The selective depression effect of sodium hexametaphosphate on the separation of chlorite and specularite

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Abstract: Flotation is the most known beneficiation method for the separation of complex and refractory iron ores. As a typical iron-containing silicates, it is difficult to separate chlorite from specularite, because of the similar surface physicochemical properties. In this study, the selective depression effect of sodium hexametaphosphate (SHMP) was conducted via the cationic micro-flotation. The surface adsorption mechanism between SHMP and the two mineral surface was explored through surface adsorption amount tests, Zeta-potential measurements, Fourier transform infrared (FT-IR) and X-ray photoelectron spectroscopy (XPS) analyses. The micro-flotation results indicated that SHMP could selectively depress around 90% of chlorite, while its effect on the floatability of specularite was negligible (<20% depressing). The surface adsorption amount tests, Zeta-potential measurements analysis demonstrated that SHMP selectively adsorb on chlorite surface while on the surface of specularite is feeble. The further surface adsorption analysis via FT-IR and XPS proved that SHMP selective adsorption occurred on the chlorite surface mainly by chemisorption mainly through the chelation reaction between O in the phosphate groups of SHMP molecular and metal ions on surface of chlorite.

Keywords: flotation, depressant, chlorite, specularite, adsorption

1. Introduction

Steel is widely used in aerospace, railway, construction materials and other fields, and about 98% of the world's iron ore is processed into steel (Zhang et al., 2021). With the rapid development of the global economy, the market's demand for steel is increasing, and the easy-to-select iron ore resources are gradually exhausted. The efficient utilization of complex and refractory iron ores has become the focus of the iron ore utilization (Lu, 2015).

Among the mineral processing methods, flotation is one of the most important and widely used for refractory iron ore, which utilizes the difference in wettability of the mineral surface to selectively enrich the target mineral (Li et al., 2019). Collectors often do not show good selectivity, and depressants need to be added during flotation process. Depressants can form a hydrophilic film or dissolve a hydrophobic film on the mineral surface, thereby achieving a depression effect (Ma, 2012). In the iron ore flotation system, starch is the most widely used depressant. Bhagyalaxmi et al. (2013) found that several starch, such as soluble starch, corn starch, potato starch and rice starch all can effectively inhibit hematite, while the floatability of quartz is basically not affected. In addition, infrared spectroscopy indicated that starch is mainly adsorbed through the hydroxyl group in starch molecule and Fe on hematite surface. Bai et al. (2019) researched the effect of soluble starch on the separation effect of hematite and apatite, also confirmed that soluble starch is adsorbed on hematite surface via chemical adsorption of hydroxyl groups, but only weakly physical adsorption with apatite.

Although starch exhibits a strong selective depression effect on iron oxide ore, it is difficult to effectively separate iron-containing-silicate-type-iron-ore (ICSTIO) using starch when there is iron-containing silicate in gangue. This is mainly because the surface and magnetic properties of iron-containing silicate gangue minerals are very similar to those of iron oxide ore. Veloso et al. (2018) investigated the adsorption mechanism of dextrin and corn starch on iron-containing silicate minerals (chlorite, diopside and epidote), and found that corn starch and dextrin had a strong depression effect on iron-containing silicate minerals, and the adsorption on their surface is less affected by the pH value. Thus, starch and its derivatives are not suitable as a flotation depressant for ICSTIO. Further mechanism analysis proved that corn starch was adsorbed on iron-containing silicate surface in the form of chemical complexation reaction, which is the same as the adsorption pattern of starch on the surface of iron oxide ore. Our early research results also confirmed that although starch has a good selective depression effect on hematite, when there is iron-containing silicate in the gangue, starch loses its depression selectivity (Li et al., 2019).

For iron ore flotation separation, it is necessary to add an appropriate amount of depressant after adjusting the pH value of the pulp to suppress iron ore or gangue minerals. Whether it is fluorosilicate (Fan et al., 2020), sulfhydryl depressants (Mei et al., 2020) commonly used in direct flotation, or polysaccharide polymers such as starch and their modified agents (Veloso et al., 2018; Poperechnikova et al., 2017) commonly used in reverse flotation as flotation depressants, depressants were added prior to collectors, and the depressed ores were difficult to be reactivated, especially for ICSTIO. Therefore, the selectivity and depression effect of depressants of iron ore play a crucial role in the overall effect of flotation separation. Li et al. (2020) used chitosan as a selective depressant to separate specularite from chlorite based on the element difference between specularite and chlorite. The results of direct flotation revealed that chitosan can effectively depress chlorite while without significant effect on the floatability of specularite, which confirmed chitosan as a potential depressant for the separation of specularite/chlorite. Further mechanism analysis demonstrated that the adsorption of positively charged chitosan increased the surface positive of chlorite and weakened the subsequent adsorption of cationic collectors on the surface of chlorite. As an environmentally friendly depressant, humic acid (HA) was addressed effective to separate hematite from augite and hornblende, and the depression impact order of HA was: hematite ≫ augite > hornblende. Although HA adsorb on hematite through the interaction with Fe through chemical and hydrogen bonding the same as starch, HA's selectivity is superior to starch (Tohry et al., 2021).

Sodium hexametaphosphate (SHMP), as shown in Fig. 1, is a white crystalline inorganic compound with high solubility in water. It is mainly used as a quality improver, pH regulator, metal ion chelating agent, adhesive, and expansion agent in the food industry. In the field of mineral processing, SHMP has been found to selectively adsorb on the surface of serpentine by bonding with Mg to enhance the electronegativity of the surface. The adsorption of SHMP on serpentine surface wakened the heterogeneous coacervation effect between serpentine and pentlandite, which eliminated the interference effect of serpentine on pentlandite and improved the separation efficiency of the two minerals. (Li et al., 2021). In addition, SHMP was also found to selectively inhibit dolomite by binding to Ca on the dolomite surface, thereby facilitating the separation of dolomite/magnesite (Hu et al., 2020). In the research on the synergistic depression of calcite and SHMP on scheelite flotation, it was found that calcium ions on the surface of calcite can be desorbed into the solution under the action of SHMP, which increases the calcium ion concentration in the solution and enhances the depression effect of SHMP on scheelite (Wang et al., 2018). SHMP can also selectively bond with Al ions on mineral surface. For example, SHMP can inhibit both bauxite and kaolinite in bauxite/kaolinite separation, but when the amount of collector increases, the floatability of diaspore depressed by SHMP gradually becomes better, while that of kaolinite changes little. The main reason is that there is competitive adsorption between SHMP and the collector sodium oleate on these two mineral surfaces, and sodium oleate shows stronger adsorption capacity on the surface of diaspore than that of kaolinite (Zhang et al., 2001).

So far, there has not been a lot of research on SHMP in iron ore flotation, especially ICSTIO separation. It can be seen from the literature above that SHMP can selectively bond with Ca, Mg and Al atoms. As a typical iron-containing silicate, the biggest difference between the surface element

composition of chlorite and specularite is that the surface of chlorite has more of these three elements than that of specularite. Therefore, in this work, SHMP was explored as a selective depressant for specularite/chlorite flotation separation, and its depression effect on chlorite was investigated in detail, and its adsorption mechanism was addressed via detection methods as zeta potential, adsorption capacity, Fourier Transform infrared (FT-IR) and X-ray photoelectron spectroscopy (XPS).
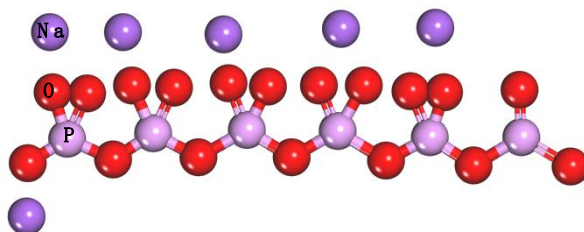

Fig. 1. The molecular structure of SHMP

2. Materials and methods

2.1. Materials and reagents

The single-mineral specularite and chlorite were applied by the Lilou Mine, Anhui Province, and Jianshan Mine, Shanxi Province, China, respectively. Lumps of high-grade specularite and chlorite ores were handpicked after crushing for removing visible impurities. The handpicked samples were then ground to -74 µm via a porcelain ball mill before sieving to obtain -74+37 µm and -37 µm size fractions for further analyses.

Analytical-grade sodium hexametaphosphate (SHMP), dodecylamine (DDA), HCl and NaOH were acquired from Adamas-beta® (Shanghai Titan Scientific Co.). DDA was dissolved in a 5% concentration of HCl solution. DDA, SHMP, HCl and NaOH was used as the collector, depressant, and the pH value modifier respectively. The resistivity of deionized water using in all tests was 18.2 MΩ·cm.

2.2. Methods

2.2.1. Micro-flotation experiments

Micro-flotation experiments were conducted using an XFG-5 flotation machine. The flotation cell volume and agitating speed was 40 mL and 1460 rpm, respectively. For a typical test, as shown in Fig. 2, 2 g single mineral and 30 mL water was added into the flotation cell. After adjusting the pH value of slurry by HCl or NaOH solutions, a certain amount of SHMP and DDA were injected to the slurry in sequence. The condition and flotation time was 3 min and 5 min, respectively. Then, both the froth product and sinking product were collected and dried. The recovery of product was calculated according to the weight distribution.
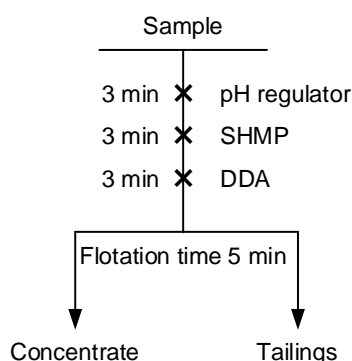

Fig. 2. The flowsheet of micro-flotation

2.2.2. Zeta potential detections

The zeta potential of specularite and chlorite measured using an electrophoretic analyzer (Brookhaven Instruments Ltd. US.) to identify the electrical feature deference of the two mineral particles in water

and SHMP solutions. 5 mg of -2 μm sample was suspended in 50 mL KCl background electrolyte solution with 1×10^{-3} mol/L. Then, the pH value of solution was adjusted by NaOH or HCl. After adding SHMP, the suspension was agitated and maintained for 5 min and 2 min, respectively, to be homogenized. Finally, the slurry was transferred into an electrophoretic cell for determination. Each sample was tested three repeated times, and the results presented were the mean value.

2.2.3. Adsorption experiment

The total organic carbon (TOC) analysis method can be used to analyze the organic matter content in solutions. TOC analysis can be applied to detect the concentration changes of reagents before and after adsorption to obtain the adsorption amount of reagents on mineral surfaces. For an typical adsorption experiment, 2.0 g of specularite or chlorite was put into a conical flask with 30 mL pH 10 of SHMP solution. Then the slurry was agitated using an incubator shaker at 160 rpm for 1 h, before for further analysis of total organic carbon (TOC) via a TOC-L analyzer (Shimadzu Japan). The residual concentration approach was applied to detect the adsorption amount of depressant. The equation for the adsorption amount is as Eq. (1):

$$C = \frac{(C_0 - C_1) \times V}{W} \tag{1}$$

where C (mg/g) is the adsorption amount of the depressant to mineral, C_0 (mg/L) and C_1 (mg/L) was the depressant concentrations before and after adsorption, respectively, V (L) was the solution volume, W (g) was the weight of sample.

2.2.4. FT-IR detections

Fourier Transform infrared (FT-IR) data was collected at room temperature ($25 \pm 1°C$) in a Nicolet 6700 FT-IR spectrometer (Thermo Fisher Scientific, USA). The materials were prepared as follows: 1 g of specularite or chlorite sample with -2 μm size fraction was mixed with 100 mL of water at pH 10 and 25°C before putting into a 250 mL conical flask with or without 30 mg/L SHMP solution. The agitation time was 1 h, then the fully interacted sample was filtered, washed twice with the corresponding pH solution, vacuum-dried at 25°C for 24 h. For a typical measurement, 2 mg of the sample was mixed with 50 mg of KBr and was ground in an alumina mortar until a transparent pellet was obtained.

2.2.5. XPS measurements

XPS measurements were performed on a Thermo ESCALAB 250XI X-ray photoelectron spectrometer (Thermo Fisher Scientific, USA) with monochromatic Al $K\alpha$ radiation as an excitation source. The pass energy and energy step size were set as 100 eV and 1 eV, respectively. All spectra of O1 s, Mg 1s, Al 2p, Fe 2p and Si 2p were calibrated to the binding energy of C 1s at 284.6 eV. In the case of a typical XPS measurement, 1 g of -2 μm size fraction chlorite and 50 mL of deionized water was placed into a beaker at pH 10. The slurry was agitated for 1 h before filtering and drying for 24 h in a vacuum oven at 25°C.

3.   Results and discussion

3.1. Flotation of chlorite and specularite without SHMP

The flotation behavior of chlorite and specularite are shown in Fig. 3 at different pH values and DDA concentration. Fig. 3a illustrates the relationship between pH and flotation recovery with 6 mg/L DDA in the absence of depressant. With the increase of slurry pH value, the recovery of the two minerals shows an increase trend in the pH range of 4 to 8, while shows a downward trend when pH value over 8. The maximum recovery of specularite reach 83.41% at pH 10. Keep pH=10 unchanged, Fig. 3b presents the relationship between the floatability of two minerals and DDA concentration. The floatability of two minerals is positively correlated with DDA concentration. When the DDA concentration exceeds 10mg/L, the recovery of chlorite and specularite will not increase significantly, at which the optimum recovery is 79.92% and 85.89% respectively. The difference between the recoveries of these two minerals is mainly due to the poor selectivity of DDA. Therefore, without the use of depressant, DDA is ineffective for their separation.
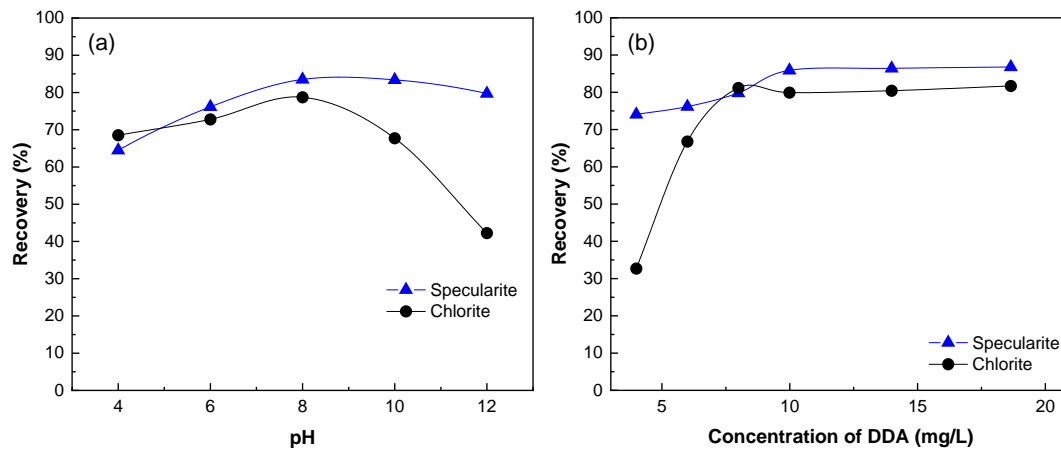
Fig. 3. Flotation behavior of chlorite and specularite with different (a) pH values and (b) DDA concentration

3.2. Flotation of chlorite and specularite with SHMP

It is not ideal to separate specularite from chlorite only using collector DDA, thus, SHMP is applied as a depressant. The recovery as a function of SHMP concentration and pH value are shown in Fig. 4. It is clearly that, the floatability of chlorite decreased significantly with the addition of SHMP, while the recovery of specularite remained high lever. The largest recovery difference (60.86 percentage point) can be found at pH 10 with 18 mg/L SHMP. It can be found from Fig. 4(b) that, the floatability of chlorite decreased sharply with SHMP while the decrease of recovery rate of specularite is not obvious. The optimal flotation result was obtained with 24 mg/L SHMP at pH value 10, in which the recovery difference was 61.60 percentage point. SHMP shows excellent depression effect and selectivity for the separation of specularite and chlorite, and is a potential depressant for iron oxide flotation.
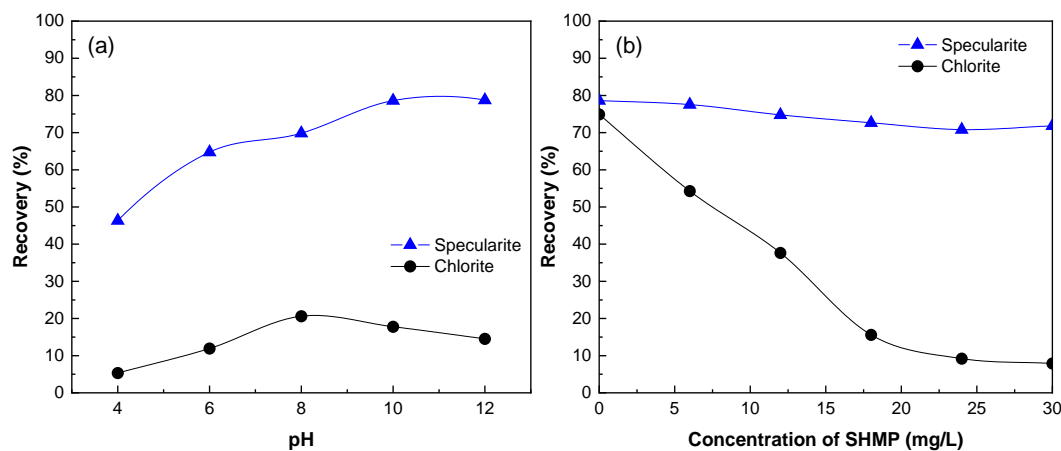

Fig. 4. Flotation behavior of chlorite and specularite at different (a) pH values and (b) SHMP concentration

3.3. Zeta potential measurements

In order to study the effect of adsorption of SHMP on the surface electrical properties of two minerals, the Zeta potential measurements were carried out with 24 mg/L SHMP. As shown in Fig. 5, the Zeta potential of both minerals shifted to the right with the increase of pH value, which is closely related to the pH value. The iso-electric point (IEP) of chlorite and specularite in nature condition was 4.72 and 5.06, respectively, which are consistent with experiments done by researchers (Chen et al., 2019; Quast, 2017; Rohem et al., 2019). For chlorite sample (Fig. 5a), its zeta potential changed significantly with the addition of SHMP. The IEP of chlorite negatively shifted from 4.72 to 2.60 after introducing SHMP into the suspension. This indicates that SHMP shows strong adsorption interaction with chlorite, which leads to low floatability of chlorite.

It is different that, compared with chlorite, the Zeta potential shift of specularite was inconspicuous, as shown in Fig. 5b. At low pH conditions, the IEP of specularite only shifted from pH 5.06 to 4.62. This

inappreciable shift demonstrated that the adsorption between SHMP and specularite surface is feeble. Overall, the adsorption of SHMP on chlorite surface greatly weaken the floatability of chlorite and enlarged the floatability difference between the two minerals..
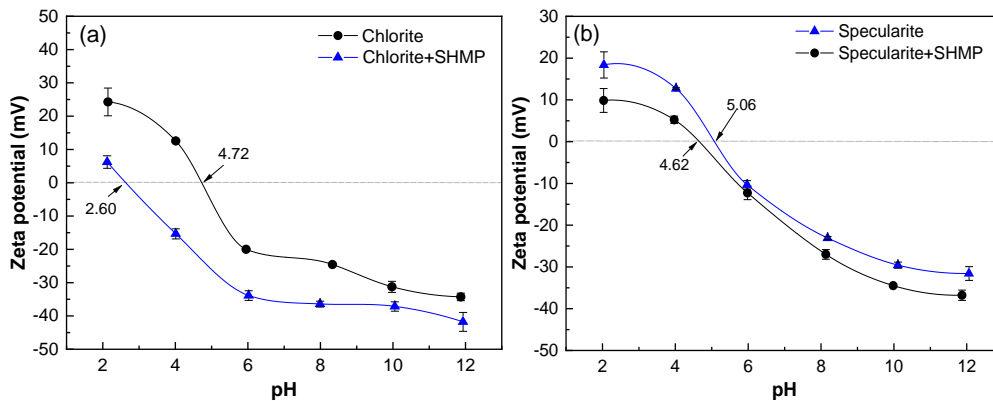

Fig. 5. Zeta potential measurements with and without SHMP at different pH

3.4. Adsorption amount measurements

The adsorption affinity differences on different mineral surfaces display the selectivity of a depressant and directly determine its depression performance (Yang et al., 2020). In order to research the adsorption performance of SHMP on the two minerals, the adsorption amount of SHMP with different concentration was measured. As displayed in Fig. 6, with the increase of SHMP solution concentration, the adsorption capacity of SHMP on the surface of the two minerals increases. The adsorption amount of SHMP on chlorite surface is apparently higher than specularite, which demonstrated that the adsorption affinity of SHMP for specularite is weaker than that of chlorite. The adsorption affinity SHMP on chlorite and specularite surface increase their hydrophilicity difference, which is consistent with the results of flotation.
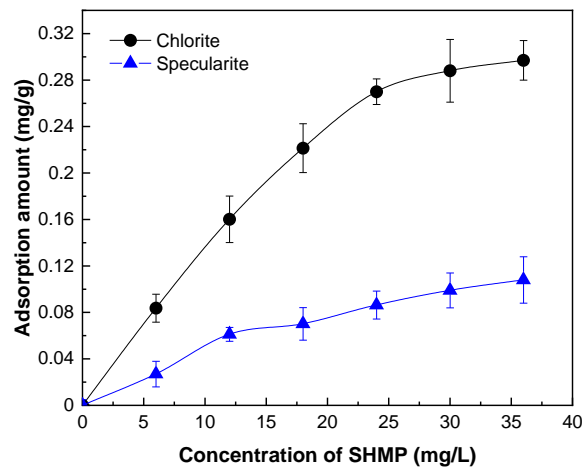

Fig. 6. Adsorption performance of SHMP on chlorite and specularite surface at different concentration

3.5. FT-IR analysis

In order to explore the adsorption mechanism of SHMP, FT-IR tests were conducted on the surface of specularite and chlorite before and after treating with SHMP. As shown in Fig. 7, the peaks at 1281 cm^{-1} and 868 cm^{-1} are the stretching vibration of P=O bond and P-O-P bond, respectively, while 1090 cm^{-1} and 991 cm^{-1} are mainly characteristic peaks of P-O bond, and the characteristic peaks are all in good agreement with the standard spectrum (Feng et al., 2011; Li et al., 2017). After the adsorption of SHMP on chlorite, the absorption peak of -OH group near 3440 cm^{-1} became two new absorption peaks, indicating that SHMP was hydrolyzed after adsorption and the -OH group was involved in the adsorption. The right shift of the -OH group peak to 3414 cm^{-1} indicates that the -OH group reacts with

some metal ions on chlorite surface, and the stretching vibration of the -OH group at 1640 cm⁻¹ of chlorite also proves this process. The absorption peak at 995 cm⁻¹ of chlorite shifted to the right, and two new absorption peaks at 785 cm-1 and 648 cm⁻¹ were generated, which may be due to the interaction of phosphate anions of SHMP with chlorite. The phosphate anions complexed with metal ions on the chlorite surface, resulting the adsorption of SHMP on the chlorite surface via -P-O-P and -P-O groups (Kasomo et al., 2020).

The complexation of surface metal ions leads to a large amount of adsorption of -P-O-P groups and -P-O groups on the surface of chlorite. In summary, SHMP depress chlorite mainly through chemical adsorption, and the complexation between phosphate and metal ions is the dominant factor for adsorption.
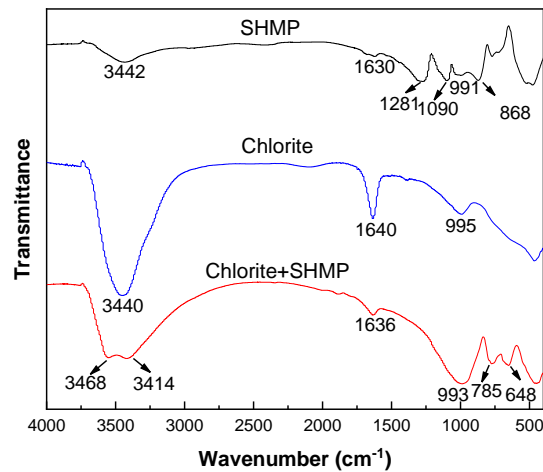

Fig. 7. FT-IR spectra of SHMP adsorption on chlorite surface

3.6. XPS

To further explain the inhibition performance of SHMP on the surface of chlorite, XPS analysis was carried out on chlorite before and after SHMP treatment. As shown in Fig. 8, metallic elements Mg, Fe, Al and non-metallic elements C, O, P and Si are detected on chlorite surface. Combined with table 1, the atomic concentrations of P increase from 0.21% to 0.70% after addition of SHMP, indicating the adsorption of SHMP on the surface of chlorite.
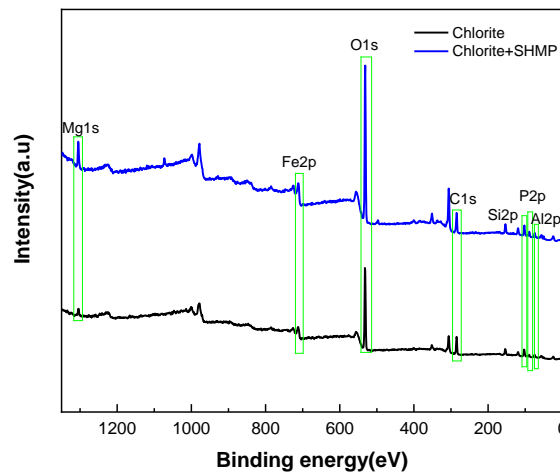

Fig. 8. XPS survey of chlorite before and after addition of SHMP

The high-resolution XPS spectra and their fitting curves of chlorite Mg1s, Fe2p, Al2p, O1s and Si2p before and after SHMP treatment are shown in Fig. 9. In Fig. 9a, the peak at 531.9 eV corresponds to SiO$_2$ (Wei et al., 2019; Haber et al., 1976) on the chlorite surface, and in Fig. 9b a new peak corresponding to the phosphate group of SHMP appears at 530.5 eV, indicating that SHMP is adsorbed on the surface of chlorite.

Table. 1. The relative atomic contents on chlorite surface before and after addition of SHMP

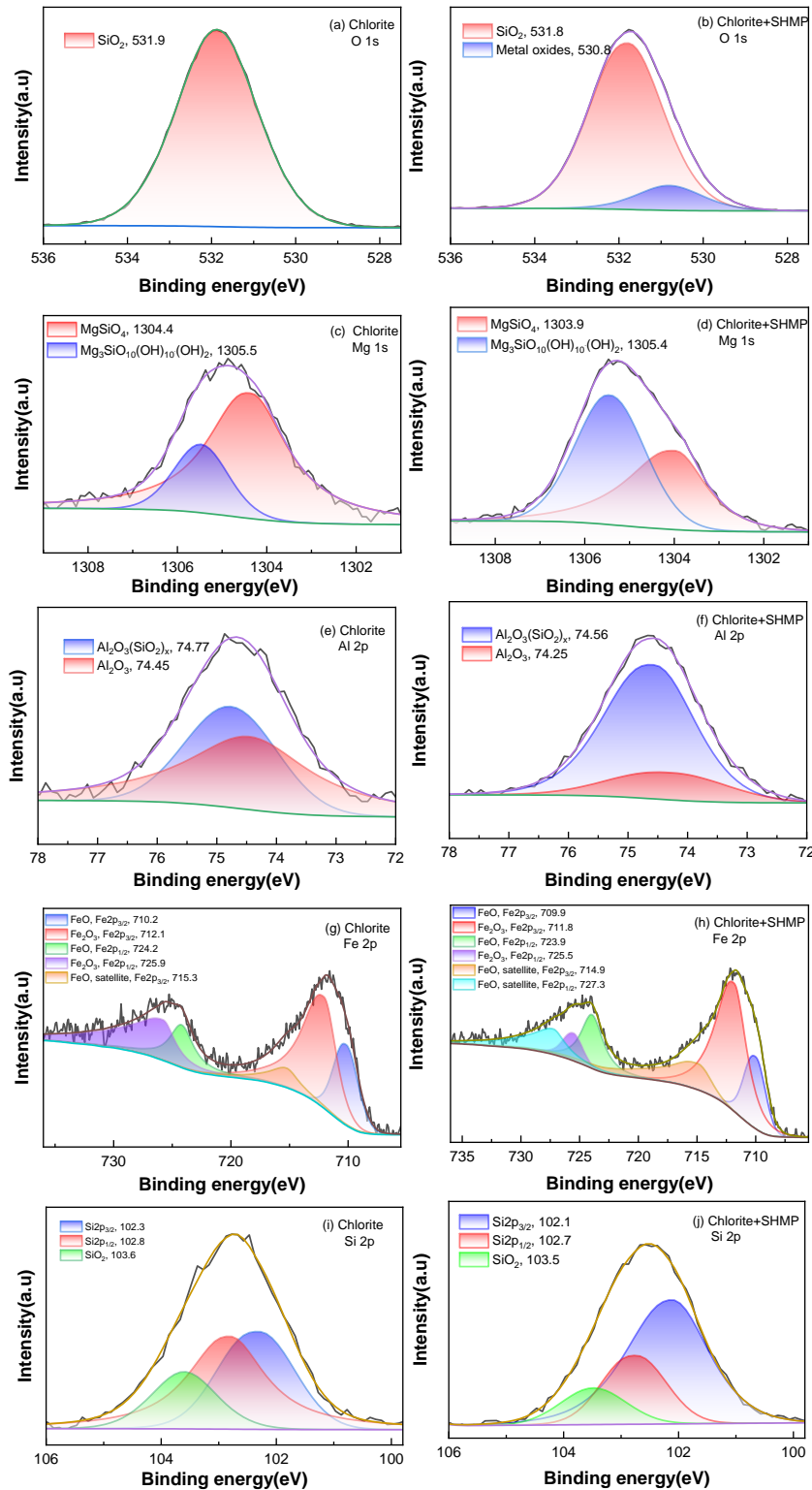| Samples	Element/%	
	O	P
Chlorite	33.08	0.21
Chlorite + SHMP	54.91	0.70

Fig. 9. High-resolution XPS spectra of O 1s, Mg 1s, Al 2p, Fe 2p and Si 2p on chlorite surface before and after being treated with SHMP

In Fig. 9c and Fig. 9d, the two Mg 1s peaks at 1304.4 eV (Mg(SiO4)) and 1305.5 eV (Mg$_3$SiO$_{10}$(OH)$_{10}$(OH)$_2$) shifted to the lower binding energy to 1303.9 eV and 1305.4 eV, indicating that the Mg ions on the surface of chlorite accept electrons during SHMP adsorption process. In Figure 9e-9f, peaks of Fe 2p and Al 2p are the same as Mg 1s, and almost all shifted to the lower binding energy with the displacement above 0.2 eV. For example, Al 2p moved from 74.77 eV (Al$_2$O$_3$(SiO$_2$)$_x$) to 74.56 eV, and Fe 2p moved from 712.1 eV (Fe$_2$O$_3$, Fe2p3/2) to 711.8 eV, indicating that SHMP is chemically adsorbed on the surface of chlorite (Wang et al., 2021; Han et al., 2022), which may be caused by the electron-donating phosphate groups of SHMP donating electrons to metal sites on the surface of chlorite to form metal complexes (Wang et al., 2022; Li et al., 2022). In addition, in Fig. 9i and 9j, the peak in Si 2p also shifted towards lower binding energies, indicating partial electron transfer from the phosphate groups to Si. Although theoretically the tetrahedral closed structure of SiO$_2$ hinders the transfer of electrons, the transfer of electrons from phosphate groups to Si may be caused by the metal ions bound to Si acting as a bridge to transfer electrons (Li et al., 2022).

In summary, SHMP donates electrons to metal ions (Mg, Al, and Fe) on chlorite surface by means of the phosphoric acid groups. The adsorption process increases the hydrophilicity of chlorite, which expands the floatability difference between chlorite and specularite.

The possible adsorption reaction model of between SHMP and chlorite surface is proposed as shown in Fig. 10. SHMP molecules adsorb on chlorite surface mainly through the chelation reaction between O in the phosphate group and metal ions. Electron transfer occurs between O and these metal ions during the adsorption process, and electrons are transferred from O to these metal ions. In addition, due to the presence of metal ions bonded to Si that act as an electron transfer bridge, the phosphoric acid group partially transfers electrons to Si. Moreover, since DDA acts on the surface of chlorite through physical adsorption and hydrogen bond adsorption, while for SHMP is mainly chemical adsorption, because the selectivity and action intensity of chemical adsorption are significantly greater than those of physical adsorption and hydrogen bonding. Therefore, the selective adsorption of SHMP on chlorite surface hinders the subsequent adsorption of collector DDA, thereby increasing the hydrophobicity difference between the surface of chlorite and specularite, and finally realizing the effective separation of the two minerals.
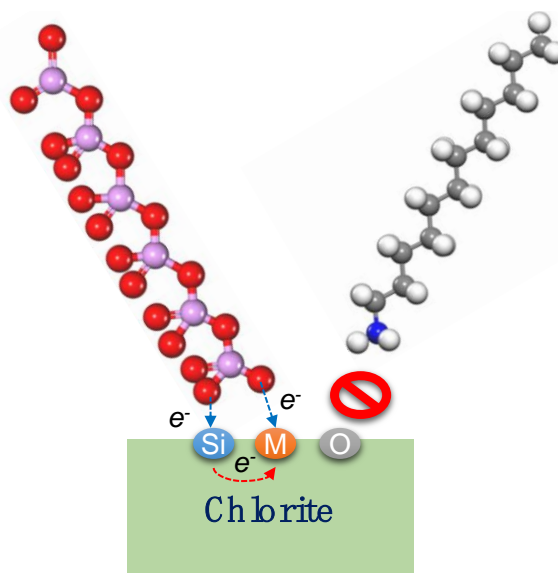

Fig. 10. The possible schematic adsorption reaction model of SHMP on the surface of chlorite (M represents the Fe, Al, Mg ions on chlorite surface). The gray, white, red, blue and pink balls represent carbon, hydrogen, oxygen, nitrogen and phosphorus, respectively

4. Conclusions

This work involves research of the selective depression effects of SHMP on improving specularite/chlorite separation. The adsorption mechanism of SHMP was addressed through various characterization and analyses methods. The collector effect of DDA on the separation of specularite and

chlorite was unsatisfactory without a depressant, while SHMP displayed remarkable selectivity depression to chlorite, and the optimal separation result with 61.60 percentage point of the recovery difference was achieved with 24 mg/L SHMP and 14.4 mg/L DDA at pH 10. Mechanism analyses have verified that SHMP adsorbs on the surface of chlorite favourably than that of specularite via the interaction between O in phosphate groups and metal ions, which increased the floatability difference of the two minerals.

Acknowledgments

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