.038%. Thus, this technology has significant technical advantages and environmental potential for processing sulfur-bearing refractory iron ores.

A method for beneficiating fine fractions of low-grade hematite ore containing carbonates using magnetic roasting and magnetic separation was proposed [27]. Hematite and siderite undergo nearly complete transformation into magnetite with the addition of 8 wt.% coal at a roasting temperature of 800 °C for 8 minutes. Under optimal conditions, a high-grade magnetic concentrate assaying 65.4 wt.% iron was produced at an iron recovery of 92.7%. The results show that the magnetic susceptibility of the hematite ore can be significantly enhanced by magnetization roasting via the selective transformation of hematite and siderite into magnetite, thereby facilitating the separation of the ore from the non-magnetic minerals. The majority of weakly magnetic sulfides are discharged to tailings as a result of magnetic beneficiation, except for a small amount of monoclinic pyrrhotite, which is ferromagnetic [26]. To meet metallurgical requirements, the product's sulfur content must not exceed 0.06%. It has been proposed to reduce the magnetic activity of pyrrhotite by purging the pulp with oxidizing agents in magnetic-gravity separators.

Currently, flotation is considered an effective method for removing pyrrhotite from magnetite [29-34]. Research on iron ore flotation mainly focuses on selecting collectors across different pH ranges. The importance of depressants in reverse flotation for the recovery of iron oxides has also been noted.

Studies on the flotation of pyrrhotite using a single collector are known. It has been established that xanthate is a good collector for pyrrhotite.

Published data on the separation of pyrrhotite from magnetite indicate a reduction in the level of sulfide minerals to below 0.2%.

Reference [35] reported the effect of pH, activator and collectors on the flotation of high-sulfur magnetite ore. Under the optimum flotation conditions – grinding fineness of 0.074 mm, pH 6, 400 g/t $CuSO_4$ and 400 g/t combined collectors – and after the magnetic separation, the sulfur content in the magnetite concentrate decreased from 3.20% to 0.18%, and the iron content increased from 57.29% to 71.17%. The addition of $CuSO_4$ as an activator promoted the formation of S^0 and Cu+ ions, thereby enhancing pyrrhotite floatability. The efficiency of flotation separation of sulfur minerals can be improved by using a collector mixture.

Results of studies on the flotation recovery of sulfide minerals from high-sulfur magnetite are described [36-41].

The behavior of pyrite during flotation as a function of pH and Fe(II) ion concentration was studied [42]. Flotation suppression in the neutral pH range explained by the mechanism of physical adsorption of the collector. The propagation rate of xanthate-Fe(II) ion interaction products at the gas-water interface varies depending on concentration and pH. The identified mechanism explains both the reduction of pyrite flotation in neutral media and its activation under alkaline conditions.

A study on the processing of an iron ore deposit in Botswana is presented, with potential for practical implementation through the maximum utilization of resources via re-duction roasting followed by magnetic separation using sub-bituminous coal as a reductant [43].

Flotation is used to separate pyrrhotite from magnetite in sulfur-rich iron ores. A process has been developed to remove pyrrhotite from the ore, using potassium butyl xanthate as the principal collector. Copper sulfate is introduced as an activator to accelerate the flotation rate of pyrrhotite. Flotation is conducted at the natural pH of the ground ore (7.5 to 9.0). The main problem is the loss of valuable minerals in the tailings. Hence, a reagent scheme is suggested to successfully separate and enhance the concentrate recovery by establishing an appropriate chemical environment.

New data on the separation of iron-bearing minerals is given in [44]. Froth flotation using xanthate- and dixanthogen-based collectors was used to separate pyrrhotite from magnetite. It was found that xanthate or dixanthogen alone do not provide selective separation of pyrrhotite and magnetite by flotation.

When using xanthate, significant magnetite losses were observed, whereas with dixanthogen, the degree of desulfurization was low. When xanthate and dixanthogen were mixed as a collector, a high degree of desulfurization was achieved with minimal magnetite losses. The synergistic effect of the mixed collector on pyrrhotite was investigated using electrokinetic measurements. It was established that xanthate, adsorbed on the pyrrhotite surface, determines its selectivity relative to magnetite, while dixanthogen bonded to xanthate enhances its hydrophobicity.

Flotation provides excellent selectivity and is crucial for obtaining high-quality concentrates.

For instance, reference [45] describes a combined magnetic separation-flotation process applied to low-grade complex iron ore. After magnetic beneficiation of iron-bearing minerals, the silicate waste rock was separated by flotation and obtained 69% Fe from a feed material containing 20.06% Fe. The developed innovative technology can be applied to the beneficiation of complex iron ore.

A novel collector based on a sulfur-containing ionic liquid (SCIL), using isobutyl xanthic acid with an N-tetrabutyl quaternary ammonium salt, was proposed [46]. The reagent was used for the first time as a flotation collector for the selective separation of pyrrhotite from magnetite by reverse flotation. The flotation test results showed that SCIL possesses superior collecting ability and selectivity for pyrrhotite compared to the conventional sodium isobutyl xanthate (SIBX) collector. It was established that SCIL chemisorbs on the pyrrhotite surface via CN^- and CS^- groups; the sulfur content in the feed decreased from 25.72% to 2.92% in the concentrate.

Results of beneficiation tests on ores from three magnetite deposits (Sahavaara, Pellivuoma, and Hannukainen) are presented, with the aim of removing pyrrhotite and obtaining high-quality magnetite concentrates with a sulfur content below 0.05 wt.% [47]. The process involved grinding samples to relatively fine particles to liberate valuable minerals, magnetic separation to recover magnetite, and flotation to remove pyrrhotite.

A study was conducted to measure the degree and rate of reaction of a relatively pure pyrrhotite sample using a standard xanthate collector at various pH values and conditioning times [48]. The study showed that, in most cases, the surface coverage of pyrrhotite by SIBX (sodium isobutyl xanthate)-derived compounds is multilayer, and that pre-treatment with copper sulfate facilitates this. Guar gum suppresses the hydrophobicity of pyrrhotite.

One method for reducing the sulfur content in magnetite iron ores is reverse flotation (flotation of pyrite into the tailings) [49]. Xanthates are readily adsorbed onto the pyrite surface, and the pyrite surface can oxidize the xanthate to dixanthogen, resulting in effective pyrite flotation up to pH 11 within a mineral particle size range of 10-100 µm. Pilot tests of the reverse flotation technology were carried out to remove sulfur from the pelletizing feed - the magnetic concentrate of a beneficiation plant (Golgohar mine, Iran). It was established that the sulfur content in the iron concentrate can be lowered from 1.2% to 0.33% in a single stage of pyrite flotation.

Experiments on microbial desulfurization were conducted using mixed acidophilic microbiota and Acidithiobacillus ferrooxidans with S and FeS_2 as electron donors [50]. The degrees of desulfurization were 79.8% and 45.7% over 30 days. Recovery coefficients exceed 90% and 88%, respectively. It was established that the sulfur transformation pathway proceeds from S^{2-}/S_2^{2-} to S_n^{2-}/S^o and finally to SO_4^{2-}.

During the mineralogical investigation of some high-sulfur iron ores, complex dispersed intergrowths between pyrrhotite and magnetite were identified, as well as a low degree of liberation of fine-grained sulfurous pyrrhotite. It was established that the grinding process and grinding fineness are important factors affecting the efficiency of flotation desulfurization of high-sulfur iron ore.

It was found that the degree of desulfurization in multi-stage grinding is significantly better than in direct single-stage fine grinding [51]. Combined activators were sequentially applied to the ore pulp, followed by reverse flotation of pyrrhotite with combined collectors to obtain magnetite concentrate.

The resulting iron ore concentrate contained 64.28% Fe and 0.42% S, respectively, with an iron recovery of 53.62% and a desulfurization degree of up to 90%. Technology is oriented for industrial application in the desulfurization of high-sulfur iron ore.

One promising approach to intensifying and enhancing the efficiency of the flotation process is electrochemical treatment, which makes it possible to influence the flotation activity and the speciation of reagents in the flotation pulp, to regulate the surface properties of minerals and increase the contrast of their processing characteristics, and to alter the ionic composition of the liquid phase and the redox potential of the pulp [52-54].

An effective method for reducing the sulfur content in magnetite concentrate is the leaching of impurity iron sulfide minerals (pyrite, pyrrhotite) using aqueous solutions with a high concentration of strong oxidizers, such as oxygen and active forms of chlorine-bearing ions produced by the electrolysis of mineralized aqueous systems [55].

The leaching of sulfur from magnetite concentrate was studied using a solution of active chlorine obtained by the electrolysis of mineralized waters, proceeding through the oxidation of iron sulfide to sulfate by electrolytic oxygen and active forms of chlorine-bearing ions. A sulfur removal degree of 30 to 89% was achieved, reducing the concentrate's sulfur content from 0.1% to 0.01-0.07% and yielding concentrates that meet world-market requirements.

In investigating the leaching of sulfur from magnetite concentrate using an active chlorine solution, the possibility of removing 90% of the sulfur was established [56]. Thus, the use of electrolytic hypochlorite solutions for lowering the sulfur content in magnetite concentrate has been substantiated, yielding standard-grade magnetite concentrates with a sulfur content of 0.01%.

The partial desulfurization of mining waste using froth flotation was investigated, enabling the removal of more than 95% of the pyrrhotite [57]. The desulfurized waste contains a minor number of sulfides (less than 0.5 wt.% S), mainly in the form of pyrrhotite-lizardite intergrowths. The feasibility of using the desulfurized material as a component of a drainage cover was evaluated.

A comparison of predicted magnetite liberation indicators for difficult-to-beneficiate iron ores from the Yun-Yaginsky deposit in the Polar Urals with actual disintegration results was conducted [58]. The high accuracy of calculations using optical-geometric analysis methods enables rapid assessment of the technological properties of iron ore occurrences in the early stages of geological exploration.

CONCLUSION

Many studies aimed at reducing the sulfur content in iron ores and iron ore beneficiation waste have been reported, using flotation, leaching, bioleaching, thermal decomposition, and magnetic separation. The development of an iron ore desulfurization process and the selection of an appropriate method depend on factors such as the state of sulfur, its content, particle size, and crystal structure.

Iron ore desulfurization is possible through thermal decomposition. The process has several advantages: complete iron yield; no need to use chemicals as in flotation and hydrometallurgical processes; the oxidizing agent is oxygen, which is an accessible natural resource. However, the thermal decomposition process has two main disadvantages: a change in the composition of iron oxide and the release of sulfur oxides into the environment.

In iron ore desulfurization by sintering, several challenges arise: large investments in equipment and energy infrastructure, high operating costs, waste generation, changes in iron ore composition, and low desulfurization efficiency, which make the process uncompetitive compared to flotation under industrial conditions.

For biodesulfurization processes, research on technical improvements to the bioleaching process is needed. It is necessary to compare the influence of various types of bacteria on the rate and efficiency of desulfurization, as well as to describe the optimal conditions for maximum bacterial activity and, consequently, maximum desulfurization rate.

The use of the leaching process is limited by the high cost of leaching reagents. In addition, regeneration of leaching reagents is required. The possibility of simultaneous desulfurization and recovery of valuable metals from the ore should also be studied.

Currently, flotation is an effective method for removing sulfur from iron ores and concentrates. The ability to modify flotation conditions to simultaneously remove several types of impurities makes this process attractive for industrial desulfurization. One advantage of the method is the ability to combine flotation with magnetic separation. At the same time, the method has the following disadvantages: loss of sulfide iron during desulfurization and inability to process sulfurous minerals to obtain industrial products.

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