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Article

Influence of Sodium Phosphate Salts with Different Chain Length on the Flotation Behavior of Magnesite and Dolomite

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Abstract: This paper analyzes the influence of sodium phosphate salts with different chain lengths as depressants on the flotation behavior of magnesite and dolomite through single mineral flotation test, contact angle test, and theoretical analysis. Flotation tests show that depressants should be added for the flotation separation of magnesite and dolomite. The inhibition of sodium phosphate salts on dolomite is significantly stronger than magnesite, and the flotation difference of minerals is affected by the chain length of phosphate depressants. The order of flotation separation enhancement of different sodium phosphate depressants is sodium hexametaphosphate ≈ sodium tetrapolyphosphate > sodium tripolyphosphate > sodium pyrophosphate. This result could also be supported by the contact angle measurement.

Keywords: magnesite; dolomite; phosphate; depressant; flotation

1. Introduction

Magnesite can be used to refine metallic magnesium, refractory materials, viscous materials, and magnesia chemical materials [1]. However, with the exploitation and utilization of resources, the grade of magnesite resources deteriorates, and the development of corresponding beneficiation technology is relatively lagging. Dolomite is a common type of associated gangue mineral in the flotation separation of magnesite [2]. Due to the similar crystal structure and surface property of the two minerals, the flotation separation is difficult, and the basic research on flotation is relatively inadequate [3,4].

In the system where magnesite and dolomite coexist, because dolomite dissolves in the solution to produce calcium ions and adsorbs on the surface of dolomite, the surface properties of the two minerals are similar, so flotation separation is difficult [5–9]. Modifying agents are important to improve the flotation selectivity, and they play the role of depressant agents or activators [10]. Modifying agents like carboxymethyl cellulose, sodium hexametaphosphate, sodium silicate, and sodium fluorosilicate have been used in the flotation separation of magnesite and dolomite [11–13]. Among them, sodium phosphate salts, which include sodium pyrophosphate, sodium tripolyphosphate, sodium tetrapolyphosphate, and sodium hexametaphosphate, have been developed as modifying agents for flotation of different target minerals. These sodium phosphate salts with different chain lengths have different surface-active properties in aqueous solutions, which will affect the flotation separation of magnesite and dolomite [14].

At present, the influence of sodium phosphate salts with different chain lengths on the flotation separation of magnesite and dolomite is not yet clear. Based on this consideration, this paper analyzes the influence of sodium phosphate salts with different chain lengths as depressants on the flotation separation of magnesite and dolomite through single mineral flotation test and contact angle test. Through the research in this article, the establishment of the relationship between sodium phosphate salts with different chain lengths and the flotation separation effect of magnesite and dolomite is of guiding significance for improving the grade of magnesite concentrate.

2. Materials and Methods

2.1. Minerals and Reagents

The pure mineral crystals of magnesite and dolomite were obtained from Liaoning area. The pure mineral preparation methods required for this test are manual crushing, grinding, and screening. The products under 0.106 mm fraction were collected as the microflotation test samples.

The X-ray diffraction results of magnesite and dolomite are shown in Figure 1a,b, respectively. The main chemical composition analysis of magnesite and dolomite is shown in Table 1. Comprehensive analysis of the test results shows that the purity of the prepared test sample is higher than 95%.

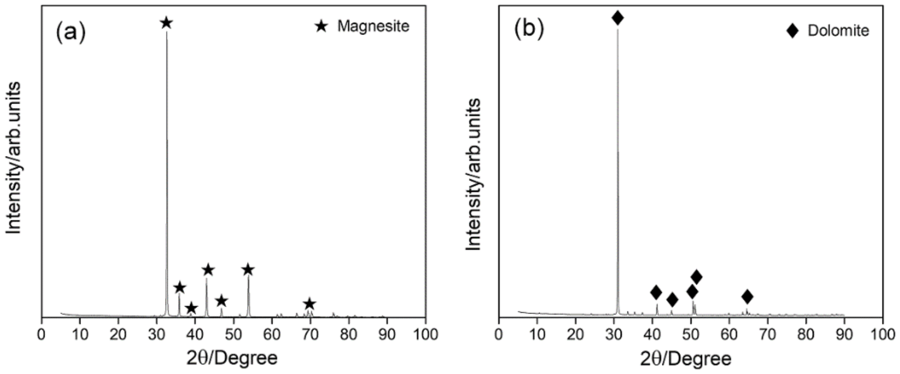

Figure 1. X-ray diffraction pattern of magnesite (**a**) and dolomite (**b**).

Table 1. Chemical composition of magnesite and dolomite.

Sample	MgO/%	CaO/%	SiO_2/%	Al_2O_3/%	FeO/%
Magnesite	47.20	0.50	0.62	<0.02	0.17
Dolomite	21.61	30.85	0.05	-	-

The sample was ground, then sieved, and the fraction of −0.106 mm was collected for microflotation test. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) were used as pH regulators. Sodium oleate was used as a collector. Sodium phosphate salts of different structures, such as sodium pyrophosphate ($Na_4P_2O_7$), sodium tripolyphosphate ($Na_5P_3O_{10}$), sodium tetrapolyphosphate ($Na_6P_4O_{13}$), and sodium hexametaphosphate ($Na_6P_6O_{18}$), were used as depressants. The reagents used in this study were all analytical grade. Deionized water was used in all experiments.

2.2. Flotation Tests

The flotation test was operated on an XFGC II flotation machine (Jilin Exploration Machinery Plant, Changchun, China), using a 40 mL cell volume. In total, 2.0 g ground ore sample with deionized water was placed into the flotation cell and then agitated under the speed of 2000 r/min for 1 min. The pH regulator, depressant, and collector were successively added into the flotation slurry. The stirring time of these added reagents was 2 min, and the flotation time was 4 min. The gas used in the microflotation unit was air. The flotation foam products and tailings were collected, and the dry mass percentage of foam products was calculated as the mineral recovery. Each test was measured three times, to obtain the average value. The flotation test flowsheet of pure minerals is shown in Figure 2.

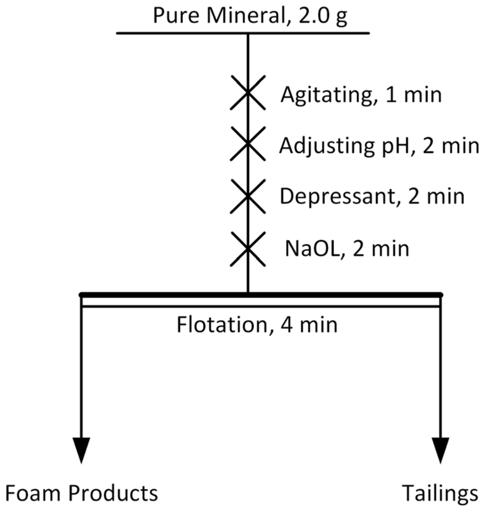

Figure 2. Flotation test flowsheet of pure minerals.

2.3. Contact Angle Measurements

The measurement of contact angle was carried out by using a JC2000DM contact angle tester (Shanghai Powereach Digital Technology Co., Ltd., Shanghai, China). The flotation concentrate with treatment of different depressants were respectively placed into the YP-15 tablet press (Tianjin, China), to obtain the test samples. The contact angles of prepared samples were measured by droplet method, using the analyzing software (Version 2.0, Shanghai Powereach Digital Technology Co., Ltd., Shanghai, China) of this JC2000DM contact angle tester. Each sample was measured three times, to obtain the average value.

2.4. Contact Angle Measurements

Zeta potential measurements of magnesite and dolomite with and without depressant were performed by using a Malvern Nano-ZS90 zeta potential analyzer (Malvern Panalytical, Malvern, UK). In total, 50 mg of mineral sample (−5 µm) was dispersed into 50 mL KCl background electrolyte solution (1×10^{-3} mol/L). The pH was adjusted by using NaOH or HCl solution. The supernatant was used for zeta potential measurements after 10 min conditioning. Each sample was measured three times, to obtain the average value.

3. Results and Discussion

3.1. The Effect of Sodium Oleate on Magnesite and Dolomite

Using sodium oleate as a collector, we carried out the flotation test of magnesite and dolomite under different concentrations of collector. The test result is shown in Figure 3. The test shows that, under the natural pH of 8.0, the recoveries of magnesite and dolomite increase with the increase of the collector concentration, and the recovery of magnesite is slightly higher than dolomite. As the sodium oleate has different collection abilities toward magnesite and dolomite, the flotation difference of the two minerals can be enlarged by adjusting the concentration of sodium oleate. When the concentrations of sodium oleate collector are 3×10^{-4} mol/L, the biggest differences in recoveries of magnesite and dolomite are obtained, which are 93.82% and 74.84%, respectively. This result indicates that magnesite and dolomite cannot be separated effectively by adding only collector.

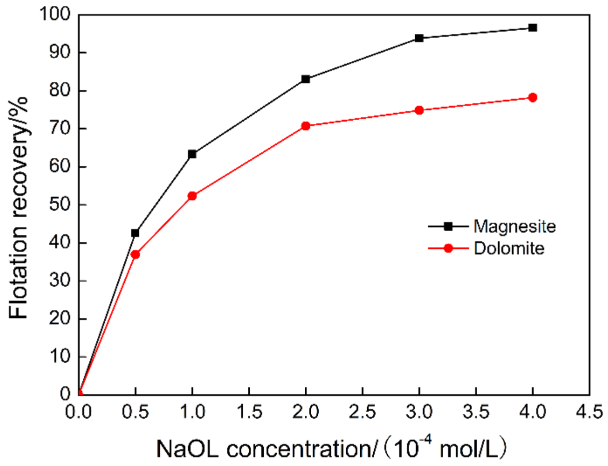

Figure 3. The effect of the concentration of collector on the flotation separation of magnesite and dolomite.

In the sodium oleate system, flotation tests of magnesite and dolomite were carried out under different pH conditions. The test results are shown in Figure 4. The test shows that the recoveries of magnesite and dolomite increases with the increase of pH, and the recovery of magnesite under the same pH condition is higher than that of dolomite. The recovery of magnesite increases first and then decreases slightly with the increase of pH, while the recovery of dolomite increases with the increase of pH. The test shows that the alkaline environment is more suitable for the flotation system with sodium oleate as a collector. Moreover, sodium oleate has a certain collection effect on magnesite and dolomite under alkaline environment. However, the flotation recoveries of the two minerals are not much different, as the difference in recoveries of magnesite and dolomite should be enlarged further, and the recovery of dolomite should be reduced to a certain value at the same time, in order to achieve the effective flotation separation and obtain the magnesite concentrate with high purity. Furthermore, appropriate depressants need to be added into this flotation system for separation of magnesite and dolomite.

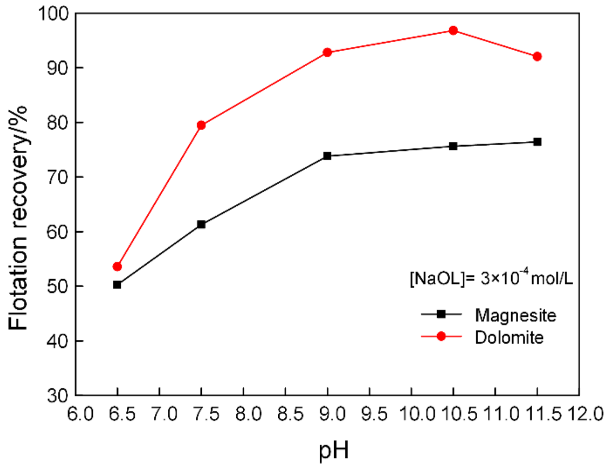

Figure 4. The influence of slurry pH on the flotation separation of magnesite and dolomite.

3.2. Influence of Sodium Phosphate Salts with Different Chain Length on the Flotation Separation of Magnesite and Dolomite

At natural pH and with sodium oleate as a collector, the flotation separation of magnesite and dolomite was investigated with different depressant concentration; the result is shown in Figure 5. Figure 5a shows that, in a flotation system with sodium oleate as a collector and sodium pyrophosphate as a depressant, the recovery of the flotation concentrate of magnesite decreases slowly with the increase in the concentration of sodium pyrophosphate. Meanwhile, the flotation recovery of dolomite decreases slowly. In the same environment, the inhibition of sodium pyrophosphate on dolomite is slightly stronger than that of magnesite. When sodium pyrophosphate was used as a depressant, as shown in Figure 5b, the two minerals showed nearly the same trend as sodium pyrophosphate, but the difference in the floatability of the two minerals was enhanced. From Figure 5c, it can be seen that the flotation recovery of magnesite decreases slowly with the increase in the concentration of sodium tetrapolyphosphate, and the flotation recovery of dolomite decreases rapidly. The inhibition of sodium tetrapolyphosphate for magnesite is weak, and the inhibition of dolomite is stronger. As shown in Figure 5d, the flotation recoveries of magnesite and dolomite decrease rapidly as the concentration of sodium hexametaphosphate increases. Moreover, the sodium hexametaphosphate shows strong inhibition on both minerals. The biggest difference in the floatability can be obtained at the concentration of 300, 200, 100, and 4 mg/L for sodium pyrophosphate, sodium tripolyphosphate, sodium tetrapolyphosphate, and sodium hexametaphosphate, respectively. It can be indicated that the concentration of depressant required decreases as the phosphate chain length increases.

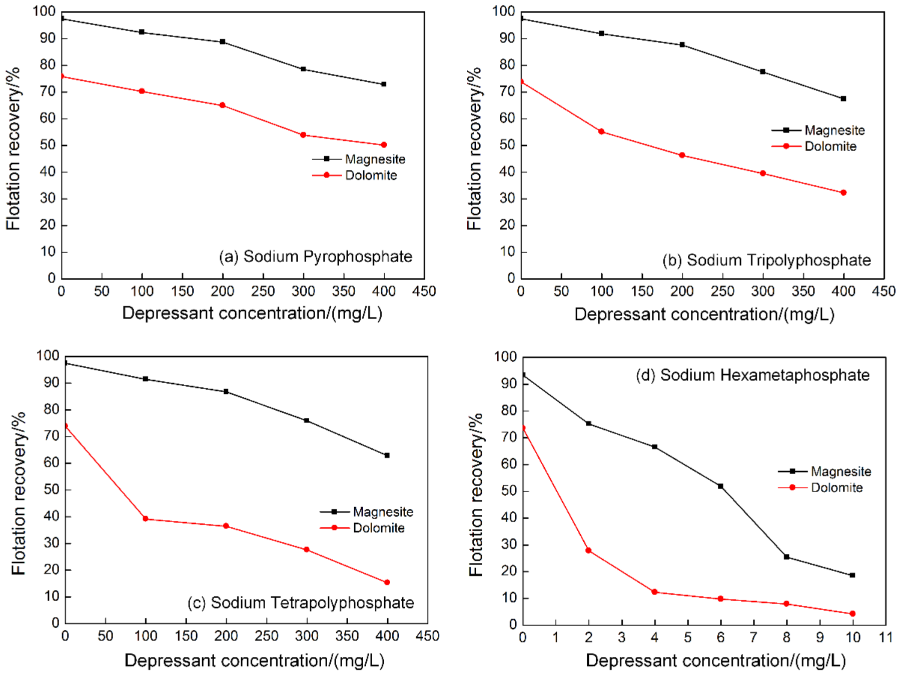

Figure 5. The influence of the concentration of phosphate with different chain lengths on the recovery of mineral flotation. Sodium Oleate 3×10^{-4} mol/L. (**a**) Sodium Pyrophosphate, (**b**) Sodium Tripolyphosphate, (**c**) Sodium Tetrapolyphosphate, (**d**) Sodium Hexametaphosphate.

Using sodium oleate as a collector and sodium phosphate salts with different chain lengths as depressants, we performed flotation and separation of magnesite and dolomite under different pH conditions. The test results are shown in Figure 6. The results show that the difference in the floatability of the two minerals is enhanced under alkaline environment. The floatability differences with the sodium tetrapolyphosphate and sodium hexametaphosphate as depressants are significantly bigger than other two depressants. The biggest difference in the floatability can be obtained at the pH of 11.5, 10.0, 8.0, and 10.0 for sodium pyrophosphate, sodium tripolyphosphate, sodium tetrapolyphosphate, and sodium hexametaphosphate, respectively.

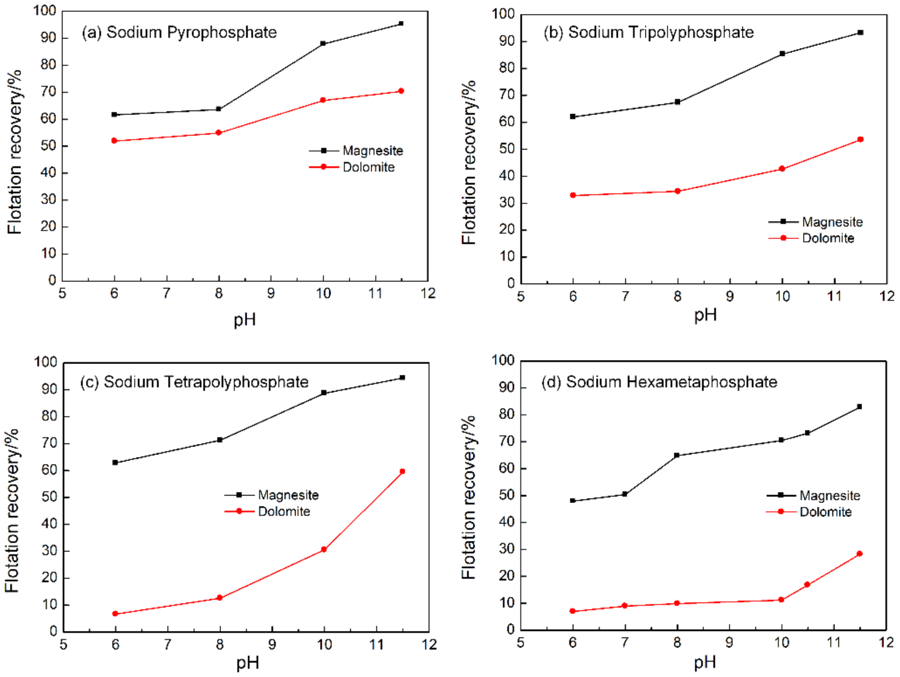

Figure 6. The effect of pH on mineral flotation recovery with different depressants. Sodium Oleate 3×10^{-4} mol/L. (**a**) Sodium Pyrophosphate 300 mg/L, (**b**) Sodium Tripolyphosphate 200 mg/L, (**c**) Sodium Tetrapolyphosphate 100 mg/L, (**d**) Sodium Hexametaphosphate 4 mg/L.

The contact angles of magnesite and dolomite were measured under different chain lengths of phosphate as depressant and sodium oleate as collector. The results are shown in Figure 7. It can be seen from Figure 7a that, in the system of sodium pyrophosphate and sodium oleate, the contact angle between magnesite and dolomite decreases with time, and there is little difference in the contact angle between magnesite and dolomite. The increase tends to be the same. The flotation test shows that sodium pyrophosphate has a certain inhibition on magnesite and dolomite, but the inhibition is small, that is, sodium pyrophosphate has poor selectivity. Under the system of sodium tripolyphosphate and sodium oleate, as shown in Figure 7b, the contact angle between magnesite and dolomite decreases with time, and the contact angle difference between magnesite and dolomite is small, implying the selectivity is poor and the flotation separation cannot be achieved effectively. Under the effect of sodium tetrapolyphosphate and sodium hexametaphosphate, as shown in Figure 7c,d, the contact angle difference is enhanced, and the selectivity will be improved.

Figure 8 shows the effect of phosphate chain length on the separation selectivity of magnesite and dolomite. The sodium phosphate salts have inhibition on both of magnesite and dolomite. Moreover, the inhibition becomes stronger with the increasing phosphate chain length. Under the effects of sodium pyrophosphate, sodium tripolyphosphate, sodium tetrapolyphosphate, and sodium hexametaphosphate, the recoveries of magnesite are 95.32%, 85.32%, 71.23%, and 70.51%, respectively. The recoveries of dolomite are 70.33%, 42.68%, 12.56%, and 11.17%, respectively. The recovery differences between magnesite and dolomite are 24.99%, 42.64%, 58.67%, and 59.34%, respectively, indicating that the separation selectivity of magnesite and dolomite is enhanced with the increasing phosphate chain length. Effective separation of the two minerals cannot be achieved by the effects of sodium pyrophosphate and sodium tripolyphosphate. Sodium tetrapolyphosphate and sodium hexametaphosphate are the helpful depressants for the separation selectivity of magnesite and dolomite. It should be noted that the recoveries of magnesite and dolomite under the effects of sodium tetrapolyphosphate and sodium hexametaphosphate are nearly the same, indicating the same flotation separation enhancement.

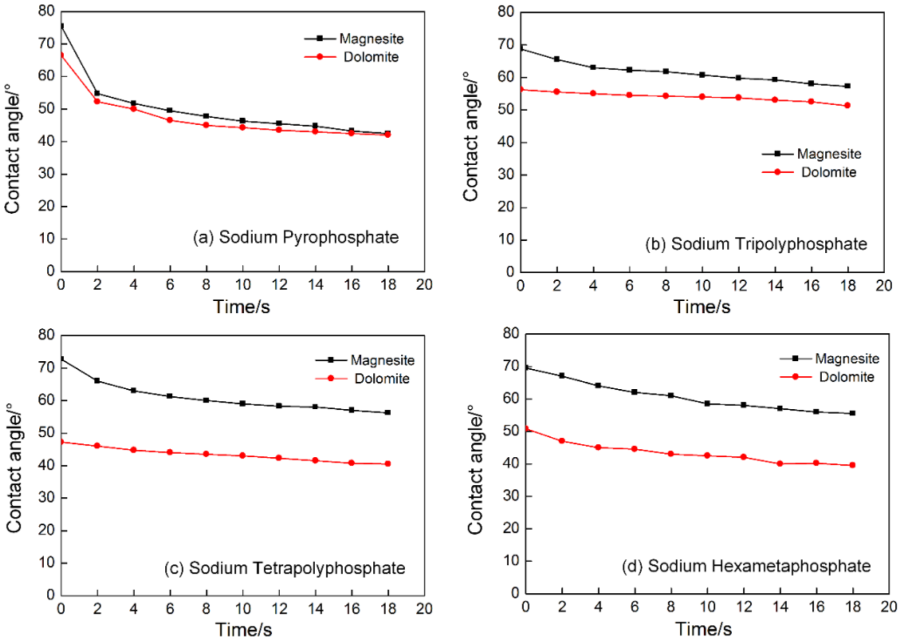

Figure 7. The relationship between contact angle and time under the effect of the sodium phosphate salts. Sodium Oleate 3×10^{-4} mol/L. (**a**) Sodium Pyrophosphate 300 mg/L, (**b**) Sodium Tripolyphosphate 200 mg/L, (**c**) Sodium Tetrapolyphosphate 100 mg/L, (**d**) Sodium Hexametaphosphate 4 mg/L.

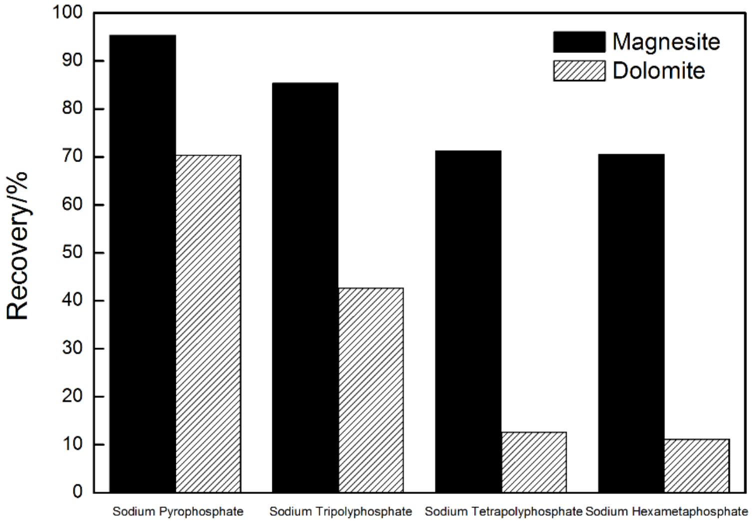

Figure 8. The influence of phosphate chain length on flotation selectivity.

The zeta potential measurement results for magnesite and dolomite with and without treatment of sodium tripolyphosphate are shown in Figure 9. It can be seen from Figure 9 that the isoelectric points of magnesite and dolomite are around pH 6.0, which is in agreement with the previous report [2]. When the magnesite and dolomite are treated with the sodium tripolyphosphate of 200 mg/L, the zeta potentials of them decrease with the increasing pH. Moreover, the zeta potential of dolomite decreases more significantly, as compared to magnesite, implying that the sodium tripolyphosphate has a higher inhibition on dolomite than magnesite. It should be mentioned that the zeta potentials of magnesite and dolomite exhibit nearly the same trend when they are treated with the sodium tetrapolyphosphate and sodium hexametaphosphate, respectively.

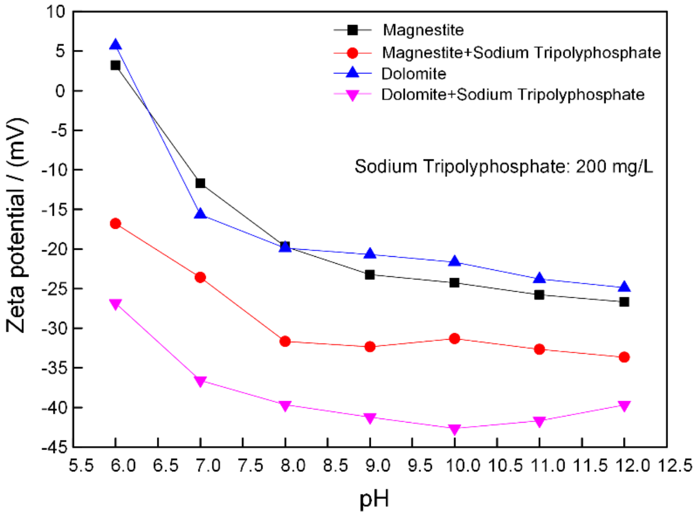

Figure 9. Zeta potential measurements for magnesite and dolomite with and without treatment of sodium tripolyphosphate.

In general, the inhibition of sodium phosphate salts on dolomite is significantly stronger than that of magnesite, and the floatability difference of two minerals are affected by the chain length of sodium phosphate depressants. The order of flotation separation enhancement of different sodium phosphate depressants is sodium hexametaphosphate ≈ sodium tetrapolyphosphate > sodium tripolyphosphate > sodium pyrophosphate. Using sodium hexametaphosphate or sodium tetrapolyphosphate as flotation depressant can achieve the effective separation of magnesite and dolomite.

3.3. The Inhibition Mechanism of Sodium Phosphate Salts

Magnesite and dolomite have similar surface characteristic and flotation behavior [15,16]. Based on the cleavage properties of magnesite and dolomite, the structure diagram of the most common cleavage surface (104) of the two minerals is obtained, as shown in Figure 10. It can be seen from Figure 10 that the spatial structures of the cleavage surfaces of the two minerals are very similar. Table 2 shows the relevant parameters of the metal sites on the (104) planes of magnesite and dolomite. It can be concluded that the metal sites of the two minerals are very small, and the number of broken bonds is the same, indicating that the difference in the crystal chemistry of the two minerals is mainly reflected in the surface Ca and Mg activity.

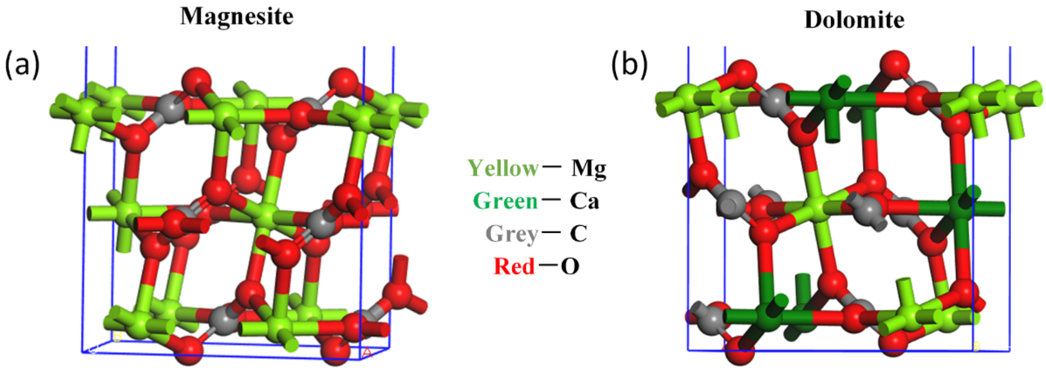

Figure 10. Structure diagram of common crystal cleavage planes of (**a**) magnesite and (**b**) dolomite.

Table 2. Metal site properties of (104) planes of magnesite and dolomite.

Minerals	Metal Site Density/nm^2	Number of Broken Bonds at Metal Site
Magnesite (104)	Mg:5.70	2
Dolomite (104)	Ca: 2.704; Mg: 2.704	2

Phosphate interacts with Ca or Mg through ionic bonds, and the action strength is related to the electronegativity difference between the phosphate group and Ca (or Mg) [17]. Among them, the electronegativity of Ca and Mg are 1.0 and 1.2, respectively. Moreover, the electronegativity of the phosphate group is greater than that of O (3.5). Therefore, the electronegativity difference between phosphate and Ca is greater than that of Mg. The strength of the interaction between phosphate and Ca is stronger than that of Mg, and this result is also consistent with the solubility product data [18].

The surface electrical properties and crystal structure (spatial arrangement and size of sites) of magnesite and dolomite are similar [19,20]. Thus, using collector alone cannot effectively separate magnesite and dolomite in the flotation. The active site of magnesite is magnesium ions, and the active site of dolomite is calcium ions [21,22]. Since phosphate and calcium ions interact stronger than magnesium ions, phosphate is a good depressant of the flotation separation process of magnesite and dolomite [13,23,24].

The chain length of phosphate also has an important influence on the flotation separation of magnesite and dolomite. As the chain length increases, the complexing ability of phosphate to calcium ions and magnesium ions increases, that is, the inhibition on dolomite and magnesite continues to increase. Among them, the inhibition of dolomite is stronger than that of magnesite. Therefore, the increase of phosphate chain length is beneficial to increase the difference in floatability between magnesite and dolomite, but when the phosphate chain length increases to a certain range, the flotation separation of the two ores is not obvious (like sodium tetrapolyphosphate and sodium hexametaphosphate); this is consistent with the flotation test results. In addition, as the phosphate chain length increases, the concentration of depressant required also decreases.

4. Conclusions

The floatability difference between magnesite and dolomite was small, due to their similar surface electrical properties and crystal structure. Using sodium phosphate depressants can realize the flotation separation of magnesite and dolomite, and the inhibition of dolomite was stronger than magnesite. With the increase of phosphate chain length, the flotation separation effect of minerals was gradually strengthened to a certain degree. The sodium hexametaphosphate and sodium tetrapolyphosphate are potentially effective depressants for the flotation separation of magnesite and dolomite. Moreover, the recoveries of magnesite/dolomite are 71.23%/12.56% and 70.51%/11.17%, respectively. The order of flotation separation enhancement of different sodium phosphate depressants is sodium hexametaphosphate ≈ sodium tetrapolyphosphate > sodium tripolyphosphate > sodium pyrophosphate, which can be further verified by contact angle test. In addition, the concentration of depressants can be reduced with the phosphate chain length increased.

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