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## Effect of pulp temperature on separation of magnesite from dolomite in sodium oleate flotation system

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**Abstract:** The influence of pulp temperature on the floatability of magnesite and dolomite were studied by flotation test. Inductive Coupled Plasma Emission Spectrometer (ICP) was used to measure the dissolved metal ion content in the pulp by minerals in solution. X-ray photoelectron spectroscopy (XPS) was used to measure the presence and relative content of metal ions on mineral surfaces and the amount of sodium oleate adsorbed on mineral surfaces was measured by UV-Visible Spectrophotometer (UV-Vis). The results show that magnesite and dolomite have a great difference in flotation performance when the pulp temperature is 15 °C and the effective separation of magnesite from dolomite can be achieved. The main reason is that after the pulp is stirred at a pulp temperature of 15 °C and the pH of the pulp is adjusted with HCl and NaOH, the amount of metal ions remaining on the surface of the magnesite is much larger than that on the surface of the dolomite. Therefore, the active targets (metal ion) adsorbing oleate ions on the surface of the magnesite are more than that on the dolomite. When magnesite and dolomite coexist, oleic acid ion mainly acts on the surface of magnesite at the optimum temperature, which makes magnesite float up and the separation of magnesite from dolomite could be achieved.

**Keywords:** magnesite, dolomite, temperature, adsorption, flotation separation

### 1. Introduction

Magnesite ( $\text{MgCO}_3$ ) is the main source of magnesium oxide materials, which is widely used in metallurgy, construction, the chemical industry, and other fields (Karaoglu et al., 2016; Yao et al., 2016). Magnesite reserves in China are among the world's best, but due to over-exploitation, high-grade magnesite is gradually reduced. In addition, in many carbonate minerals, the magnesite comes out with other carbonate minerals that are similar in chemical structure and crystal structure, so their effective separation becomes a very important issue in the use of magnesite. Particularly, dolomite ( $\text{CaMg}(\text{CO}_3)_2$ ), a carbonate gangue minerals frequently seen in magnesite deposits, has a crystal structure and chemical characteristics very similar to magnesite (Luo et al., 2017; Marouf et al., 2009; Liu and Liu., 2004-a, Liu and Liu., 2004-b; Luo., et al., 2016). Therefore, in the process of magnesite flotation, reducing CaO content from dolomite is the key to improve magnesite quality, and the main calcium-containing gangue mineral in magnesite is dolomite (Anastassaki., 1999; Botero, et al., 2008).

Anionic collectors such as fatty acids and alkyl sulfate have been used in direct flotation (Florek, 1995; Brando and Poling, 1982). Oleic acid and sodium oleate can be widely used in the flotation of minerals as the collector. They were often used in the flotation of magnesite and dolomite. The high recoveries are due to chemisorption of sodium oleate on the magnesite and dolomite surface (Rao et al., 1990). The major cause is to reduce the amount of oleic acid adsorbed on the dolomite surface by metal ions

covering the dolomite surface and as the result, the decalcification is achieved. In recent years, many of the most recent experiments for separating magnesite from dolomite have been conducted in terms of regulators. Gence proposed the contact angles of magnesite and dolomite are clearly different when a certain concentration of sodium oleate and sodium silicate is present (Gence, 2006). Kangal's study showed that when the anion collector floats dolomite, the inhibition effect of sodium silicate is weaker than that of carboxymethyl cellulose (Kangal., 2005). Zhang Hao proposed monohydric alcohols can intensify dodecylamine adsorption on magnesite and dolomite surfaces (Zhang et al., 2018). However, many problems have yet to be uncovered in selective and effective separation of magnesite from dolomite (Elmahdy et al., 2007; Gence and Ozbay., 2006; Darius et al., 2018).

The crystal structure of the two minerals is similar, but there is a certain difference in dissolution characteristics and soluble species, and the behavior of the species in the solution and the effect of the species on the surface of the two minerals are also different. These complex actions are influenced by the pulp temperature, and thus the effect of selective separation of the two minerals is also different. Therefore, the influence of the pulp temperature on the selective separation of magnesite and dolomite in the flotation system of sodium oleate as a collector was investigated and made a reference for the separation of low-grade magnesite ore.

## 2. Materials and methods

### 2.1. Minerals and reagents

The magnesite and dolomite used in all experiments were taken from the Liaoning Province, China. The XRD analysis results of the mineral samples by an X'Pert Pro diffract are shown in Fig. 1. The compositions of magnesite and dolomite by ICP are listed in Table 1, which indicates that the purity of magnesite and dolomite are 96.18% and 99.03%, respectively. Mineral samples were dry ground and sorted by sieving, and the -74+38  $\mu\text{m}$  fraction was used for flotation experiments and some analyzes. The sodium oleate was used as a collector. NaOH and HCl were used to adjust pH. Deionized water was used for all of the experiments.

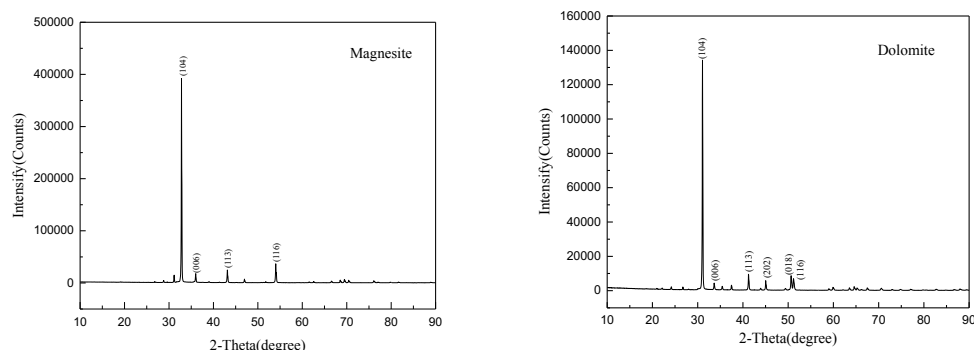


Fig. 1. The XRD analysis of mineral samples

Table 1. The chemical composition of minerals samples

Sample	CaO (%)	MgO (%)	SiO <sub>2</sub> (%)	Total Fe (%)	Al <sub>2</sub> O <sub>3</sub> (%)	Purity (%)
Magnesite	1.24	45.80	0.27	/	/	96.18
Dolomite	30.34	21.36	/	0.078	0.25	99.03

## 2.2. Experiment

### 2.2.1 Flotation tests

The flotation of a single mineral and the separation of artificially mixed samples were carried out in an XFG laboratory flotation cell (Jilin Exploration Machinery Plant, China). During all of the experiments, set the impeller speed to 1800 r/min. In order to control the temperature of the experiment, the

temperature of the laboratory was adjusted close to the experimental temperature by using the air conditioner first. In addition, the deionized water used in the experiment was adjusted to temperature in a constant temperature water bath. Because the experiment time was relatively short and there was no significant difference between the flotation cell and the ambient temperature, the temperature of cell did not change significantly during the experiment. The temperature of the flotation cells was measured with a thermometer at 1 minute intervals. By using geothermal heat in practice, it is possible to avoid seasonal influences and ensure stable temperature environment. The pulp for micro-flotation was prepared by adding 2 g of single mineral or samples mixed the two minerals at the rate of 1:1 and deionized water to 20 cm<sup>3</sup>. The reagent addition orders and the time of conditioning pulp are shown in Fig. 2. Both the froth products and the tailing were collected, filtered, and dried. The recoveries were calculated based on the weight of the products and the content of the elements in the obtained products was analyzed.

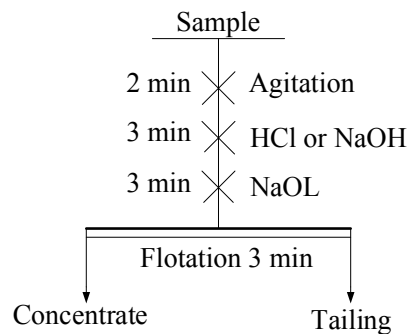


Fig. 2. The procedure of single mineral and mixed mineral flotation tests

### 2.2.2. Calcium and magnesium ion concentration measurements

The dissolved metal ions including Ca<sup>2+</sup> and Mg<sup>2+</sup> in solution were measured with Inductive Coupled Plasma Emission Spectrometer (ICP) model Prodigy XP.

According to the experimental procedure, 20 cm<sup>3</sup> of deionized water was added to 2 g of the sample, stirred for 2 min, and the pH was adjusted to 11 for 3 min. Then, the pulp was centrifuged and the supernatant was analyzed.

### 2.2.3. X-ray Photoelectron Spectroscopy (XPS) measurements

XPS measurements were carried out by an America Thermo VG ESCALAB 250Xi spectrometer using Al  $\alpha$  X-rays (1486.6 eV) as a sputtering source at a power of 150 W (15 kV 10 mA). The binding energy scale was corrected based on a C1s peak from contaminations (around 284.8 eV) as the internal binding energy standard (Zhu., et al., 2015). 20 cm<sup>3</sup> of deionized water is added to 2 g of the sample, and the pH is adjusted (3 min) after the solution is stirred for 2 min. Then the solid and liquid are separated and the solid portion is dried and analyzed.

### 2.2.4. Adsorption measurements

Ultraviolet spectrophotometry was performed to measure the amount of sodium oleate adsorption on the mineral surface. The absorbance of sodium oleate was obtained by using a UV1901PCUV-Vis spectrophotometer with a wavelength of 194 nm. The linear regression equation is obtained as  $y=0.0307x+0.0954$  ( $y$ -absorbance,  $x$ -sodium oleate concentration),  $R^2=0.9984$ . To ensure consistency in the flotation process, a 2 g of sample was mixed with 20 cm<sup>3</sup> of distilled water in a 30 cm<sup>3</sup> flotation cell and stirred for 2 min. The NaOH solution was added to maintain the pH at about 11. Then, the sodium oleate was added and the pulp was adjusted for 3 min. The pulp was placed for 3 min and then filtered. Only the supernatant was collected for UV spectrometry analysis. The residual concentration of sodium oleate was calculated based on the absorbance. From the residual concentration of oleic acid remaining in the solution, the amount of oleic acid adsorbed on the surface of the mineral is calculated as follows (Luo et al., 2016; Yin et al., 2019; Fu et al., 2018):

$$\Gamma = \frac{(c_0 - c)V}{W} \quad (1)$$

where  $\Gamma$  is the adsorption amount, mg/g;  $c_0$  is the initial concentration of sodium oleate in the solution, mg/L;  $c$  is the residual concentration of sodium oleate in the supernatant, mg/L;  $V$  is the solution volume, L; and  $W$  is the mineral weight, g.

### 3. Results and discussion

#### 3.1. Effect of pH and sodium oleate dosage on mineral flotation behavior

When the pulp temperature is set to 25°C and the sodium oleate dosage is 90 mg/L, the effect of pulp pH on the flotation recoveries of single minerals are shown in Fig. 3.

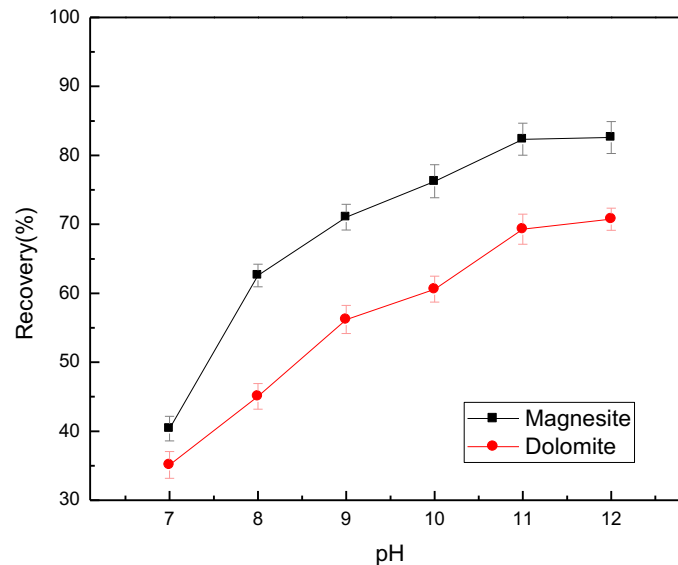


Fig. 3. Effect of pulp pH on flotation recoveries of single minerals

It can be seen from Fig. 3 that the flotation recoveries of single minerals increase with the increase of pulp pH value and the recovery of magnesite is higher than that of dolomite under the same flotation pH conditions. At pH 7-11, the recovery of magnesite increased from 40.40% to 82.35%, and the recovery of dolomite flotation increased from 35.10% to 69.30%. Thus, the recoveries of the two minerals increase linearly with increasing pH but stabilize at pH 11 or higher. Therefore, the flotation experiments for this study proceeded at pH 11.

In the condition of the pulp temperature of 25°C and pulp pH 11, the effect of NaOL dosage on the flotation recoveries of magnesite and dolomite is shown in Fig. 4. It can be seen from Fig. 4 that with the increase of sodium oleate dosage, the flotation recoveries of magnesite and dolomite are gradually increased, and under different conditions of sodium oleate dosage, the floatability of magnesite is greater than that of dolomite. In the sodium oleate dosage of 30 - 90 mg/L, the recovery of magnesite increases from 37.70% to 82.35%, and the flotation recovery of dolomite increases from 29.25% to 67.30%. It can be seen that the recoveries of the two minerals increase linearly with NaOL dosage at 30-90 mg/L. When the sodium oleate dosage is over 90 mg/L, the recoveries of the two minerals tend to be stable. Therefore, the dosage of sodium oleate 90 mg/L can be set as the best flotation collector dosage for the two minerals.

#### 3.2. Effect of pulp temperature on mineral flotation behavior

At the pulp pH value of 11 and the sodium oleate dosage of 90 mg/L, the effect of pulp pH temperature on the flotation recoveries of magnesite and dolomite is shown in Fig. 5. It shows that with the increase of pulp temperature, the flotation recoveries of magnesite and dolomite increase gradually, and the floatability of magnesite is higher than that of dolomite at different pulp temperatures. In particular, at the pulp temperature of 5 - 25°C, the flotation recovery of magnesite

increases from 48.75% to 82.35%. When the pulp temperature is over 25 °C, the recovery of magnesite flotation tends to be stable. For dolomite, at the pulp temperature of 5 - 15°C and 35 - 45°C, the flotation recovery relatively stable. And at the pulp temperature of 15 - 35°C, the difference in the recoveries of the two minerals with temperature becomes larger. Comparing the recoveries of the two minerals under different conditions, at the pulp temperature of 15 °C, the flotation recoveries of magnesite and dolomite are 70.35% and 15.15% respectively. In other words, the difference in the recoveries of the two minerals is greatest at the pulp temperature of 15°C. Therefore, the pulp temperature of 15°C can be set as the optimum condition of temperature for the flotation separation of the two minerals.

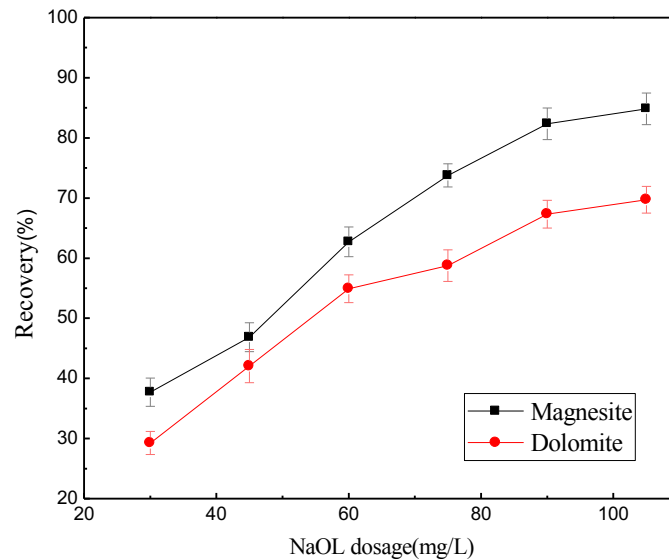


Fig. 4. Effect of sodium oleate dosage value on the recovery of mineral flotation

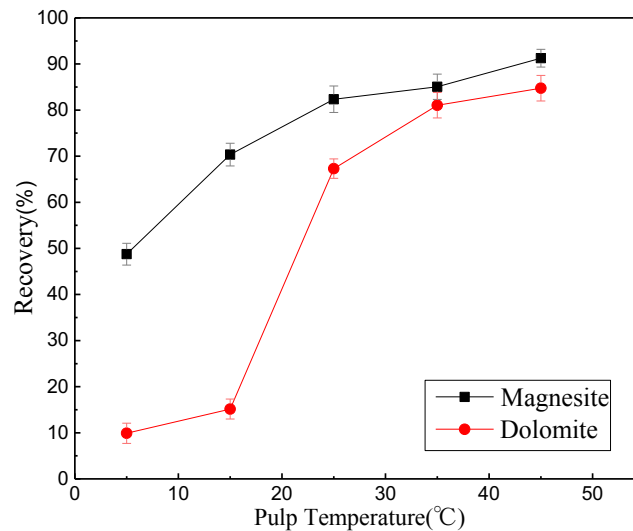


Fig. 5. Effect of pulp temperature on the recovery of mineral flotation

### 3.3. Micro-flotation test of the mixed minerals

From the single mineral flotation test, it can be concluded that the floatability of magnesite and dolomite differ greatly at the pulp temperature of 15°C. In order to verify the separation of magnesite and dolomite under this condition, a flotation experiment of artificially mixed sample of magnesite and dolomite was carried out.

In each experiment, 2g of sample is used, which is a mixture of two minerals at a ratio of 1: 1. The flotation test is carried out according to the single mineral test procedure and the flotation reagent system. The experiments for evaluating the effect of temperature on the flotation were carried out under conditions of pH 11 of the pulp and 90mg/L of the oleic acid dosage.

Table 2 and 3 show the flotation test results of the mixed minerals the pulp temperature of 25°C and 15°C, respectively. It can be seen from table 2 and 3 that as the pulp temperature decreases, the recovery of the flotation of both minerals also decreases. At the pulp temperature of 25°C, the flotation recoveries of magnesite and dolomite are 88.79% and 70.41%, respectively, which the difference is 18.38%. It indicates that the magnesite and dolomite are similar in floatability and cannot be effectively separated. When the pulp temperature is 15°C, the flotation recoveries of magnesite and dolomite are 65.78% and 10.42%, respectively, and the difference of them is 55.36%, indicating that there is a possibility of effectively separating the two minerals by using differences in the floatability of the two minerals depending on the pulp temperature.

Table 2. Results of the flotation test of mixed minerals at the pulp temperature of 25°C

Product	Yield/%	Grade/%		Recovery/wt. %	
		CaO	MgO	Magnesite	Dolomite
Concentrate	79.60	13.46	36.17	88.79	70.41
Tailings	20.4	22.04	28.84	11.21	29.59
Raw Ore	100.00	15.17	33.58	100.00	100.00

Table 3. Results of the flotation test of mixed minerals at the pulp temperature of 15°C

Product	Yield/%	Grade/%		Recovery/wt. %	
		CaO	MgO	Magnesite	Dolomite
Concentrate	38.10	4.16	42.27	65.78	10.42
Tailings	61.90	21.99	28.31	34.22	89.58
Raw Ore	100.00	15.17	33.58	100.00	100.00

### 3.4. Mechanism analysis of pulp temperature affecting the separation of magnesite from dolomite

Both magnesite and dolomite belong to carbonate minerals that are dissolved in water (Beenezeth., et al., 2009; Marta., et al., 2011). At different pulp temperatures, the results of the ion concentration measurement in solution after the pulp is stirred and the pH of the pulp is adjusted are shown as Fig. 6.

It can be seen from Fig. 6 that at the pulp temperature of 5 - 25°C, the concentration of metal ion  $Mg^{2+}$  in the solution of magnesite single mineral gradually increases with the increase of temperature, and at the temperature of 25°C it reaches to a maximum of 3.12 mg/L. When the temperature is over 25°C, the concentration of metal ions  $Mg^{2+}$  in the solution tends to be stable. For dolomite, the concentrations of metal ions  $Ca^{2+}$  and  $Mg^{2+}$  increase with the increase of temperature at 5~15°C, and the maximum values are 2.97 mg/L and 1.80 mg/L respectively. When the temperature is over 15°C, the concentrations of metal ions  $Ca^{2+}$  and  $Mg^{2+}$  tend to be stable, and the ratio of  $Ca^{2+}$  and  $Mg^{2+}$  dissolved from dolomite at different pulp temperatures is about 2 : 1. Comparing the metal ion concentration under different temperature conditions, it can be seen that when the pulp temperature is 15°C, the ratio of the metal ion concentrations dissolved from magnesite and dolomite has the largest difference as 1 : 4.72. It indicates that when the pulp temperature is 15°C, the metal ion concentration dissolved from the surface of dolomite is greater than that of magnesite, which causes the active target for adsorption of oleic acid on dolomite surface to be much less than magnesite. When the temperature is lowered in the mixed mineral flotation, the oleic acid ion mainly acts on the surface of the magnesite and it makes magnesite float up, which results in the possibility of separating the magnesite from the dolomite.

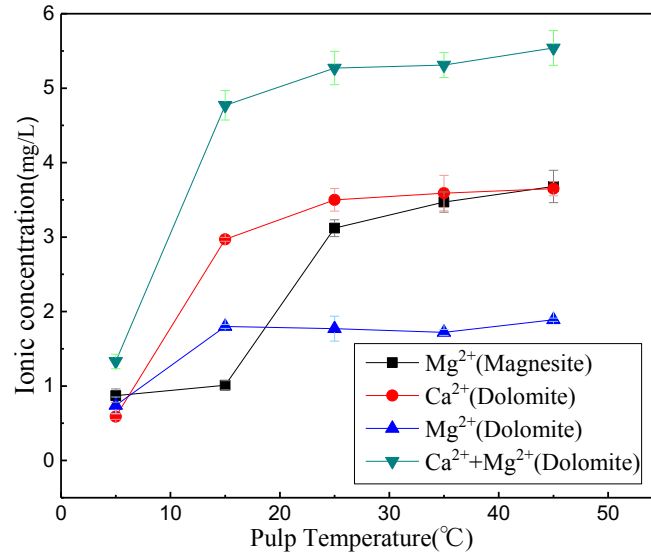


Fig. 6. Metal ion concentration of pulp solution

In order to detect the change of metal ion concentration on the mineral surface, X-ray photoelectron spectroscopy (XPS) analysis was carried out on the surface of the single mineral. The XPS test results of the raw ore sample are shown in Fig. 7. The single mineral XPS analysis results after the pulp are stirred and the pH of the pulp is adjusted, at the pulp temperature of 15°C are shown in Fig. 8. Table 4 shows the relative contents of atoms on the surface obtained by treating XPS analysis results of samples after controlling the temperature and raw samples using Thermo Advantage v5.976. Because the comparison between the treated and untreated samples is more important than theoretical calculation, the relative contents (at. %) for the distribution of atoms on the surfaces of raw ore samples were used as the standard.

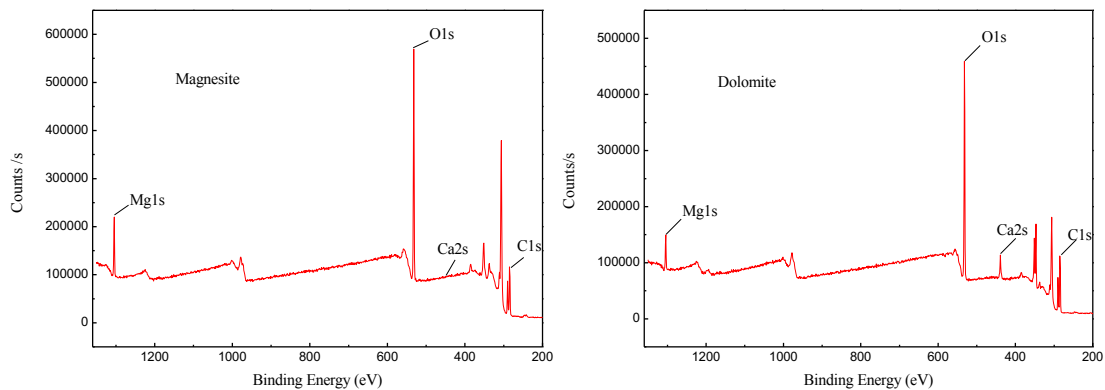


Fig. 7. Raw ore sample XPS analysis results

As can be seen from Table 4, for magnesite raw ore samples and the magnesite ore sample after pulping and pH adjustment at a pulp temperature of 15°C the relative total content of surface exposed metal cations was 7.20% and 6.58%, respectively, with the difference of 0.62%, and for dolomite 6.60% and 4.24%, respectively, with the difference of 2.36%. According to the data comparison, under the condition of pulp temperature of 15°C, after pulping and pH adjustment, the content of metal ions on the surface of magnesite and dolomite decreased. However, the content of metal ions remaining on the surface of dolomite is much lower than that of magnesite. It indicates that the dissolved metal ions on the surface of dolomite are more much than that on magnesite, which causes the active target for adsorption of oleic acid on dolomite surface to be much less than magnesite. Therefore, in the flotation pulp of the ore in which the two minerals coexist, oleic acid ions mainly act on the surface of the magnesite.

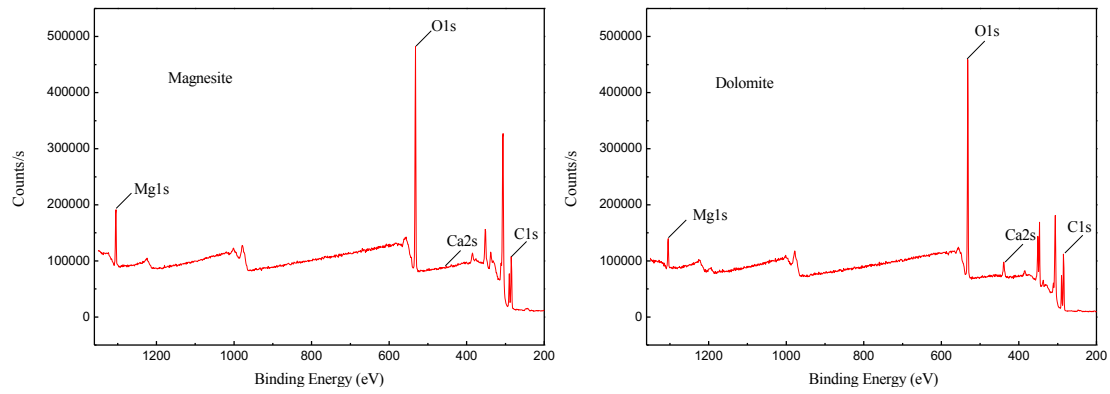


Fig. 8. Single mineral XPS analysis results after stirring the pulp and adjusting the pH of the pulp at a pulp temperature of 15°C

Table 4. Analysis of relative content of mineral surface elements

Mineral	Category	Surface Element	Binding Energy(eV)	Relative Content(at. %)	Surface Total Metal Cation Content(at. %)
Magnesite	Raw Ore	C1s	284.92	41.08	7.20
		O1s	532.10	51.72	
		Mg1s	1304.20	7.05	
		Ca2s	439.51	0.15	
	The sample after stirring the pulp and adjusting the pH of the pulp at a pulp temperature of 15 °C	C1s	285.00	42.43	6.58
		O1s	532.21	51.01	
		Mg1s	1304.31	6.37	
		Ca2s	439.62	0.21	
Dolomite	Raw Ore	C1s	284.93	43.95	6.60
		O1s	531.96	49.45	
		Mg1s	1304.20	3.89	
		Ca2s	439.22	2.71	
	The sample after stirring the pulp and adjusting the pH of the pulp at a pulp temperature of 15 °C	C1s	285.00	44.95	4.24
		O1s	532.01	50.81	
		Mg1s	1304.22	3.15	
		Ca2s	439.03	1.09	

In order to explore the effect of temperature on mineral adsorption of sodium oleate the adsorption measurements were carried out at different pulp temperatures, and the results are shown in Fig. 9. It can be seen from Fig. 9 that with the increase of pulp temperature, the adsorption amount of sodium oleate to magnesite and dolomite increases gradually, and the adsorption amount of sodium oleate on magnesite is higher than dolomite at different pulp temperatures. When the pulp temperature is 5 - 25°C, the adsorption capacity of magnesite to sodium oleate increases from 0.21 mg/g to 0.43 mg/g.



At the pulp temperature of over 25°C, the adsorption amount of magnesite to sodium oleate tends to be stable. For dolomite, the adsorption amount of sodium oleate is relatively stable when the pulp temperature is 5 - 15°C, and there is an increasing tendency at the pulp temperature of 15 - 45°C. Comparing the adsorption of the two minerals to sodium oleate under different conditions, when the pulp temperature is 15°C, the adsorption of magnesite and dolomite to sodium oleate are 0.37 mg/g and 0.09 mg/g, respectively, and this difference in adsorption amount is greater than that at other temperature conditions of this experiment.

As described above, the difference in adsorption amount of sodium oleate with temperature when magnesite and dolomite coexist shows the possibility of separation of two minerals by beneficiation.

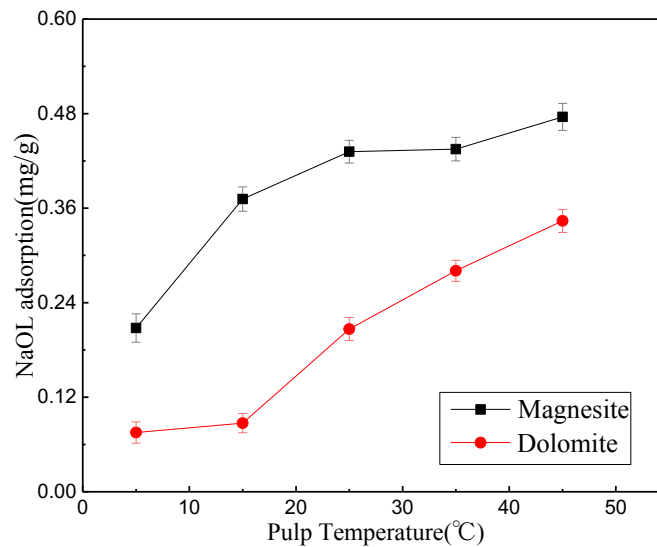


Fig. 9. Adsorption of sodium oleate by minerals at different pulp temperatures

#### 4. Conclusions

1. When the pulp temperature is 15°C, the difference in floatability between magnesite and dolomite is significant, and the separation of magnesite from dolomite can be achieved.

2. When the pulp temperature is 15°C, after pulping and adjusting the pH value, the metal ions dissolved on the surface of the magnesite are more than that on the dolomite and as a result, the active target of the oleic acid adsorbed on the surface of the magnesium salt is more than the dolomite.

3. Under the condition that the pulp temperature is 15°C, the adsorption capacity of magnesite to sodium oleate is much larger than that of dolomite. When magnesite and dolomite coexist, oleate ion mainly acts on the surface of magnesite.

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