Research on new beneficiation process of low-grade magnesite using vertical roller mill

Chuang Li1,2,4, Chuan-yao Sun2, Yu-lian Wang2,4, Ya-feng Fu1,5, Peng-yun Xu3, and Wan-zhong Yin1,5

1) School of Resources and Civil Engineering, Northeastern University, Shenyang 110819, China
2) State Key Laboratory of Mineral Processing, Beijing 102628, China
3) National Engineering Research Center of WEEE Recycling Engineering, Jingmen 448124, China
4) School of Materials Science and Engineering, Shenyang Ligong University, Shenyang 110819, China
5) College of Zijin Mining, Fuzhou University, Fuzhou 350108, China

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Abstract: In this study, we investigated whether the vertical roller mill can be efficiently used in the beneficiation of low-grade magnesite and whether it can improve upon the separation indices achieved by the ball mill. We conducted experiments involving the reverse flotation and positive flotation of low-grade magnesite to determine the optimum process parameters, and then performed closed-circuit beneficiation experiments using the vertical roller mill and ball mill. The results show that the optimum process parameters for the vertical roller mill are as follows: a grinding fineness of 81.6wt% of particles less than 0.074 mm, a dodecyl amine (DDA) dosage in magnesite reverse flotation of 100 g·t⁻¹, and dosages of Na₂CO₃, (NaPO₃)₆, and NaOL in the positive flotation section of 1000, 100, and 1000 g·t⁻¹, respectively. Compared with the ball mill, the use of the vertical mill in the beneficiation of low-grade magnesite resulted in a 1.28wt% increase in the concentrate grade of MgO and a 5.88% increase in the recovery of MgO. The results of our causation mechanism analysis show that a higher specific surface area and greater surface roughness are the main reasons for the better flotation performance of particles ground by the vertical roller mill in the beneficiation of low-grade magnesite.

Keywords: low-grade magnesite; vertical roller mill; reverse flotation; positive flotation; new beneficiation process

1. Introduction

Magnesite (MgCO₃), one of the world’s most important magnesium-containing minerals, is widely utilized in many fields such as metallurgy, aerospace, building and electronic industries, and manufacturing of basic refractories [1–2]. China holds the second largest proportion of the world’s magnesite resources, around 31%, and the industrial type of magnesite is comparatively complete, including original as well as secondary minerals. Magnesite resources in China are mainly distributed in the Liaoning and Shandong provinces and are characterized by comprehensive mineral types, a concentrated distribution, and huge deposits [3]. Although China is rich in magnesite resources, after more than 40 years of predatory mining, China’s natural commodity-grade magnesite has been exhausted, and high-grade magnesite is also becoming scarcer, which brings about the possibility of being unable to meet industrial needs. Meanwhile, since the reform and opening up of China in the 1980s, the reasonable development and utilization of magnesite in China has been overtaken by a one-sided pursuit of economic benefits. In particular, some enterprises selectively exploit rich ores and discard a large number of medium- and low-grade magnesite ores, which has resulted in the serious waste of magnesite resources [4]. To address the shortage of high-grade magnesite resources and avoid wasting low-grade magnesite resources, it is necessary to remove the silicon and improve the grade of magnesium in low-grade magnesite by mineral processing.

Currently, research on the desiliconization and magnesit-
um beneficiation of low-grade magnesite is concentrated on the flotation reagent [5], reagent system [6–7], and flotation process [8–12]. All of these excellent research efforts have proved that removing the silicon and improving the grade of magnesium in low-grade magnesite by mineral processing are beneficial for improving the utilization ratio of magnesite resources. However, there has been scant research on the beneficiation of low-grade magnesite with respect to grinding. Considering the recent progress in grinding technology and equipment [13–15], research on the beneficiation of low-grade magnesite with respect to grinding has become more important. In particular, good separation indexes have been obtained by the application of a vertical roller mill in mineral processing [16], which undoubtedly brings new ideas for research on the beneficiation of low-grade magnesite with respect to grinding.

The vertical roller mill mainly comprises systems for feeding, discharge, separation, grinding, and providing drive [17], as shown in Fig. 1. In the grinding process of the vertical roller mill, particles are transported to the center of the mill disc along a feeding pipe. The mill disc rotates at a constant speed to evenly disperse the particles outward by centrifugal force to form a particle bed with a certain thickness. Then, the particles on this bed are simultaneously crushed and ground by multiple rollers on the mill disc. By continuous centrifugal force, the particles continue to move toward the outer edge of the mill disc. The separation system controls the fineness of the finished products as they exit the roller. Particles larger than the specified size are separated and returned to the mill disc. These returned large particles are ground again while the particles that meet the fineness requirements are transported to the finished-product warehouse via the separation system. Thanks to this unique grinding principle, the vertical roller mill has the advantage of energy saving and the surface activities of its finished particles are stronger than those produced by other grinding methods.

To our knowledge, however, the use of the vertical roller mill in the beneficiation of low-grade magnesite has not yet been described in the literature. Therefore, here, we focus on the following two aspects: (1) verification of whether the vertical roller mill can be efficiently used in the beneficiation of low-grade magnesite and (2) verification of why the vertical roller mill can improve upon the separation indices achieved by the ball mill under the same separation conditions.

2. Materials and methods

2.1. Materials

The magnesite samples for our research were obtained from Dandong, Liaoning province, China. The chemical composition of the magnetite ore was as follows: 45.01wt% MgO, 2.43wt% CaO, 1.36wt% SiO₂, 0.16wt% Al₂O₃, and 0.32wt% Fe₂O₃. Based on the X-ray diffraction (XRD) patterns shown in Fig. 2, we can see that the useful mineral in the ore is magnesite and the main gangue minerals are quartz, talcum, dolomite, calcite, and other carbonate minerals. All the chemical reagents used in this work, including NaOL, dodecyl amine (DDA), Na₂CO₃, terpenic oil, (NaPO₃)₆, and sodium silicate, were chemically pure and purchased from China National Pharmaceutical Group Corp.

2.2. Experimental methods

2.2.1. Grinding experiments using vertical roller mill

We conducted the grinding experiments using a vertical roller mill in a closed-circuit experimental system designed by the Gebr-Pfeiffer (Germany). As shown in Fig. 3, this experimental system mainly comprises a material silo, hot-blast stove, vertical roller mill, cyclone, circulation fan, bag filter,
and finished-product warehouse. All of the grinding experiments were conducted using magnesite samples with particles smaller than 12 mm. Samples were first fed into the material silo and were then ground by the vertical roller mill under reasonable parameter conditions such as fan speed and degree of ventilation. Finally, the finished products produced from the vertical roller mill were gathered by the cyclones and bag filter and fed to the flotation experiment.

2.2.2. Grinding experiments using ball mill

For a comparison with vertical grinding, corresponding ball-grinding experiments were conducted according to the flow chart shown in Fig. 4. Similar to the vertical-roller-mill grinding experiments, the ball-mill grinding experiments were conducted using magnesite samples with particles smaller than 12 mm. First, the samples were fed into the roll crusher and crushed to < 3 mm. Then, these magnesite samples were delivered to the ball mill. By adjusting the grinding time of the ball mill, qualified samples were prepared and fed to the flotation experiment.

![Flow chart of the vertical-roller-mill grinding system](image)

**Fig. 3. Flow chart of the vertical-roller-mill grinding system.**

1—material silo; 2—vertical roller mill; 3—cyclone; 4—bag filter; 5—hot-blast stove; 6—circulation fan; 7—finished-product warehouse.

2.2.3. Flotation experiments

According to the functional approach used, the magnetite flotation experiments can be divided into the reverse flotation and positive flotation sections. All of the reverse and positive flotation experiments were conducted in the laboratory’s single XDF-0.75 flotation cell (Jilin Exploration Machinery Plant, China) at room temperature (25°C). The volume of this flotation cell is 750 mL and its impeller speed is set to 1992 r·min⁻¹. First, we purified the magnesite samples using reverse flotation and then further refined them using positive flotation, as shown in Fig. 5. We investigated factors related to the collector and frother in the reverse flotation section. Correspondingly, we investigated factors related to the regulator, inhibitor, and collector in the positive flotation section.

![Flow chart of the flotation test process](image)

**Fig. 5. Flow chart of the flotation test process.**

2.3. Characterization methods

2.3.1. Particle size distribution and specific surface area

The distribution frequencies of the particle sizes were measured and calculated by a BT-9300S laser particle analyzer (Dandong Bettersize, China). The mineral samples of magnesite were air-dried by an FZG-E ventilation dryer (Nanjing Buffalo Mechanical Equipment Co., Ltd., China) and then 5-g specimen powders were diffused by adding absolute alcohol. After the above steps were completed, we used a Scientz-1500F ultrasonic disperser (Zhengzhou Nanbei Instrument Co., Ltd., China) to prepare alcohol solutions of the sample powders for 15 min, after which they were moved into a laser particle-size analyzer for analysis. We measured the specific surface areas of the sample powders using a DBT-127 electric Brinell permeability surface area analyzer (Wuxi Jianyi Instrument & Machinery Co., Ltd., China) in accordance with the national standard GB/T 8074–2008 (China).

2.3.2. Scanning electron microscopy and atomic force microscopy

To observe the morphology of treated test samples, we used an EVO-18 scanning electron microscope (SEM, Carl Zeiss, Germany). To observe the three-dimensional morphologies of the sample powders and obtain more information about their surface topographies, we used a multimode-8 atomic force microscope (AFM, Bruker, Germany). For the microscopic observation test, we placed the specimen
powders on the test-bed in air atmosphere and then operated the probe in tapping mode. A more detailed description of surface roughness measurement by AFM can be found in the literature [18–20].

2.3.3. Adsorption measurements

To measure the amount of NaOL adsorbed by the collectors, we used UV spectrophotometry. According to the residual method [21–22], we plotted the working curve of the ultraviolet spectrophotometer (UV-2012, Unico, USA) by the adsorbance of sodium oleate at concentrations of 2, 4, 6, 8, 10, 12, and 20 mg L⁻¹, as shown in Fig. 6.

![Figure 6](image)

**Fig. 6.** Effect of NaOL concentration on absorbance.

We placed the samples (5 g) in 25 mL of distilled water, stirred for 1 min and adjusted the solution pH to the required value. Then we added a certain amount of NaOL to the solution according to the required dosage of the flotation test, stirred for 5 min, and allowed static settlement for 30 min. The supernatant of the solution was then suctioned out to measure the adsorbance using the ultraviolet spectrophotometer. Then, we calculated the residual concentration of the supernatant by the adsorbance and working curve. We calculated the amount of NaOL absorbed on the mineral particles based on the residual concentration using the following equation:

\[
\tau_m = \frac{(C_0 - C)V}{1000w}
\]

where \(C_0\) and \(C\) are the initial and supernatant concentrations of NaOL, respectively, \(V\) is the solution volume, \(w\) is the sample quality, and \(\tau_m\) is the amount of NaOL adsorbed on the mineral particles.

3. Results and discussion

3.1. Grinding fineness

The degree of liberation of valuable minerals is one of the most important factors affecting concentrate grade in the separation process [23]. The purpose of performing grinding before flotation is to liberate useful minerals from gangue minerals and to ensure that the size of the samples is suitable for flotation [24]. Therefore, the particle fineness achieved by vertical-roller-mill grinding should be a primary consideration. We determined the grinding fineness by the mass fraction of particles < 0.074 mm in the grinding product and compared the experimental results obtained in the vertical roller mill’s reverse flotation section. Fig. 7 shows the results of the reverse flotation experiments.

Fig. 7(a) shows that the grade and recovery of MgO in the reverse flotation concentrate first increase and then decrease with increases in the grinding fineness. Fig. 7(b) shows that the grade of SiO₂ in the reverse flotation concentrates increases steadily with increases in the vertical-roller-mill grinding fineness. Based on the results shown in Figs. 7(a) and 7(b), we can conclude that the optimum grinding fineness in the vertical roller mill is 81.6 wt% less than 0.074 mm.

3.2. Dosage of DDA for reverse flotation

As the most common collector used in silicate mineral
flotation, DDA can ionize the cation of R-NH$_3^+$, which contains a hydrophobic hydrocarbon group and an amidogen group [25]. Based on this unique structure of R-NH$_3^+$, DDA is highly selective in the flotation of silicate minerals [26]. Therefore, we used DDA as the collector for the desiliconization process in magnesite reverse flotation. We investigated the use of different dosages of DDA in reverse flotation by conducting flotation experiments, the results of which are shown in Fig. 8.

Fig. 8 shows that the recovery of MgO and the grade of SiO$_2$ in the reverse flotation concentrate decrease gradually with increases in the collector DDA, whereas the grade of MgO first increases and then ultimately decreases. Furthermore, when the dosage of DDA is 100 g·t$^{-1}$, the peak value of MgO grade is 46.72%. Obviously, increasing the dosage of DDA is beneficial in strengthening the process of desiliconization in magnesite reverse flotation. Based on the results shown in Figs. 8(a) and 8(b), we can conclude that the optimum dosage of DDA in magnesite reverse flotation is 100 g·t$^{-1}$.

3.3. Dosage of Na$_2$CO$_3$ for positive flotation

As a regulator, Na$_2$CO$_3$ is widely used in various mineral flotation processes due to its low cost. Normally, Na$_2$CO$_3$ plays two important roles in flotation processes [27–28]: (1) Na$_2$CO$_3$ can stabilize the pH value of pulp and (2) by removing unavoidable ions in the pulp, Na$_2$CO$_3$ reduces the influence of unavoidable ions in the flotation system. We investigated the use of different dosages of Na$_2$CO$_3$ by performing positive flotation experiments under optimum grinding fineness and DDA dosage, as described in Sections 3.1 and 3.2.

Specifically, we conducted positive flotation experiments following the reverse flotation experiments, the results of which are shown in Fig. 9.

As shown in Fig. 9 (a), the grade and recovery of MgO in positive flotation concentrate increase first and then decrease with the increase of Na$_2$CO$_3$ dosage, and finally reach the stable status. Similar trends were observed in the grades of SiO$_2$ and CaO in the positive flotation concentrate, as shown in Fig. 9(b). Based on the results shown in Figs. 9(a) and 9(b), we can conclude that the optimum dosage of Na$_2$CO$_3$ in magnesite positive flotation is 1000 g·t$^{-1}$. 

![Fig. 8](image_url) Effect of DDA dosage on the reverse flotation results: (a) grade and recovery of MgO; (b) grade of SiO$_2$ (Grinding fineness: 81.6%; terpenic oil: 20 g·t$^{-1}$).

![Fig. 9](image_url) Effect of Na$_2$CO$_3$ dosage on positive flotation: (a) grade and recovery of MgO; (b) grades of SiO$_2$ and CaO (NaOL: 800 g·t$^{-1}$; (NaPO$_4$)$_3$: 260 g·t$^{-1}$).
3.4. Dosage of sodium hexametaphosphate for positive flotation

Sodium hexametaphosphate (SHMP) can ionize the \((\text{Na}_4\text{P}_6\text{O}_{18})^{2−}\) cation in the pulp. By the chelation reaction between \((\text{Na}_4\text{P}_6\text{O}_{18})^{2−}\) and \(\text{Ca}^{2+}\), a stable hydrophilic chelate complex is formed on the surfaces of talc, quartz, and silicate [29]. Therefore, SHMP can be used as an inhibitor for tale, quartz, and silicate. SHMP can also disperse the slurry and reduce the influence of slime in the flotation process [30]. As such, we selected SHMP as the inhibitor and dispersant for magnesite positive flotation and investigated the dosage of SHMP in positive flotation experiments. Fig. 10 shows the results of the positive flotation experiments.

As shown in Fig. 10(a), the grade of MgO in the positive flotation concentrate first increases and then decreases with increases in the SHMP dosage. However, the recovery of MgO continues to decrease with increases in the SHMP dosage. Fig. 10(b) shows that the grades of SiO\(_2\) and CaO in the positive flotation concentrate decrease steadily with increases in the SHMP dosage. Based on the results shown in Figs. 10(a) and 10(b), we can conclude that the optimum dosage of SHMP in magnesite positive flotation is 100 g·t\(^{-1}\).

3.5. Dosage of NaOL for positive flotation

As an anion collector, NaOL is widely used in the flotation processes of oxidized ore due to its low cost and outstanding effect [31–32]. As such, we selected NaOL as the collector for magnesite positive flotation and we also investigated the dosage of NaOL in positive flotation experiments under the optimum grinding fineness and DDA dosage. Fig. 11 shows the results of the positive flotation experiments.

As shown in Fig. 11(a), the grade of MgO in the positive flotation concentrate first increases and then decreases with increases in the NaOL dosage. However, the recovery of MgO first increases and then remains stable with increases in the NaOL dosage. In Fig. 11(b), we can see that the grades of SiO\(_2\) and CaO in the positive flotation concentrate increase steadily with increases in the NaOL dosage. Based on the results shown in Figs. 11(a) and 11(b), we can conclude that the optimum dosage of NaOL in magnesite positive flotation is 1000 g·t\(^{-1}\).
3.6. Comparison of ball mill and vertical-roller-mill flotation processes of low-grade magnesite

To simulate the actual situation at an industrial beneficiation plant, we investigated the beneficiation of low-grade magnesite by a vertical roller mill in closed-circuit beneficiation experiments. We conducted these experiments under the optimal conditions determined in the experiments described above. Specifically, Table 1 lists the experimental conditions used in each procedure of the closed-circuit beneficiation experiments and Fig. 12 shows the experimental results.

In Fig. 12, we can see that the one-stage grinding process of the vertical roller mill–reverse flotation (one roughing and two scavenging)–positive flotation (one roughing and two cleaning) is suitable for the beneficiation of low-grade magnesite. When the feed grade of MgO is 43.08%, the beneficiation of low-grade magnesite by the vertical roller mill can obtain a concentrate with an MgO grade of 46.94% and a recovery of 76.02%.

To evaluate the performance of the vertical roller mill, we also studied the beneficiation of low-grade magnesite by the ball mill in a closed-circuit beneficiation experiment. We conducted this experiment using the same experimental methods and conditions as those used with the vertical roller mill. Fig. 13 shows the results of the closed-circuit beneficiation experiment using the ball mill.

In Fig. 13, we can see that the beneficiation of low-grade magnesite using a ball mill obtained a concentrate with an MgO grade of 45.66% and a recovery of 70.14% when the feed grade of MgO was 43.08%. Comparing the results shown in Figs. 12 and 13 we can see that the application of a vertical mill in the beneficiation of low-grade magnesite resulted in a 1.28% increase in the concentrate grade of MgO and a 5.88% increase in the recovery of MgO. Therefore, we can conclude that the vertical roller mill can improve the separation indices obtained using the ball mill under the same separation conditions.

4. Causation mechanism analysis

Although above experimental results confirm that the vertical roller mill can achieve better separation indices than the ball mill under the same separation conditions, we do not know why these improved separation indices are obtained, so the details of its causation mechanism must be clarified. Specifically, we need to characterize the microscopic physical and chemical characteristics of particles ground by the vertical roller mill and ball mill to explain the causation mechanism.

4.1. Particle size distribution and specific surface area

Previous studies [33–34] have shown that the adsorption of flotation reagents on the surface of powders is a crucial
factor in the flotation process. Thus, investigating the particle size distributions and specific surface areas of products obtained using different grinding methods can facilitate our understanding of the flotation results described above. Given the grinding fineness, Fig. 14 shows the influence of the grinding method used on the particle size distribution and specific surface area.

In Fig. 14, we can see that vertical-roller-mill products have a narrower size distribution than the ball-mill products when 81.6 wt% of the grinding products were smaller than 0.074 mm. The specific surface area of the powders ground by the vertical roller mill (235.5 m²·kg⁻¹) is higher than that for powders ground by the ball mill (191.6 m²·kg⁻¹) for a given grinding fineness. Researchers [35–36] have reported that a higher specific surface area and narrower particle size distribution are beneficial to the flotation of mineral particles. Based on those findings and our conclusions, we consider that the size distribution and the specific surface area of the products of the vertical roller mill are better than those of the ball mill.

4.2. Surface roughness

Although we can explain the causation mechanism of the improved flotation achieved by the vertical roller mill, we should not neglect the vital function of particle surface roughness in the flotation performances of particles [37]. In general, the function of high surface roughness in flotation involves two aspects: (1) higher surface roughness means a greater specific surface area, which increases the probability of adsorption by the flotation collector; (2) higher surface roughness increases the number of crystal sites exposed as “tip points,” which increases the surface energy of the powders and improves the adsorption ability of the collectors at the crystals sites [38–39]. As such, we used SEM to observe the surface morphologies of the particles. In addition, we used an AFM to observe the three-dimensional morphologies of the particles, and calculated the surface roughness of the particles based on the three-dimensional morphology data.

Fig. 15 shows images of the surface morphologies of the powders ground by the vertical roller mill and ball mill and Fig. 16 shows three-dimensional surface images of these
powders. By comparing the SEM images of the different grinding products, we can see that the surface roughness of the particles was greatly affected by the grinding patterns. As shown in Fig. 15, we know that the vertical-roller-mill products had a rougher surface than the ball-mill products. Furthermore, we found some micro-cracks in the vertical-roller-mill products, which explain why the specific surface area of vertical-roller-mill products is higher than that of the ball-mill products at the given grinding fineness.

To obtain surface roughness values, we statistically analyzed the altitude values determined by the AFM probes, the results of which are shown in Fig. 16. The surface roughness values of particles ground by the vertical roller mill and ball mill were 0.19093 and 0.10446 nm, respectively. These values are in good agreement with observations made based on the SEM images. Based on the results shown in Figs. 15 and 16, we can confirm that higher surface roughness is another reason for the better flotation performance of particles ground by the vertical roller mill in the beneficiation of low-grade magnesite.

4.3. Adsorption capacity

To further evaluate the flotation performance of particles obtained by different grinding methods, we measured and compared the adsorption capacities of products ground by the vertical roller mill and ball mill, the results of which are
shown in Fig. 17

In Fig. 17, we can see that vertical-roller-mill products have a stronger adsorption capacity than ball-mill products. This result is consistent with the research results presented in Sections 4.1 and 4.2. In summary, products ground by a vertical roller mill have a higher specific surface area and greater surface roughness, which can promote the adsorption capacity of the collector at the mineral surface. These findings and the reasoning described in Sections 4.1 and 4.2 demonstrate that a higher specific surface area and greater surface roughness are the fundamental reasons for the better flotation performance of vertical-roller-mill products in the beneficiation of low-grade magnesite.

5. Conclusions

(1) We conducted experiments to identify the optimum parameters to use in the beneficiation of low-grade magnesite. The results show that the optimum parameters are as follows: a vertical-roller-mill grinding fineness of 81.6wt% particles < 0.074 mm and a DDA dosage in magnesite reverse flotation of 100 g·t⁻¹. In the positive flotation section, the optimum dosages of Na₂CO₃, (NaPO₃)₆, and NaOL are 1000, 100, and 1000 g·t⁻¹, respectively.

(2) We conducted closed-circuit beneficiation experiments using a vertical roller mill under the above optimal conditions. The results showed that a one-stage grinding process by the vertical roller mill–reverse flotation (one roughing and two scavenging)–positive flotation (one roughing and two cleaning) is suitable for the beneficiation of low-grade magnesite.

(3) Compared to the ball mill, the application of a vertical mill in the beneficiation of low-grade magnesite resulted in a 1.28wt% increase in the concentrate grade of MgO and a 5.88% increase in the recovery of MgO. Therefore, we can conclude that the vertical roller mill is a more effective grinding tool than the ball mill when used in the beneficiation of low-grade magnesite.

(4) The products ground by a vertical roller mill were found to have a higher specific surface area and greater surface roughness, which are the fundamental reasons for their better flotation performance in the beneficiation of low-grade magnesite.

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