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REMOVING IRON IMPURITIES FROM MAGNESITE ORE BY ASYNCHRONOUS REVERSE FLOTATION

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The main impurity elements in magnesite ore are silicon, calcium, and iron. In general, the cationic amine collector has good desilication by reserve flotation, and removing impurity of calcium and iron arouses the concern for their weak separation effect. The crude grade of the test sample was 92.48%, and the contents of SiO_2, CaO, and Fe_2O_3 were 1.65%, 1.11%, and 0.79%, respectively. At a yield of 71.22%, a concentrate with a grade of 97.16% and impurity contents of 0.18% SiO_2, 0.60% CaO and 0.49% Fe_2O_3 was obtained by 3 stages of reverse flotation using the KDL collector. The floated material was rejected as tailings, and the fraction that did not float offered the anionic reverse flotation. At a yield of 70%, a concentrate with a grade of 97.5% and impurity contents of 0.16% SiO_2, 0.55% CaO and 0.39% Fe_2O_3 was obtained by reverse flotation using the NaOL collector. Zeta potential and infrared spectroscopy analysis showed the water glass, sodium hexametaphosphate, and sodium oleate were chemically adsorbed on the magnesite, dolomite, and limonite. Besides, the physical adsorption occurred between 3 kinds of minerals and KDL.

Keywords: magnesite, reverse flotation, removing impurity, asynchronous reverse flotation

1. Introduction

China is rich in magnesite resources, accounting for about 1/4 of world reserves. The main impurities in the magnesite ore are silicon, calcium, and iron. Silicon-bearing minerals are usually talc, quartz, and chlorite, and the main calcium-bearing mineral is dolomite. Iron-bearing metallic minerals are mainly pyrite, pyrrhotite, magnetite, and hematite, and most of the iron sulfide minerals are changed into limonite after weathering. Refractory is the main product of magnesite. The iron in the magnesite ore destroys the quality of the refractory products. Thus, there are strict demands for the content of iron in the magnesite ore for refractory use. Although the high grade and fewer impurities of China's magnesite orebody, few high-quality mineral resources have been left with mining year after year. In the face of a lower and lower grade of raw magnesite ore, it is essential to removing iron impurity from magnesite. Since silicon and calcium are the impurities in the magnesite ore, singly removal of iron impurities can not satisfy the quality requirements of the magnesite concentrate. Therefore, the

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removal of silicon and calcium should be considered in the iron-removal process [1]. Depending on the type of ore deposit and the ore properties, different beneficiation processes are adopted for magnesite. Generally, the flotation method is used to separate the magnesite ore. Quartz or silicate minerals can be removed from the magnesite ore with amine collectors by reverse flotation for the selectivity between magnesite and silicon-bearing minerals. Separating dolomite from magnesite ore is under alkaline pH conditions, with fatty acid collectors by positive flotation (floating Mg and inhibiting Si and Ca). After the flowsheet of reverse flotation, the positive flotation is used to process the magnesite ore for most magnesite ores containing quartz/silicate and carbonate gangue minerals [2,3]. Wang Qianqian et al. [1] removed the Fe by different combinations of reserve flotation and high-intensity magnesite, and obtained the useful indexes.

Since the fatty acid collectors used in positive flotation need to be heated for separation, after reverse flotation, the positive flotation is performed, which depresses Ca and Si floating Mg after depressing Mg floating Si. The reagent consumption is significant because the magnesite is depressed and floated, and the floated magnesite in positive flotation is more than 96% of the feed. Therefore, there are some problems in the reverse-positive flotation process, such as relatively complex process conditions, high cost of beneficiation, reduced backwater utilization, and severe loss of magnesite resources. In the 1990s, the reverse-positive flotation was replaced by a single reverse flotation. Combined flowsheet of reserve flotation and high intensity magnetic has reduced application for its complex technology and the high price of intensity magnetic separator. The research on the removal of calcium and iron has become a hotspot in the industry with single reverse flotation.

Flotation separation of magnesite and dolomite with dodecyl phosphate [4-6] shows that the adsorption of dolomite is superior to that of magnesite due to phosphate in the collector. It proves the feasibility of separating magnesite and dolomite with dodecyl phosphate. Luo Na [7] studied the adsorption mechanism of sodium oleate (NaOL) on the surfaces of magnesite and dolomite. NaOL mainly exists in the ionic and molecular complexes in the slurry, and the adsorption with gauge minerals is mainly chemical adsorption [7]. The study of Yuan Shiquan shows that the concentration of Ca^{2+} and Mg^{2+} in the pulp is closely related to the surface electro-kinetic potential of magnesite/dolomite [7-9].

In this study, the asynchronous reversed the flotation process of removing the most silicon and dolomite impurities with cationic collector by reverse flotation. Then the iron impurities with anionic NaOL collector were removed by reverse flotation. The interaction of magnesite, dolomite, and limonite with the medicament was studied by the zeta-potential determination and infrared spectrum analysis.

2. Test materials and methods

2.1. Test samples

2.1.1. Magnesite ores

2.1.1.1. Mineral composition and content

The magnesite ore for the test was from Haicheng Magnesite Refractories General Factory. The samples were broken, sieved, mixed, and divided for chemical examination and testing. , The identification results of unbroken raw ore by a polarized microscope showed that the mineral composition of Haicheng magnesium ore was relatively complex with many kinds of minerals. The main mineral is magnesite with a content of 86.55%, followed by talc, brucite, quartz (opal), dolomite, and clinochlore, accounting for 4.83%, 4.13%, 1.53%, 1.50% and 1.25%, respectively. A small amount of metallic iron minerals referred to pyrite, pyrrhotite, magnetite, and hematite, and most of the iron sulfide minerals were changed into limonite after weathering. Talc and clinochlore were the easily slimed minerals in the ore (accounting for 5.38%), which affected the mineral processing.

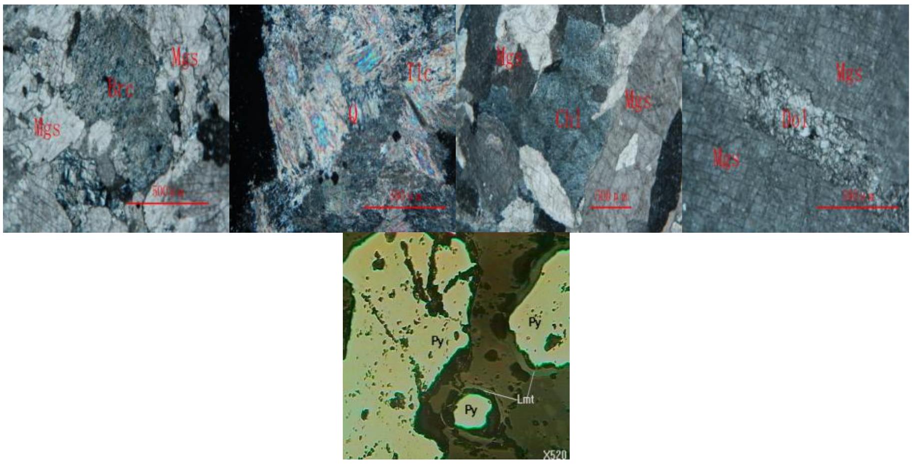


Fig. 1. Characteristics of mineral output
Mgs—magnesite, Tlc—Talc, Brc—brucite, Q—quartz (opal)
Dol—dolomite, Chl—Clinochlore, Py—pyrite, Lmt—limonite

2.1.1.2. Chemical composition analysis

The main chemical composition of silicon, calcium, and iron in the ore was analyzed (See Table 1).

Table 1

Chemical analysis of samples

Composition	IL	CaO	Fe_2O_3	Al_2O_3	SiO_2	MgO	MgO(IL=0)
Content/%	49.99	1.11	0.79	0.22	1.65	46.25	92.48

Table 1 shows the grade (MgO content% when a burned loss for 0) of crude is only 92.48%, and the contents of impurity silicon, calcium, and iron are high. Purification and impurity removal are necessary to satisfy the refractories.

2.1.2. Test of mineral sample

Haicheng Magnesite Refractory Material General Factory supplied single magnesite mineral. After being crushed and sorted by hand, it was ground and classified. The magnesite with a particle size of 0.045-0.124 mm was washed and dried for usage. The single dolomite mineral was selected from the Dolomite Mine of Yuxi Ma Steel in Rongcheng town. Some impurity particles were picked out by hand, then ground by vibration mill. The minerals with a particle size of 0.045-0.124 mm were washed three times and dried at low temperature for usage. The single limonite mineral was supplied by an iron mine in Hebei province, which was ground by vibration mill after being crushed and sorted by hand. The single limonite mineral with a particle size of 0.023-0.106 mm was washed and dried. Table 2 shows the main chemical compositions of magnesite, dolomite, and limonite, and Fig. 2 shows the X-ray pattern analysis.

Table 2

Chemical analysis results of a single mineral

Magnesite	Composition	MgO	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO(IL=0)	IL
	Content/%	47.35	0.32	0.12	0.32	0.19	98.03	51.70
Dolomite	Composition	MgO	CaO	SiO_2	Al_2O_3	Fe_2O_3	K_2O	CO_2
	Content/%	22.24	29.99	0.641	0.342	0.145	0.070	46.5
Limonite	Composition	Fe_2O_3	TiO_2	MgO	Al_2O_3	SiO_2	SO_3	P_2O_5
	Content/%	95.10	0.423	0.402	2.00	1.50	0.228	0.13

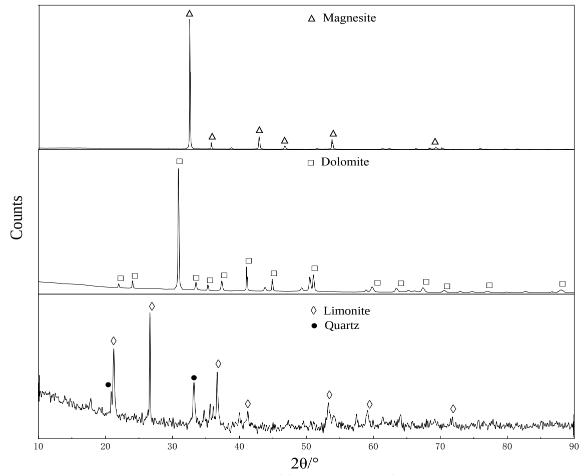


Fig. 2. X-ray diffraction of minerals

According to Table 2 and Fig. 2, the MgO content of the single magnesite mineral is 47.35%, and the MgO content of pure magnesite is 47.81%. The content of $MgCO_3$ in single magnesite is over 97%. The single dolomite mineral is mainly composed of dolomite, with a small number of impurity elements such as Fe, Si, and Al. The content of Fe_2O_3 in limonite single mineral samples is more than 95%, and there are more impurity elements except for iron. However, its content is less, and the purity of the single limonite mineral is high. Therefore, the magnesite, dolomite, and limonite can meet the requirements of a single mineral test.

2.2. Test Methods

2.2.1. Flotation test of magnesite ore

In the flotation test, use the 1 L hanging-cell-type XFD flotation machine, and add 400 g of mineral and 600 mL of water in the flotation cell. Add and stir the pH adjusting agent of slurry for 5 min, and handle the collector in the same way for 3 min at the stirring speed of 1,800 r/min. The floatation and scraping time was 4 min. Then the products were graded after being filtered, dried, and weighed. The content of impurity was analyzed for calculating the yield rate and removal rate of impurity.

2.2.2. Determination of zeta-potential

The mineral sample was ground by ZXM-1 vibration to -0.005 mm accounting for 100% by JS94H microelectrophoresis apparatus. Take a 250 mg ore sample into the 250 mL volumetric flask for usage. Then put 30 mL ore slurry into a beaker, and adjust the dosage of each reagent for measurement. The 0.5 mL slurry was placed in the electrophoresis tank, and each sample was measured 4-6 times in parallel. Finally, record the average value.

2.2.3 Determining infrared spectrum

The compression of KBr determined the infrared spectrum. After compressing, the sample was analyzed by the Nicolet 380 FT-IR spectrometer.

2.2.4 X-ray measurements

The sample was ground to -0.037 mm accounting for 100% before X-ray diffraction analysis. Then the sample was pressed into a plane on the slide and tested by an X-ray diffractometer at room temperature. Diffraction pattern analysis was according to the international diffraction data center (JCPDS-ICDD) in the PDF2-2004 card version.

3. Test Results and analysis

For the flotation of magnesite ore, the amine collectors were used in the reverse flotation, and the fatty acid collectors were used in the positive flotation

[10,11]. The collector KDL, consisting of two kinds of amines and two kinds of alcohol, was developed because of the weak removal effect of calcium and iron with the dodecyl amine as a collector by reserve flotation. The reserve flotation investigated the main variables of grinding fineness, pulp pH, adjustor dosage, and collector dosage with the KDL as a collector, followed by the reserve flotation with NaOL as a collector.

3.1 Investigation of grinding fineness investigation

Grinding fineness was an essential factor affecting the flotation index, which determines whether the valuable components were liberated. The increased grinding fineness can increase the liberation degree of mineral as well as the dosage of slime. The "over-grinding" was disadvantageous for the flotation effect. The effect of grinding fineness on flotation indexes was investigated under the conditions of pH *5.5*, water glass for 1500 g/t, sodium hexametaphosphate for 75 g/t by reserve flotation. Fig. 3 shows the indexes of concentrate.

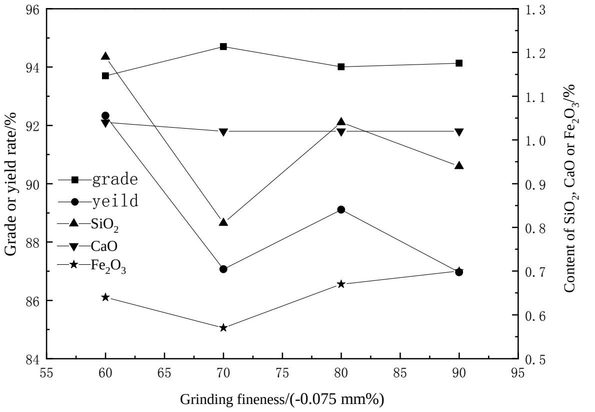


Fig. 3. Experimental results of grind fineness

In Fig. 3, grinding fineness has a significant influence on the recovery of concentrate and the removal effect of impurity. The suitable grinding fineness is -0.075 mm (accounting for 70%), and the results are consistent or similar to previous studies [12-15].

3.2 Investigation of pulp pH

The reverse flotation of removing silicon from magnesite ore was suitable in an acid condition. The pH value will influence the ionization of reagent in water and the solubility of minerals. It also affects the surface electric properties of minerals with the H^+ and OH^- as locating ions. The pH value was tested under the conditions of grinding fineness for -0.075 mm (accounting for 70%), the dosage of a water glass, sodium hexametaphosphate, KDL for 1,500, 150 and 150 g/t (75, 50, 25, calculated by amine quantity) separately by three times of reserve flotation. Fig. 4 shows the indexes of the concentrate.

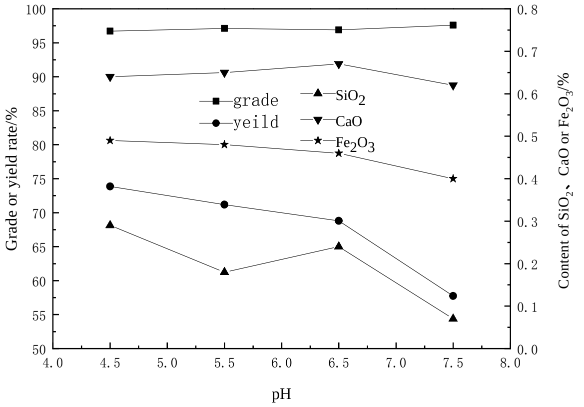


Fig. 4. Experimental results of pH

At pH 4.5, the concentrate yield rate is most significant, and grade is little; at pH 7.5, the concentrate grade is the biggest, with less yield. Indexes of concentrate grade and yield rate at pH 5.5 are better than those at pH 6.5. The optimal pH value is 5.5, and the results are consistent with previous studies [12-16].

3.3 Test of water glass dosage

The water glass was a joint adjusting agent in mineral flotation, which has the advantages of extensive source and low cost. Not only the selective *depressant of silicate* but also the dispersant reduced the influence of slime on flotation. There were many fine minerals in the magnesite ore, with the high distribution rate of excellent silicon, calcium, and iron mineral. The dosage of water glass was investigated on the conditions of grinding fineness of -0.075 mm (accounting for 70%), pH 5.5, the dosage of sodium hexametaphosphate of 150 g/t, the dosage of KDL (75, 50 and 25 g/t), respectively, by reverse flotation three times. Fig. 5 shows the indexes of the concentrate.

Fig. 5 shows that the dosage of water glass is 500-2,000 g/t, with the increased dosage of the water glass, the increased concentrate yield rate, the increased concentrate grade after the decrease. The contents of SiO_2, CaO, and Fe_2O_3 suffer the process of increase, decrease, and increase. The suitable dosage of water glass is 1,500 g/t.

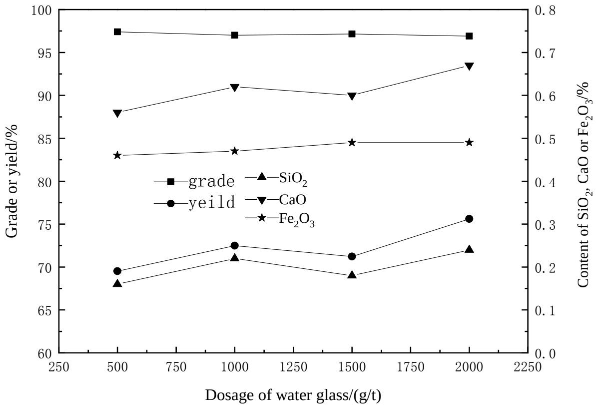


Fig. 5. Results of dosage of water glass

3.4 Test of sodium hexametaphosphate dosage

Sodium hexametaphosphate used as selective activators of gangue minerals can enhance the collection of silicate minerals and dolomite by collectors. On the other hand, the sodium hexametaphosphate, an inorganic ionic dispersant, will be adsorbed on the surface of silicate minerals, dolomite, and limonite when added to the pulp for ionization. As a result, the surface electronegativity of ore particles, as well as the electrostatic repulsion force between the ore particles, increases, and the particles tend to be suspended and dispersed. The interaction between collectors and gangue minerals increases with the increased removal rate of gangue minerals. Fig. 6 shows the indexes of concentrate of sodium hexametaphosphate dosage test.

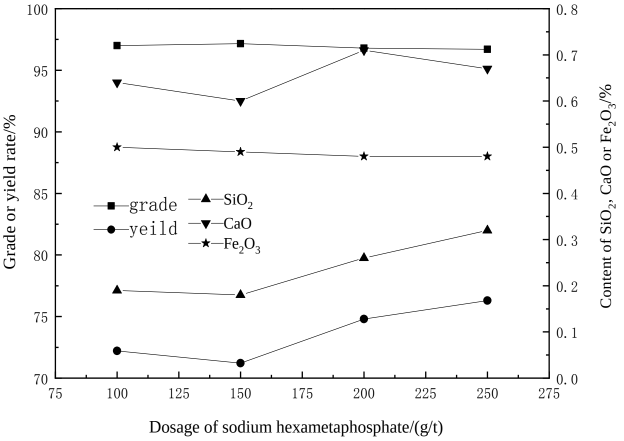


Fig. 6. Results of dosage of sodium hexametaphosphate

In Fig. 6, with the increased sodium hexametaphosphate dosage, the concentrate yield rate increases, and the concentrate grade decreases after an increase. When the dosage of Sodium hexametaphosphate is 0-150 g/t, the concentrate yield rate decreases slightly, and the grade increases gradually, which indicates the activation of sodium hexametaphosphate to gangue minerals. When the dosage of sodium hexametaphosphate is over 150 g/t, the concentrate yield rate increases with the decreased grade of concentrate, due to the excessive adsorption of sodium hexametaphosphate on the gangue minerals and the inhibition of gangue. There is more gangue entering magnesite concentrate, so the concentrate yield rate increases, and the concentrate grade decreases. Moreover, excessive sodium hexametaphosphate reduces the selectivity of flotation, which has a significant influence on flotation indexes. The optimal dosage of sodium hexametaphosphate is 150 g/t.

3.5 Test of KDL dosage

The contents of calcium and iron in crude were 1.11% and 0.79%, respectively. The removal effect of calcium and iron, as well as that of silicon, should be considered to ensure the quality of the concentrate. KDL made from the modified amine collector was used as the collector in this experiment to investigate the effect of its dosage on the flotation index, thus enhancing the removal of calcium and iron. Fig. 7 shows the indexes of concentrate.

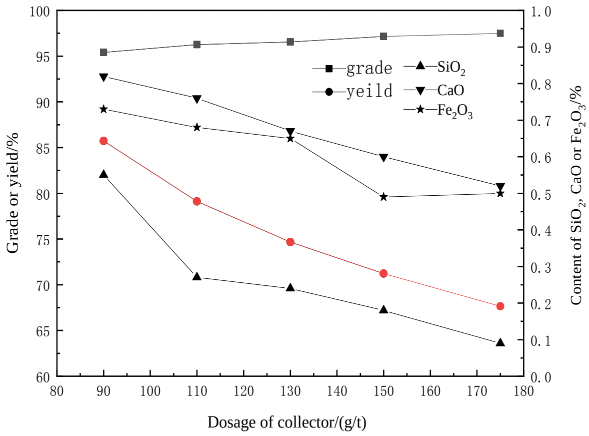


Fig.7. Results of dosage of KDL

In Fig. 7, with the increased KDL, the concentrate yield rate and the contents of impurities (CaO, SiO_2, and Fe_2O_3) in the concentrate decrease. When the collector KDL is 150 g/t, the concentrate yield rate is 71.22%; the contents of CaO, SiO_2, and Fe_2O_3 in the concentrate are 0.60%, 0.18%, and 0.49%, respectively; the concentrate grade is 97.16%.

3.6 Pulp pH test using NaOL as a collector by reverse flotation

In general, the content of SiO_2 lower than 0.25%, and the content of CaO of 0.5%-0.6% met the requirement of high-quality concentrate. In other words, it was better to have the lower contents of SiO_2 and CaO when CaO/SiO_2 was more than 2. The content of Fe_2O_3 lower than 0.40% met the requirement of high-quality concentrate, and the lower content of Fe_2O_3 was better.

The content of Fe_2O_3 in concentrate was still 0.49% after three times of reverse flotation using KDL as collect. Increasing the dosage of KDL decreased the content of Fe_2O_3 in concentrate, but the yield rate was low for poor selectivity of KDL. The effect of pH value on the removal of iron was investigated using the fraction that did not float in three times of reverse flotation as feed by reverse flotation with NaOL for 50 g/t as the collector. Fig. 8 shows the indexes of concentrate.

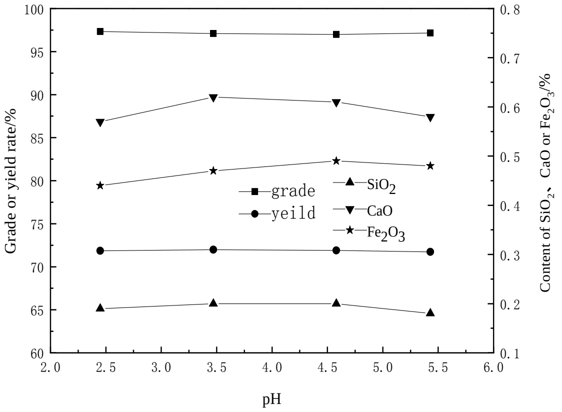


Fig. 8. Results of pH by reverse flotation with NaOL as the collector

In Fig. 8, the Fe_2O_3 content in concentrate can be reduced from 0.49% to 0.44% at pH 2.5. Meanwhile, the high-quality concentrate with grade (MgO content) above 97.3% is obtained with sodium oleate as a collector by the reverse floating concentrate of reverse flotation three times with KDL as the collector.

3.7 Test of NaOL dosage

The effect of NaOL dosage on flotation indexes was investigated at pH *2.5* by reserve flotation. Fig. 9 shows the effect of NaOL dosage on the concentrate index.

Fig. 9 shows NaOL increases, with increased concentrate grade, decreased concentrate yield rate, and decreased contents of impurities (CaO, SiO_2, and Fe_2O_3) in the concentrate. When the collector NaOL is 80 g/t, the concentrate yield rate is over 70%; the contents of SiO_2, CaO, and Fe_2O_3 in the concentrate are 0.16%, 0.55%, and 0.39%, respectively; the concentrate grade is over 97.5%.

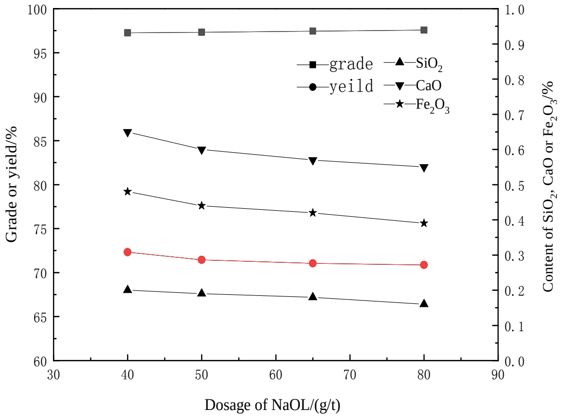


Fig. 9. Results of NaOL dosage

The isomorphism iron of in magnesite is about 0.27%, which cannot be removed in theory, so Fe_2O_3 has a good removal effect.

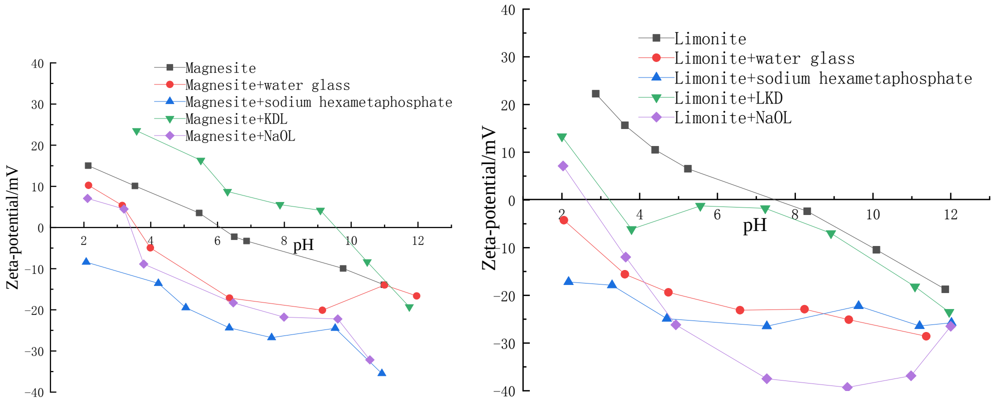
4. Mechanism of the interaction between the reagent and mineral surface

The effects of different reagents on single minerals of magnesite, dolomite, and limonite were studied by the surface electrical properties of minerals and infrared spectrum.

4.1. Zeta-potential analysis

Magnesite and dolomite were carbonate minerals, and the limonite was oxide mineral, with high solubility and dissolution rate in aqueous solution. The crystal lattice ions diffused into the medium under the action of water molecules, which caused the mineral surface to have a specific electric charge. In solution, the difference of affinity of water molecules to the surface anions and cations of mineral particles resulted in the selective dissolution of surface ions of minerals. The results showed that the adsorption capacity of these two ions on the mineral surface was different. These two aspects were the main reasons for the surface charge of minerals.

The 2 g magnesite and limonite were mixed with 30 mL distilled water to form the suspension. Then 200 mg/L water glass, 20 mg/L sodium hexametaphosphate, and 150 mg/L KDL and NaOL were added to the suspension, respectively. We determined the surface zeta-potential of magnesite, dolomite, and limonite with the reagent.



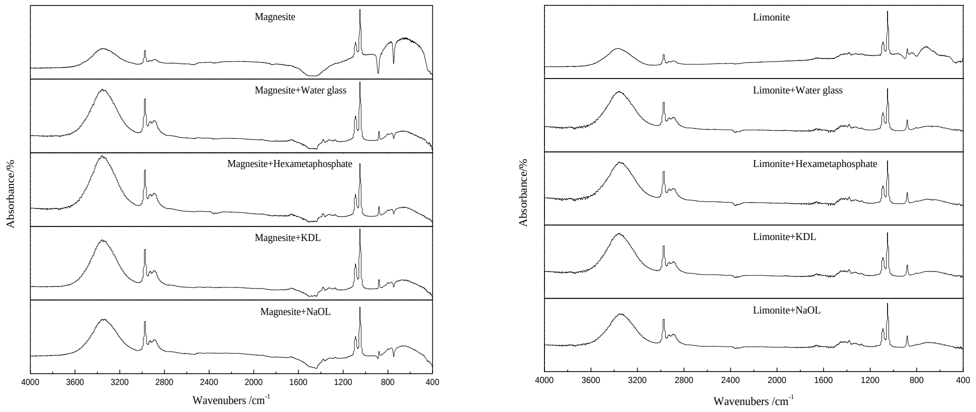
(a) (b)

Fig. 10. Zeta-potential of magnesite (a) and limonite (b)

Fig. 10 shows that the isoelectric points of magnesite, dolomite, and limonite are at pH 6.0 and 7.4, respectively. When the pH is higher than the isoelectric point, the mineral surface is negatively charged; when pH is lower than the isoelectric point, it is positively charged. The surface potential of minerals changes when they are adsorbed with water glass. The addition of water glass, having little effect on the surface potential of magnesite, can change the surface potential of limonite. It is beneficial to the flotation separation of limonite from magnesite with KDL as a collector. The electrostatic adsorption between ions of PO_4^{3-}, HPO_4^{2-} and $H_2PO_4^-$ being hydrolyzed by sodium hexametaphosphate in aqueous solution with minerals lead to the change of potential on the mineral surface. The decreased isoelectric points of magnesite and limonite with the addition of sodium hexametaphosphate indicate that the addition of sodium hexametaphosphate is beneficial to the flotation separation of limonite from magnesite with KDL as a collector. When the KDL is added, the potential of magnesite increases, but that of limonite decreases obviously, which is favorable for the flotation separation of limonite from magnesite by using the cationic collector.

4.2 Infrared spectrum analysis

2 g limonite was mixed with 30 mL distilled water to form the suspension, where 200 mg/L water glass, 50 mg/L sodium hexametaphosphate, 150 mg/L KDL, and NaOL were added, respectively. Hydrochloric acid adjusted the pH from 5 to 6. Stir the sample thoroughly in a blender. After washing the sample with distilled water three times, filter it with a vacuum filter and dry at room temperature. Fig. 11 shows the analysis of the infrared spectrum.



(a) (b)

Fig. 11. Infrared spectra analysis of magnesite (a) and limonite (b)

After the action of magnesite and water glass, the out-of-plane bending vibration peak of CO_3^{2-} moved from 886 cm^{-1} to 876 cm^{-1} and the asymmetric stretching vibration peak from 1,450 cm^{-1} to 1,460 cm^{-1}. After the action of limonite and water glass, there was no noticeable peak position change, which showed that the adsorption existed between water glass and magnesite, rather than between water glass and limonite. After magnesite reacted with sodium hexametaphosphate, the new peaks of 1,376 cm^{-1} and 1,273 cm^{-1} appeared, with the peak of 3,351 cm^{-1} stronger than that before the reaction with medicament. After the reaction with sodium hexametaphosphate, the characteristic peak of P-O-P in the sodium hexametaphosphate at 882 cm^{-1} and the peak of P-O stretching vibration at 1,207 cm^{-1} appeared in the spectrum of limonite. Therefore, the chemical adsorption occurred between sodium hexametaphosphate and minerals, but the adsorption between sodium hexametaphosphate with limonite was stronger than that with magnesite [17,18,19].

Before and after the reaction of magnesite and limonite with KDL, the spectrum had almost no change. KDL had no chemical adsorption with magnesite and limonite. According to the potential test, KDL significantly increased the potential of minerals, which was the reason for the electrostatic adsorption of RNH_3^+ and $(RNH_3)_2^{2+}$ on the mineral surface in the aqueous solution of amines. The characteristic absorption peak of COO- group in sodium oleate appeared at 1,372 cm^{-1} and the bending vibration absorption peak of C-H in sodium oleate at 882 cm^{-1} after the reaction of magnesite with NaOL. A new peak appeared at 2,920 cm^{-1} and 2,881 cm^{-1} on the infrared spectrum of limonite, which are the symmetric vibrational absorption peaks of the C-H bond in $-CH_2$-and-CH_3- [20].

In a word, the sodium Oleate adsorbed on the mineral surface partly in the form of ions and reacts with it to form calcium oleate (Fe and Mg). Furthermore, it partly adsorbed on the surface of dolomite (limonite, magnesite) in the form of the molecule. The results showed that the action of the minerals and sodium oleate refers to chemisorption and physical adsorption.

5. Conclusions

(1) The results of the flotation test of magnesite ore showed that the better removal effect was received with KDL as a collecter by reverse flotation three times. The contents of SiO_2, CaO, and Fe_2O_3 in concentrate were 0.18%, 0.60%, and 0.49%, respectively. Silicon and calcium had a good removal effect, and that of iron was not ideal. The asynchronous flotation of removing iron was tested with NaOL as a collector after removing the impurities by reserve flotation three times with KDL as a collector. Trough using asynchronous reverse flotation, the yield was higher than 70%, and the contents of impurities SiO_2, CaO, and Fe_2O_3 in the concentrate are 0.16%, 0.55%, and 0.39%, respectively. Besides, the concentrate grade was higher than 97.5% concentrate index. Concentrates with lower impurity and high-grade levels were more competitive due to the production of refractories or super-pure MgO.

(2) The zeta-potential and Infrared spectrum analysis showed that sodium silicate and sodium hexametaphosphate were chemically adsorbed on the surfaces of magnesite and limonite. The adsorption of sodium silicate and sodium hexametaphosphate on limonite was stronger than that on magnesite. The effect of the collector KDL on magnesite and limonite was physical adsorption, and the effect of NaOL on magnesite and limonite referred to both chemical and physical adsorptions.

Acknowledgments

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