New insights into the beneficial roles of dispersants in reducing negative influence of Mg$^{2+}$ on molybdenite flotation

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Due to the shortage of freshwater, seawater has been widely considered for mineral flotation. However, the presence of Mg$^{2+}$ in seawater plays an apparently negative role. In this work, two dispersants (i.e., sodium silicate (SS) and sodium hexametaphosphate (SH)) were used to reduce the detrimental effects of Mg$^{2+}$ on the flotation of molybdenite (MoS$_2$). Various measurements including contact angle, zeta potential, FTIR and XPS were conducted to understand the impacts of these two dispersants on MoS$_2$ flotation. Results indicate that both dispersants prevented the adsorption of colloidal Mg(OH)$_2$ onto MoS$_2$ surface under alkaline conditions, thereby improving MoS$_2$ floatability. In addition, both dispersants are physically adsorbed on MoS$_2$ surface, but chemically adsorbed on Mg(OH)$_2$ surface. In this study, the extended Derjaguin–Landau–Verwey–Overbeek (DLVO) calculation suggests that both SS and SH reverse the total interaction energies between MoS$_2$ and colloidal Mg(OH)$_2$ from negative (attraction force) to positive (repulsive force), with the impact of SH being more significant.

1. Introduction

Molybdenite (MoS$_2$), as the most important molybdenum (Mo)-containing mineral, 1–4 is frequently associated with Cu-bearing minerals and concentrated via flotation which consumes a significant amount of water. 5,6 Generally, the quality of flotation media highly affects mineral flotation efficiency 6 while freshwater is normally considered as an ideal medium. 7 However, freshwater only accounts for about 0.5–0.8% of the total water source on Earth. 8 Therefore, there is an increasing demand in applying alternative water sources like seawater containing high concentrations of electrolytes to minimize the usage of freshwater. 9

Previous studies show that bubble coalescence can be inhibited when the concentration of electrolytes in seawater (e.g., NaCl, KCl, CaCl$_2$, MgCl$_2$ and MgSO$_4$) exceeds the threshold for critical coalescence. 10–12 However, seawater inhibits MoS$_2$ flotation under alkaline conditions, primarily due to the adsorption of colloidal Mg(OH)$_2$ precipitated onto MoS$_2$ surface. 13–15 Therefore, great efforts have been made to reduce the negative influence of seawater (especially Mg$^{2+}$ ions). For instance, Suyantara, et al. 16 reported that the addition of emulsified kerosene prevented the adsorption of Mg(OH)$_2$ onto MoS$_2$. Jeldres, et al. 17 found that the addition of Na$_2$CO$_3$ and CaO reduce Mg-hydroxyl complexes on MoS$_2$ surface, thus improving MoS$_2$ recovery.

In both industrial implementation and fundamental studies, sodium silicate (SS) and sodium hexametaphosphate (SH) are commonly used as dispersants to disperse hydrophilic substances from the surface of valuable minerals. 18–20 Recently, more attention has been paid to the effects of these dispersants on mineral flotation in seawater. For instance, our previous studies 21,22 have shown that the addition of SH can form dissoluble complexes with Mg$^{2+}$, reducing the generation and adsorption of hydrophilic complexes on mineral surfaces. Others have also reported that Mg$^{2+}$ plays the most significantly negative role in reducing the floatability of minerals in seawater.13,23 However, the influencing mechanisms of Mg$^{2+}$ on MoS$_2$ flotation are still not fully understood when using SS or SH.

In this study, MoS$_2$ flotation was carried out in the solution containing Mg$^{2+}$ ions in the presence of SS and SH, to investigate the influencing mechanisms of these two dispersants on MoS$_2$ flotation, with the assistance of various analyses such as contact angle, zeta potential, FTIR and XPS. Moreover, the interaction force between particles was predicted based on the Derjaguin–Landau–Verwey–Overbeek (EDLVO) theoretical calculation.

2. Materials and methods

2.1 Samples and reagents

The raw MoS$_2$ sample was purchased from Guilin, Guangxi province, China, which was crushed, milled and wet sieved to...
2.2 Flotation experiments

Laboratory flotation test was carried out using a mechanically agitated XFG II flotation machine (Wuhan Exploration Machinery Factory, China). Each solution suspension (25 mL) containing 0.25 g of sized MoS$_2$ and 0.05 M MgCl$_2$ (consistent with that of Mg$^{2+}$ in seawater) was poured into the flotation cell (40 mL). When required, SS or SH was added into the pulp, followed by the addition of NaOH solution to maintain the desired pH (e.g., pH 10 to depress pyrite$^{41}$) for 6 min, prior to flotation. The pulp was stirred at 1200 rpm with a constant airflow rate of 1.2 cm s$^{-1}$, while the froth product was collected at a time interval of 10 s. Both filtered froth products and residues were vacuum-dried at 30 °C for 24 h prior to weighing and recovery calculation. No collectors were applied to investigate the mechanisms of dispersants more clearly, i.e. excluding the influence from collectors.

2.3 Contact angle measurements

The fresh MoS$_2$ surface was obtained by peeling off the top layer of MoS$_2$ slab which was immersed into the conditioned solution same to the flotation experiment. The treated surface was then washed softly using ultrapure water and air-dried. 0.25 μL of ultrapure water was dropped onto the treated MoS$_2$ surface using a micro-syringe. Subsequently, the contact angle of MoS$_2$ was measured using a JC2000C device (Shanghai Zhongchens Digital Technology Company, China). The average values of triplicate measurements were reported herein.

2.4 Zeta potential measurements

Zeta potential of MoS$_2$ particles was determined via electrokinetic mobility analysis,$^{23}$ using a Nano-ZS90 zeta potential analyzer (Malvern Co., Ltd., UK), at room temperature. Prior to analysis, 50 mg of MoS$_2$ particles (<5 μm) were mixed in 50 mL solution for 10 min. The suspension pH was adjusted to desired value by adding NaOH solution. Finally, triplicate measurements were conducted and the average zeta potential value was reported.

2.5 FTIR measurements

50 mL of suspension (pH 10) containing 0.5 g of MoS$_2$ was stirred for 10 min. The filtered sample was washed using ultrapure water, and then freeze-dried under vacuum for 24 h. 2.5 mg of dried sample was mixed with KBr (250 mg) prior to pressing into thin pellets. The FTIR measurements were carried out using a Nicolet IS-10 instrument (Thermo Fisher Scientific Inc., Waltham, MA, USA).

2.6 XRD measurements

In addition to the MoS$_2$ sample, the precipitation formed in the solution was also sampled and analyzed by X-ray diffraction (XRD, D8 Advance, Bruker, Germany). Cu target and Kα ray were used as X-ray sources, the wavelength, tube voltage and tube current were controlled at 0.154056 nm, 40 kV and 30 mA in the test, respectively. Scanning speed and range were 3° min$^{-1}$ and 10–70°, respectively. The XRD analysis indicated that the majority of this sample was well-crystallized MoS$_2$.

2.7 XPS measurements

The elemental concentration of surface chemical species on MoS$_2$ surface was determined by ESCALAB 250Xi XPS instrument (Thermo Fisher Scientific Inc., Waltham, MA, USA) with an Al Kα monochromatic X-ray source (1486.6 eV). The XPS spectra were obtained at a step size of 1.0 eV. The survey and high-resolution spectra were collected with pass energies of 100 eV and 30 eV, respectively. All XPS spectra data were analyzed using XPS Peak 4.1 software. Binding energy was calibrated based on C 1s at 284.8 eV.

2.8 EDLVO calculation

Extended Derjaguin–Landau–Verwey–Overbeek (EDLVO) theoretical model is used to predict the interaction energy between particles in aqueous, normally with van der Waals ($V_w$) and electrostatic interaction energies ($V_E$). Once external substances are added, steric hindrance interaction energy ($V_{SR}$) should be considered. Therefore, the total interaction energy ($V_T$) can be described using eqn (1).$^{26–28}$

\[
V_T = V_W + V_E + V_{SR}
\]  

($V_W$ can be calculated according to eqn (2).)

\[
V_W = \frac{A}{6H} \left( \frac{R_1 R_2}{R_1 + R_2} \right)
\]  

Fig. 1. The structures of SS (a) and SH (b).
where \( k \) represents the distance between particles. \( R_1 \) and \( R_2 \) refer to the average radius of heterogeneous particles. \( R_1 \) (33 \( \mu \)m) and \( R_2 \) (3.8 \( \mu \)m) are the average radius of MoS\(_2\) and Mg(OH)\(_2\) particles. \( A \) is the effective Hamaker constant calculated using eqn (3).

\[
A = \left( \sqrt{A_{11}} - \sqrt{A_{33}} \right) \left( \sqrt{A_{22}} - \sqrt{A_{33}} \right)
\]

(3)

where \( A_{11}, A_{22} \) and \( A_{33} \) are the Hamaker constants of MoS\(_2\), Mg(OH)\(_2\) and water, respectively. The Hamaker constants of MoS\(_2\) \((A_{11})\) and Mg(OH)\(_2\) particles \((A_{33})\) are \(9.38 \times 10^{-20}\) J and \(1.62 \times 10^{-20}\) J, respectively. The Hamaker constant of water \(A_{33}\) is \(3.7 \times 10^{-20}\) J.

The electrostatic interaction energy between MoS\(_2\) and colloidal Mg(OH)\(_2\) can be calculated using eqn (4).

\[
V_E = \frac{\Pi \varepsilon_0 R R}{R_1 + R_2} \left( \psi_1^2 + \psi_2^2 \right)
\]

\[
\left\{ \frac{2 \psi_1 \psi_2}{\psi_1^2 + \psi_2^2} \ln \left( \frac{1 + e^{-H}}{1 - e^{-H}} \right) + \ln \left( 1 - e^{-2H} \right) \right\}
\]

(4)

where \( \kappa \) is the thickness of electric double layer \((0.180 \text{ nm})^{12,13} \varepsilon_0 \) and \( \varepsilon_r \) are the vacuum and relative dielectric constants of the continuous phase, respectively, \( \varepsilon_0 \varepsilon_r = 6.95 \times 10^{-10} \text{ C}^2 \text{ ( m)}^{-1} \text{.}^{28} \) \( \psi_1 \) and \( \psi_2 \) are the surface potentials of particles.\(^{33} \)

As indicated in previous studies, the addition of SH results in steric hindrance interaction due to the steric hindrance effects.\(^{28,29,34} \) Therefore, the steric hindrance interaction should be considered and can be calculated based on eqn (5).

\[
V_{SR} = \frac{4\Pi R^2 (\delta - \frac{H}{2})}{Z(R + \delta)} kT \ln \left( \frac{2\delta}{H} \right)
\]

(5)

where \( k \) stands for the Boltzmann constant, \( 1.381 \times 10^{-23} \text{ J K}^{-1} \). \( R \) is the radius of particles, \( \delta \) represents the thickness of the adsorbed layer \((5.45 \text{ nm})\) \((\text{ref. 28})\), and \( Z \) refers to the covering area of the macromolecules on the particle surfaces.

3. Results

3.1 Flotation results

Fig. 2a shows the MoS\(_2\) recovery at 10 min as a function of dispersant (SS or SH) dosage from 0 to 50 mg L\(^{-1}\) in 0.05 M MgCl\(_2\) solution. In the absence of SS or SH, a low MoS\(_2\) recovery of 22% was observed, indicating a negative role of Mg\(^{2+}\) on MoS\(_2\) flotation, probably due to the formation and adsorption of Mg(OH)\(_2\) precipitates on MoS\(_2\) surface.\(^{13,35} \)

With the increase of SS/SH dosage, MoS\(_2\) recovery increased to various degrees, indicating the beneficial roles of SS and SH on MoS\(_2\) flotation, with the latter being more significant. Specifically, the MoS\(_2\) recovery dramatically increased from 22% to 78% when SH was increased to 30 mg L\(^{-1}\). Further increase in SH dosage to 50 mg L\(^{-1}\) only slightly increased the recovery. Differently, the MoS\(_2\) recovery increased linearly from 22% to 62% when SS dosage was increased from 0 to 50 mg L\(^{-1}\). It should be noted that the MoS\(_2\) recovery using SS was still lower than that using SH. Therefore, 50 mg L\(^{-1}\) was selected for further study.

Fig. 2b shows MoS\(_2\) recovery as a function of time. In the absence of SS or SH, MoS\(_2\) recovery was increased from 1% to 22% within 10 min, following an overall parabolic trend. However, MoS\(_2\) recovery was increased more rapidly when dispersants were added, giving significantly greater recovery at 10 min, i.e., 62% and 83% in the presence of SS and SH, respectively.

3.2 Contact angle analysis

Fig. 3 shows the effect of SS/SH on the contact angle of MoS\(_2\). The fresh MoS\(_2\) surface shows a high contact angle of 87° in pure water \((\text{Fig. 3a})\), indicating a good hydrophobicity, similar to previous findings.\(^{15,35} \) However, a contact angle of 71° was observed in 0.05 M MgCl\(_2\) solution \((\text{Fig. 3b})\). When 50 mg L\(^{-1}\) of SS or SH was added, the contact angle of MoS\(_2\) was increased to 80° \((\text{Fig. 3c})\) and 83° \((\text{Fig. 3d})\), respectively, suggesting that SS and SH increase the hydrophobicity of MoS\(_2\) surface, with the effect of SH being more apparent on increasing contact angle of MoS\(_2\).

3.3 Zeta potential analysis

Fig. 4 shows the zeta potentials of MoS\(_2\) and Mg(OH)\(_2\) with and without SS/SH at pH 10. The zeta potential of MoS\(_2\) in the
absence of SS or SH was $-57 \pm 2.2$ mV, close to that reported in Hirajima, et al. However, this value was decreased to $-62 \pm 2.8$ mV and $-72 \pm 4.8$ mV, respectively, in the presence of SS or SH, indicating that SH plays a more significant role on decreasing the zeta potential of MoS$_2$.

However, Mg(OH)$_2$ surface was positively charged at pH 10, i.e., $15 \pm 4.9$ mV, consistent with that reported in Schott. In the presence of SS/SH, the zeta potential of Mg(OH)$_2$ was decreased to $-11 \pm 3.5$ mV and $-23 \pm 1.9$ mV, respectively. The change of zeta potential of Mg(OH)$_2$ from positive (without SH or SS) to negative (with SH or SS) indicates the adsorption of negatively charged SH or SS on Mg(OH)$_2$ surface.

### 3.4 FTIR analysis

FTIR spectral analyses (Fig. 5) were carried out to further understand the interaction between dispersants and colloidal Mg(OH)$_2$ or MoS$_2$ particles. The peak located at 881.8 cm$^{-1}$ was assigned to P=O, while the peaks at 1019.3 and 1093.3 cm$^{-1}$ were due to the stretching vibration of P–O. The peak at 1274.7 cm$^{-1}$ corresponds to the asymmetric stretching vibration of P–O–P. The characteristic peaks at 631.9 cm$^{-1}$ and 763.9 cm$^{-1}$ for SS were attributed to the asymmetric deformation vibration of (H)O–Si–O(Na) and (H)O–Si–O(H), respectively. The peaks located at 856.3 cm$^{-1}$ and 925.3 cm$^{-1}$ were due to the symmetric stretching vibration of [Na]O–Si–O(H) and [Na]O–Si–O(Na), while the peaks at 1010.1 and 1169.9 cm$^{-1}$ were ascribed to the asymmetric stretching vibration of Si–O(H) and Si–O(Na).

The characteristic peaks at 3300–3600 cm$^{-1}$ were due to the stretching vibration of hydroxyl groups.

Fig. 5b shows the spectra of MoS$_2$ with and without SH/SS. No new peaks appeared on MoS$_2$ in the presence of SH and SS, indicating that the adsorption of two dispersants on MoS$_2$ surface was dominated by physical adsorption. Fig. 5c shows the spectra of Mg(OH)$_2$ in the absence and presence of SH/SS. The sharp characteristic peaks at 1384.4 cm$^{-1}$ and 3698.7 cm$^{-1}$ were attributed to O–H vibrations of Mg(OH)$_2$. The new characteristic peaks at 910.8, 1122.3, 1259.4 and 1460.2 cm$^{-1}$ were due to the presence of SH on Mg(OH)$_2$ while the peaks at 910.8 and 1122.3 cm$^{-1}$ were due to the shift of P=O peaks of SH at 881.8 and 1093.3 cm$^{-1}$, respectively. The characteristic peak at 1259.4 cm$^{-1}$ was due to shift of P–O–P of SH that originally at 1274.7 cm$^{-1}$. Therefore, SH is chemically adsorbed on Mg(OH)$_2$. Similarly, new characteristic peaks at 1035.6 and 1430.8 cm$^{-1}$ due to SS appeared on Mg(OH)$_2$ surface, indicating a chemical adsorption mechanism between SS and MoS$_2$.

### 3.5 XPS analysis

Fig. 6 shows the XPS survey for MoS$_2$ surfaces in the absence and presence of SH/SS. No characteristic peaks due to Mg 2s

| Element | Conditions | Untreated MgCl$_2$ | MgCl$_2$+SS | MgCl$_2$+SH |
|---------|------------|--------------------|--------------|--------------|
| S 2p    | BE [eV]    | 162.4              | 58           | 58           |
| O 1s    |            | 533.2              | 7            | 7            |
| Mo 3d   |            | 230.0              | 34           | 33           |
| Mg 2s   |            | 89.5               | 1            | 5            |

763.9 cm$^{-1}$ for SS were attributed to the asymmetric deformation vibration of [H]O–Si–O(Na) and [H]O–Si–O(H), respectively. The peaks located at 856.3 cm$^{-1}$ and 925.3 cm$^{-1}$ were due to the symmetric stretching vibration of [Na]O–Si–O(H) and [Na]O–Si–O(Na), while the peaks at 1010.1 and 1169.9 cm$^{-1}$ were ascribed to the asymmetric stretching vibration of Si–O(H) and Si–O(Na). The characteristic peaks at 3300–3600 cm$^{-1}$ were due to the stretching vibration of hydroxyl groups.

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### 3.5 XPS analysis

Fig. 6 shows the XPS survey for MoS$_2$ surfaces in the absence and presence of SH/SS. No characteristic peaks due to Mg 2s
were detected on the untreated MoS₂ surface, but appeared on MoS₂ surface treated in 0.05 M MgCl₂. The peak intensities of O 1s and C 1s were also increased significantly.

Table 1 shows that Mg 2s and O 1s were increased from 1 at% to 5 at% and 7 at% to 10 at%, respectively, indicating the adsorption of Mg(OH)₂ precipitates on MoS₂ surface in 0.05 M MgCl₂ solution. However, upon addition of SH or SS, the peak intensities of Mg 2s and O 1s were reduced, e.g., from 5 at% to 2 at% and from 10 at% to 7 at%, respectively, indicating that the addition of these two dispersants can prevent the adsorption of Mg(OH)₂ precipitates on the surface of MoS₂.

4. Discussion

Fig. 7 shows the XRD patterns of white precipitates formed in 0.05 M MgCl₂ solution. Mg(OH)₂ was found to be the predominant phase, with most crystal faces being detected, indicating a quick formation of crystalline Mg(OH)₂ under the flotation condition. As indicated in the previous studies and Fig. 2 herein, MoS₂ flotation recovery was reduced in 0.05 M MgCl₂ solution, primarily due to the formation and adsorption of Mg(OH)₂ on MoS₂ surface, consistent with the contact angle measurements (Fig. 3). However, the mechanism between MoS₂ and Mg(OH)₂ is still not fully understood.

The dominant force between MoS₂ and Mg(OH)₂ can be calculated based on the EDLVO theory. Generally, the more negative Vₜ between particles, the greater the attraction force to aggregates. In contrast, the more positive Vₜ responds to the stronger repulsive force between particles, resulting in a more dispersed pulp system. Fig. 8 shows that the Vₜ between MoS₂ and Mg(OH)₂ remains negative within the measured particle distance in the absence of dispersants, indicating the aggregation of MoS₂–Mg(OH)₂. In other words, Mg(OH)₂ is likely attached onto the MoS₂ surface in MgCl₂ solution in the absence of dispersants.

In the presence of SS, the Vₜ between MoS₂ and Mg(OH)₂ is gradually increased from a negative to a positive value when the distance increases, indicating that MoS₂ and Mg(OH)₂ particles repel each other. Moreover, the Vₜ value between MoS₂ and Mg(OH)₂ in the presence of SH is always positive within the range of distance examined, suggesting a predominate repulsive force due to its long chain structure. As the adsorption of Mg(OH)₂ on MoS₂ surface decreases, the surface of MoS₂ becomes more hydrophobic, giving rise to the increase of MoS₂ flotation that observed in Fig. 2. Therefore, SS and SH increase the repulsion between MoS₂ and Mg(OH)₂ particles, thereby decreasing the adsorption of Mg(OH)₂ on MoS₂ surface, with the effect of SH being more significant.

5. Conclusions

A low recovery of 22% was found for MoS₂ flotation in 0.05 M MgCl₂ solution controlled at pH 10. MoS₂ recovery was increased significantly in the presence of SS/SH. Various measurements indicate that SS and SH were chemically adsorbed onto Mg(OH)₂, reversing its zeta potential from positive to negative. However, SS and SH were physically adsorbed onto MoS₂, further decreasing the zeta potential of MoS₂. The presence of SS and SH inhibits the adsorption of Mg(OH)₂ precipitates onto the negatively charged MoS₂ surface via electrostatic repulsion, thereby increasing MoS₂ recovery in 0.05 M MgCl₂. Further theoretical calculation demonstrates that the addition of SS or SH changes the interaction force between particles from attractive to repulsive, thereby preventing the adsorption of hydrophilic colloidal Mg(OH)₂ on the MoS₂ surface, with the influence of SH being more significant.

Conflicts of interest

There are no conflicts to declare.

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