International Journal of Materials Science and Technology Studies
ISSN: 3006-7693 (Print), ISSN: 3006-3744 (Online) | Volume 1, Number 2, Year 2024
DOI: https://doi.org/10.62051/ijmsts.v1n2.07
Journal homepage: https://wepub.org/index.php/IJMSTS/index

Development Status of Low-Grade Magnesite Beneficiation Technology

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ABSTRACT

Magnesite is a non-metallic mineral rich in magnesium. Due to its excellent fire resistance, it is widely used in the manufacture of magnesia-carbon bricks, magnesia and other refractory materials, which are widely used in various fields such as chemical industry and metallurgy. Due to the uneven distribution of magnesite resources and the reduction of high-quality magnesite resources caused by large-scale mining, the selection of low-grade magnesite has become a key problem of magnesium resources. The lower grade ores are abandoned because they contain a large amount of impurities and do not meet the requirements of industrial production, which leads to the unreasonable utilization of magnesite resources. This paper summarizes the current status of low-grade magnesite separation technology and flotation reagent development in China, and gives some suggestions on the development of low-grade magnesite beneficiation technology, aiming at providing certain theory and reference value for magnesite beneficiation.

KEYWORDS

Low-grade magnesite; Magnesite separation status; Magnesite separation technology

1. INTRODUCTION

Magnesite is also known as magnesite, and its main component is magnesium carbonate ($MgCO_3$), which is a non-metallic mineral rich in magnesium. Gangue minerals are mainly silica-containing minerals dominated by quartz, talc and chlorite, calcium-containing minerals dominated by dolomite and iron-bearing minerals dominated by magnetite and red (brown) iron ore. Because of its excellent fire resistance, magnesite is used in the manufacture of magnesia-carbon bricks, magnesia and other refractory materials, and because magnesite magnesium materials have better performance than other refractory materials, it is widely used in the fields of national defense science and technology, aerospace materials, construction industry, metallurgy, chemical industry, electronics and ceramic materials.

According to the report of the United States Geological Survey (USGS), as of 2021, the global magnesite reserves reach 7.2 billion tons, mainly distributed in Russia, China, Slovakia, Australia, Greece, Brazil and Turkey and other countries, among which China's magnesite reserves are about 1 billion tons, ranking second in the world, accounting for 13.89% of the world. China has a very rich magnesite resources, in 2021 China's magnesite output reached 21 million tons, ranking first in the world. China is rich in magnesite resources, mainly distributed in Liaoning, Shandong, Gansu and other regions [1]. After years of large-scale mining, there are fewer and fewer high-quality resources that can directly meet production requirements.

In recent years, with the continuous exploitation of natural magnesite resources, the reserves of high-grade magnesite resources have gradually decreased. At the same time, due to the use of the mining method of "mining rich and abandoning poor, developing large and abandoning small" in the mining process, a relatively large number of low-grade magnesite resources have finally been abandoned and not properly utilized, resulting in environmental pollution and forming a vicious circle. At present, in the form of gradually decreasing reserves of high-grade extra grade magnesite resources in China, the average grade of magnesite in China also decreases. According to the data, low-grade magnesite reserves with relatively high content of silicon and calcium impurities in ores account for 60% of the total amount of magnesite resources in China, and such low-grade magnesite is often abandoned because of its processing difficulties. It will not only waste resources, but also have a great impact on the environment. In the future, low-grade magnesite will become the main mineral resources for the enrichment and recycling of magnesium resources.

Low-grade magnesite cannot be rationally utilized because its mineral composition contains a large number of impurities such as silicon, calcium, iron, etc. In these impurities, calcium-containing minerals are generally dolomite, calcite and other minerals, which are carbonate minerals with a lattice structure similar to magnesite, and belong to the trigonal crystal system. Therefore, these calcium-containing minerals have the same physical and chemical properties as magnesite. Therefore, qualified magnesite cannot be effectively removed in mineral flotation separation, so low-grade magnesite is often discarded as waste rock, which leads to a serious waste of magnesite resources [2]. SiO_2 of silicon-containing minerals in low-grade magnesite will produce silicate material with lower melting point due to the calcination reaction process, and the existence of such silicate material will reduce the strength of refractory materials. However, the presence of calcium-containing minerals will lead to the reaction of CaO to form $CaSiO_3$ during calcination, which will cause the loose collapse of refractory materials during cooling and fail to form qualified products. Therefore, at present, only low-grade refractories and construction materials with low demand for fire resistance can use such low-grade magnesite as raw materials. This paper summarizes the current status of low-grade magnesite separation technology and flotation reagent development in China, and gives some suggestions on the development of low-grade magnesite beneficiation technology, aiming at providing certain theory and reference value for magnesite beneficiation.

2. RESEARCH STATUS OF MAGNESITE SEPARATION TECHNOLOGY

Magnesite in China has high quality and abundant resources. However, there are some problems in the exploitation and utilization of magnesite resources in the early years. First, due to the phenomenon of "mining rich and abandoning poor" in the process of magnesite mining, high-quality magnesite with grade less than 46% has not been effectively utilized, resulting in a serious waste of magnesite resources; On the other hand, the metallurgical industry has put forward higher requirements for the quality of magnesium refractory products. Therefore, efficient purification of low-grade magnesite is an urgent problem to be solved. According to the different properties of magnesite ores, the corresponding beneficiation methods are not the same.

2.1. Research Status of Magnesite Flotation Process

Flotation method is one of the best means to realize effective separation and utilization of magnesite resources. In the separation test of magnesite, how to effectively remove silicate and carbonate gangue minerals in magnesite is the key research problem. Gangue minerals associated with magnesite are mainly silicate such as quartz, talc and serpentine, and calcium-bearing carbonate minerals such as dolomite and calcite. Flotation processes are different for different gangue minerals. For silicate gangue minerals, reverse flotation is generally used for desilication, while for calcium-containing carbonate gangue minerals, forward flotation is used for desilication.

2.1.1. Reverse flotation desiliconization of magnesite

Quartz is a common silicate gangue mineral, the main component of which is SiO_2. The crystal structure belongs to the trigonal crystal system and the silicon-oxygen tetrahedron structure. The two silicon-oxygen tetrahedrons are connected by O atoms and extend into a stable frame-like structure. There is no metal site on the surface of quartz, and the surface properties of magnesite are very different. Therefore, adding amine cationic collector to the reverse flotation desiliconization of magnesite is easier to remove by the reverse flotation process of flotation quartz. Ma Jinsheng et al. [3] conducted a reverse flotation desiliconization test on magnesite in Liaoning Province. Gangue minerals were mainly quartz and dolomite. The results showed that by using dodecamide as collector and one coarse and one refined reverse flotation process, a concentrate product with MgO content of 47.42% and SiO_2 content of 0.24% was obtained. Zhu Yangge et al. used dodecamine as collector to conduct reverse flotation desiliconization test on magnesite. Through the reverse flotation process of one crude and three fine, the yield of magnesite concentrate was 70.22%, the grade of MgO was 46.75%, the recovery rate was 72.95%, the content of SiO_2 was reduced from 3.74% to 0.18%, and the separation index was good. Ma Zhongchen [4] et al studied the reverse flotation desiliconization process of a high silicon and low grade magnesite in Liaoning Province. The process flow of one coarse selection and one fine selection, grinding fineness of -0.074mm accounted for 80%, pH value of pulp adjusted to 5.5 by hydrochloric acid, water glass inhibiting magnesite, and process conditions of collecting silicon-containing minerals with new collector Mmm-10 were determined. A magnesite concentrate with MgO grade of 46.02%, MgO recovery rate of 78.36%, SiO_2 grade of 0.275% and SiO_2 removal rate of 94.25% were obtained.

2.1.2. Flotation decalcification of magnesite

Magnesite, dolomite and calcite are all carbonate minerals with the same Ca^{2+} and Mg^{2+} ions exposed on their surfaces, and the differences are reflected in the density of Ca^{2+} and Mg^{2+} ion sites. The three minerals show similar flotation properties in flotation, which makes it difficult to separate magnesite from dolomite and calcite. In the forward flotation decalcification of magnesite, inhibitors are usually added to inhibit calcium-containing minerals such as dolomite and calcite, and then fatty acid collectors are used to flotation magnesite. Huang Chaade et al. adopted the inverse flotation process for magnesite, and under the optimal reverse flotation conditions of grinding fineness of -0.074mm, mass fraction of 79.8%, pulp pH 6, sodium silicate dosage of 500 g/t, and collector octadecylamine dosage of 300 g/t, reverse flotation concentrate with MgO grade of 44.21% could be obtained. Under the optimum positive flotation conditions of pulp pH 9, sodium hempetaphosphate dosage 150 g/t, foaming agent dosage 100 g/t and collector oleic acid dosage 1500 g/t, a concentrate with MgO grade of 46.31% can be obtained. The closed-circuit test was carried out under the best flotation conditions. The final MgO grade of the concentrate was 46.12%, the MgO recovery was 75.88%, and the mass fraction of gangue minerals SiO_2 and CaO was reduced to 0.92% and 0.81%. The quality of the concentrate met the requirements of industrial grade one. Ma Yingqiang et al. [5] studied the influence of acetyl acetone on the desilication and decalcification of magnesite by single-mineral and artificial mixed ore flotation tests under the dodecylamine collector flotation system, and analyzed the Zeta potential and infrared spectrum of the mineral surface before and after the application of chemicals to explore that acetyl acetone has a certain inhibitory effect on magnesite. The effect on dolomite and quartz is not obvious, and acetyl acetone has a good separation effect on reverse flotation desilication and decalcification of magnesite.

2.2. Development Status of other Sorting Technology of Magnesite

There are different reasons for the formation of magnesite ore deposit in each geomorphic environment, in addition to flotation, mineral processing industry workers through different tests to separate magnesite, generally gravity separation, flotation, magnetic separation, thermal separation, electrical separation, chemical separation, microbial separation and other mineral processing methods to achieve magnesite separation. This paper focuses on four aspects of flotation, electric separation, chemical separation and microbial separation to summarize.

2.2.1. Thermal preparation

Thermal separation method is a method to use the difference in thermal properties of magnesite and talc gangue to separate, it removes impurities mainly SiO_2, and part of CaO and Al_2O_3, so the thermal separation method has certain limitations in the treatment of magnesite. When the temperature reaches the decomposition temperature of magnesite, magnesite becomes magnesium oxide, its strength decreases, it is loose and fragile. On the other hand, talc forms enstatite and cristobalite with high strength. Rolling screening of calcined products results in a fine product that is a concentrate with high MgO content.

2.2.2. Electric separation

Electrical separation method is a mineral separation method that makes use of the inherent electrical properties of minerals, such as electrical conductivity, dielectric constant and rectification, and separates minerals under the action of external electric field through conduction induction charging, tribological charging and corona charging Cao Yunxiao used a high-voltage corona charged electric separator to carry out electric separation and impurity removal of magnesite broken by high-voltage pulse discharge in water. The test results showed that after electric separation, more Si-containing impurities were separated, the fine mineral yield and concentrate grade were higher, and the content of MgO exceeded 46%, indicating the feasibility and effectiveness of electric separation and impurity removal of magnesite. Zhao Xiaoxiao et al. [6] studied the feasibility of removing quartz from low-grade magnesite by tribroelectric separation, and the test results showed that under the optimal conditions, the separation index with a grade of 45.09% and a recovery rate of 68.40% was obtained, effectively achieving the removal of SiO_2.

2.2.3. Chemical separation process

Chemical dressing method is to first calcined magnesite at high temperature, enhance the surface activity of magnesite and increase the solubility of magnesite, and then leach with different leaching agents, the commonly used leaching agents are hydrochloric acid, sulfuric acid, ammonium salt and so on. According to the difference in leaching degree between magnesium and impurity minerals, the impurities are separated, and then the obtained magnesium salt solution is pyrolyzed and calcined to obtain high-purity MgO products. Li Pengcheng et al. [7] conducted a roasting-leaching test on a low-grade magnesite in Haicheng, using NH_4Cl as the leaching agent. Under the optimal roasting and leaching conditions, the leaching rate of lightly burned magnesia was 88.5%.

2.2.4. Microbial beneficiation

The hydrophobicity of the surface is determined by the structure of sugars, proteins and lipids on the surface of the microorganism, and the wettability of the mineral can be changed when the microorganism is adsorbed to the surface of the mineral. Therefore, similar to traditional mineral processing agents, microorganisms can also be used in mineral flotation. BOTERO et al. [8] studied the flotation of magnesite and calcite by Rhodococcus opacu bacteria as a microbial collector, and the results showed that Rhodococcus opacus bacteria had a greater affinity for magnesite than for calcite, which made magnesite show better floatability.

3. RESEARCH STATUS OF CHEMICALS FOR SEPARATING MAGNESITE

In the study of flotation purification of magnesite, collector and inhibitor have always been the focus of research. For the reverse flotation desilication process, amine cationic collectors are often used, and such collectors have obvious removal effect on silicate minerals in magnesite, while amine collectors and amine compound agents are more common. A large number of experts and scholars at home and abroad have studied the mechanism of action between such collectors and minerals, and found that amine collectors generally interact with mineral surfaces in the form of physical adsorption. Usually the electrostatic attraction of the mineral surface is the dominant force. In the industrial flotation of magnesite, the most common regulator is sodium hexametaphosphate and water glass. For the positive flotation decalcification process, fatty acid anionic collectors are often used. Anionic collectors include higher fatty acid salts and sodium salts, etc. Such collectors bond atoms with oxygen atom sites, such as sodium oleate, sodium sulfonate, etc. Inhibitors alter the adsorption of the collector and mineral by increasing the difference in surface properties between minerals.

3.1. Amine Cationic Collector

Cations with hydrophobic hydrocarbon groups are produced after the dissociation of amines. Most of them are organic compounds containing N and are commonly used to flotation silicate minerals such as quartz, feldspar and talc. According to the different number of hydrocarbon groups on N atom, amine collectors can be divided into primary amine, secondary amine, tertiary amine and quaternary amine, of which primary amine is the most widely used. According to the different number of N-containing atoms, amine collectors can be divided into monadic amines, binary amines and ternary amines. In addition, an ether group (O-C-O) or acyl group (-CO-) is introduced between the R group and the NH_2 group, and such agents are called ether amines and amides [9]. Dodecylamine is the most commonly used amine collector for reverse flotation desiliconization of magnesite, and the solubility of dodecylamine in water is small, or almost insoluble, so it often reacts with acetic acid or hydrochloric acid to generate the corresponding salt, which is diluted for flotation. Dodecylamine exists in solution mostly in the form of RNH^{3+} plasma and molecule-ionic polymer of $RNH_2 \cdot RNH^{3+}$, which is an effective component of harvesting. However, in flotation, dodecylamine has disadvantages such as low selectivity, poor water solubility and high foam viscosity. Li Qiang used dodecylamine or sodium oleate as collector to carry out single mineral flotation tests on magnesite, and explored the effects of sodium hexametaphosphate, sodium silicate and various inescapable ions on the floating of magnesite under different collector action systems. The test results showed that sodium hexametaphosphate and sodium silicate inhibited the floating of magnesite under different collector action systems. When the pH of the pulp is weakly alkaline, the inhibition effect of water glass on magnesite is stronger, and the inhibition effect decreases gradually with the increase of the pH of the pulp.

The mechanism of flotation of quartz by amine collector has been studied by many scholars. Yang et al. explored the action mechanism of PEOLA (pentaethoxydodoamine) in the flotation separation of magnesite and quartz. Through potentiodynamic, adsorption capacity and infrared analysis, they found that PEOLA was adsorbed on the quartz surface through hydrogen bonding and electrostatic interaction, and there was electrostatic repulsion between magnesite and PEOLA. The adsorption of PEOLA on the magnesite surface is reduced. Sun et al. [10] explored the mechanism of NDOA (octadecyl dimethyl tert-amine) in the flotation separation of magnesite and quartz. Through contact Angle, scanning electron microscope, infrared and XPS analysis, they found that NDOA was adsorbed on the quartz surface through hydrogen bond and electrostatic action, and the formation of N+-H-O hydrogen bond made the adsorption of NDOA on the quartz surface more stable. Therefore, quartz has better floatability.

3.2. Fatty Acid Anionic Collector

Fatty acid collector is a kind of anionic collector, the molecular structure contains carboxyl group (R-COO-) functional group, has a high activity. At present, the more commonly used fatty acid collectors in flotation are oleic acid, oxidized paraffin soap, tall oil, naphthenic acid, etc., which are commonly used to flotation metal oxide ore, barite, fluorite and other minerals. The fatty acid collector can form insoluble salts with the metal ions on the surface of the mineral, making the mineral hydrophobic.

Wei Qin et al. [11] carried out a purification test study on a low-grade magnesite. The contents of MgO in the raw ore were 44.16%, SiO_2 and CaO were 5.88% and 1.37%, respectively. Dotwelve amide was used as the collector in reverse flotation and NaOL was used as the collector in positive flotation. The magnesite concentrate with MgO content of 46.85%, MgO recovery of 59.48% and impurity SiO_2 content of 1.21% was obtained.

Sun et al. studied the role of Tween 80 (polyoxyethylene sorbitan monooleate) as an additive in the flotation separation of magnesite and dolomite in NaOL flotation system. The single mineral flotation test shows that Tween 80 can increase the selectivity of NaOL. In the mixed ore flotation test, adding Tween 80 with a concentration of 5 mg/L can increase the MgO content in magnesite concentrate from 44.88% to 45.73%. The CaO content in concentrate decreased from 1.92% to 1.29%, and the efficiency increased significantly. Surface tension, potentiodynamic, contact Angle, infrared spectrum and XPS tests show that Tween 80 can promote the dissolution and dispersion of NaOL, promote the adsorption of NaOL on mineral surfaces, and the ethylene oxide group in Tween 80 can form a complex with Ca^{2+}, reducing the role of Ca^{2+} and NaOL. So as to achieve the purpose of inhibiting dolomite.

In recent years, new fatty acid collectors have been widely used in the flotation of magnesite. Zhu Yimin et al. [12] made a modified fatty acid collector DYM-1 and studied its effect on the buoyability of magnesite, dolomite and quartz. The results of single mineral flotation test show that the collecting ability of DYM-1 for three kinds of minerals is magnesite > dolomite > quartz, showing strong selectivity.

3.3. Magnesite Flotation Inhibitor

In recent years, many experts and scholars have devoted themselves to the research and development of new synthetic regulators, such as chelating agents (EDTA, etc.) produced by chelating various metal ions, which have stronger inhibition effect on magnesite than traditional regulators. Tang et al. carried out flotation separation tests of magnesite and dolomite and magnesite and calcite with sodium silicate as inhibitors, and the results showed that sodium silicate showed stronger inhibition on dolomite and calcite, but weaker inhibition on magnesite, with good selectivity. Zhong Wenxing et al. [13] explored the selective inhibition effect of polyacrylic acid in the reverse flotation separation of fine magnesite and quartz. Under the dodecylamine flotation system, polyacrylic acid can effectively improve the grade and recovery rate of magnesite, and reduce the entailing phenomenon of fine magnesite. According to the calculation of density functional theory, the adsorption energy between polyacrylic acid and magnesite is more negative than that between quartz, indicating that the adsorption effect between polyacrylic acid and magnesite is stronger, so polyacrylic acid can selectively adsorb on the surface of magnesite. YAO et al. [14] developed a new chelating agent, EDDHA, to separate magnesite and dolomite. The presence of EDDHA makes it easier to separate dolomite and magnesite in an alkaline environment. The metal chelates produced are stable and can strongly inhibit the floating of magnesite. Sun Haoran et al. used dodenylamine (DDA) as collector and monosodium phosphate (MSP) as regulator in the test of separating magnesite, dolomite and quartz. The presence of MSP makes DDA have similar flotation behavior for dolomite and quartz. The presence of MSP can effectively remove carbonate and silicate minerals in magnesite, and the hydrophobicity of dolomite mineral surface will increase under the action of DDA, but it has no effect on magnesite and quartz.

4. SUMMARY AND PROSPECT

4.1. Summary of Magnesite Separation Technology

The flotation process of magnesite depends on the ore properties. At present, the composite process of positive and negative flotation method of magnesite usually depends on the in-depth study of its mineral characteristics, and the specific application of positive and negative flotation process can usually be roughly divided into a single positive and negative flotation process and a combined process of reverse-forward flotation method. At present, the overall flotation process of magnesite tends to be mature. In practical application, the flotation flow should be formulated according to the ore properties.

4.2. Summary of Reagents for Magnesite Separation

Collector is the core factor to determine whether the target mineral has good hydrophobicity. Cationic collector (dodecylamine, ether amine, etc.) is commonly used in the flotation of magnesite to remove silica containing impurities such as quartz and talc, and fatty acid collector (sodium oleate, etc.) to remove calcium containing impurities such as dolomite and calcite. At present, the development and utilization of flotation reagents have been paid more and more attention, and the research on combination reagents has become more and more in-depth. The combination of different reagents can effectively improve concentrate grade and flotation efficiency, reduce the dosage of reagents and save production costs. The research on the combination of chemicals can also develop new beneficiation methods and promote the development of flotation.

4.3. Development Trend of Magnesite Separation Technology

4.3.1. It is inclined to the high efficiency purification of low grade magnesite resources and the development of flotation agents

In recent years, the reverse flotation purification of low-grade magnesite has gradually become a research hotspot. Some achievements have been made in the research and development of new reagents for reverse flotation, the improvement and optimization of existing reagents, the optimization of reverse flotation process, the pretreatment operation before flotation, the adsorption mechanism of reagents and minerals, etc., but there is still progress to be made in the application rate of new reverse flotation reagents and the improvement of fine mineral yield. It is necessary to reduce or eliminate the impact of inevitable ions on the reverse flotation effect, but also to reduce the negative impact of the interaction between various minerals.

4.3.2. It tends to reveal the mechanism of interaction between pharmaceutical molecules and mineral surface

One of the future trends of magnesite separation technology is to use modern detection and analysis methods, combined with theoretical knowledge and computational chemistry to clarify the nature of the interaction between amine collectors and different minerals. Based on the simulation calculation and model construction, the adsorption model of the interaction between amines and minerals was studied more deeply and extensively, so as to clarify the synergistic effect between ions and bond energy, and further reduce the problem of the foam viscosity of amines collector according to the ionic effect.

4.3.3. It is inclined to the structure-activity relationship and molecular design of amines

Traditional studies on the structure-activity relationship of amine collectors only consider the structure of the chemical molecules themselves, ignoring the interaction between the chemical molecules and the interaction surface of the water molecules and minerals in the actual flotation process. Although the simulation calculation based on the model can reflect the performance of the amine in the solution system to a certain extent, it cannot describe the microscopic mechanism of the interaction between the amine and the mineral and water interfaces. No accurate microstructure information can be provided. In the future, the research on chemical molecule design should provide suitable molecular simulation for different mineral interface systems, which is an important direction for the development of amine drugs.

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