

*Received November 3, 2016; reviewed; accepted March 23, 2017*

## **Interaction between mineral particles during ascharite flotation process and direct force measurement using AFM**

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**Abstract:** Interaction between mineral particles during ascharite flotation was investigated by flotation, zeta potential, Derjaguin-Landau-Verwey-Overbeek (DLVO) theory, atomic force microscope (AFM) and SEM-EDS tests. Flotation results showed that the ascharite recovery decreased significantly in the presence of serpentine. In order to improve the ascharite recovery, negative charged quartz was used to limit the detrimental effect of serpentine on ascharite flotation in this research. The effect of quartz on improving ascharite recovery was attributed to the particle surface interactions. The DLVO theory was applied to study the particles interaction in the aqueous solution. The fitting curves showed that serpentine could attach to the surface of quartz easily, and quartz would be well dispersed with ascharite. Therefore, quartz can improve the ascharite flotation performance effectively by interaction between particles. Zeta potential tests showed the opposite charges on the surfaces of mineral particles, which was the basic reason leading to particles interaction. The force measurement results of AFM indicated that the attraction force existed between serpentine and ascharite, as well as serpentine and quartz, but the force between quartz and ascharite was repulsive. The DLVO theory was in a good agreement with the results of AFM. Eventually, particles coating were observed by SEM-EDS, which supported the results of DLVO theory and AFM measurements. During the flotation process, addition of quartz would lead to attachment of serpentine to the quartz surface, so the adverse effect of serpentine on decreasing floatability of ascharite was weakened.

**Keywords:** *ascharite, serpentine, quartz, interaction, AFM, DLVO*

### **Introduction**

Boron is widely used in chemical and metallurgical industries due to its special chemical and physical properties (Qin et al., 2015). Ascharite is an important raw material for boron industry in China and often associated with serpentine, which is a phyllosilicate mineral with a strong hydrophilic ability. Due to the low hardness, serpentine is prone to be slimed, which decreases ascharite flotation recovery significantly (Li et al., 2007). It was reported that fine serpentine particles attached to

valuable minerals as slime coatings, such as the attachment of serpentine on surfaces of pentlandite, pyrite and chromite (Gallios et al., 2007; Feng et al., 2012; Zhou et al., 2015). Then, the hydrophobicity of valuable minerals was decreased by serpentine particles and resulted in a low flotation recovery.

Several researches have been done on inhibiting the adverse effect of serpentine slime, but most of these researches focused on sulphide flotation. Different kinds of reagents, such as sodium hexametaphosphate, sodium silicate, carboxymethyl cellulose and sodium carbon were used to get rid of the serpentine slime particles on surface of sulphide mineral (Kirjavainen et al., 2007; Lu et al., 2011). Nevertheless, the high reagent dosage needed to disperse serpentine slime led to considerable economic pressure for application. Furthermore, the detrimental effect of serpentine on ascharite flotation was seldom studied before.

References showed that adsorption of colloidal particles was selective and it was related to the mineral surface potential, which played an important role in flotation (Vishal et al., 2011; Yin et al., 2012; Alvarez et al., 2016; Gao et al., 2016). For example, galena flotation was strongly depressed by ferric oxide and alumina particles, but unaffected by kaolinite and gypsum particles. Galena, kaolinite and gypsum used in the research were negatively charged, whereas ferric and alumina were positively charged (Gaudin et al., 1960). Therefore, particles interaction could be used to inhibit the detrimental effect of serpentine during ascharite flotation. Hydrophilic quartz with strong negative charge on the surface was used to remove the serpentine slime on the ascharite surface.

Charges on serpentine and ascharite surfaces are opposite during ascharite flotation. Therefore, we consider that an electrostatic attraction force is dominant between particles interaction leading to serpentine slime covering the surface of ascharite. The coating of serpentine slime particles can decrease the recovery of ascharite. Nevertheless, no direct force measurements have been reported about interactions between serpentine and ascharite.

In the past few years, atomic force microscope (AFM) has emerged as a powerful tool for studying particle surface interaction and property via measurements of force-distance curves in air and solution (Butt et al., 1995; Kishimoto et al., 2008; Kusuma et al., 2014; Gao et al., 2016; Leiro et al., 2017). Many researches showed that AFM results on measuring colloidal forces in solution agreed with the classic Derjaguin-Landau-Verwey-Overbeek (DLVO) theory (Toikka et al., 1997; Trefalt et al., 2014). The colloidal probe technique based on AFM has been normally used to measure the forces between various micrometre-sized rigid solids and surface, and has been extended to study the interaction between a deformable bubble and an oil droplet in flotation systems, as well as between pairs of individual particles (Butt, 1991; Xing et al., 2016; Gui et al., 2016). Although there were deviations caused by either hydration forces or surface roughness at very short distances, the AFM colloidal probe technique has been widely used to measure surface interaction (Bordag et al., 1995; Gupta et al., 2010; Browne et al., 2015).

In this work, quartz was used to inhibit the adverse effect of serpentine during ascharite flotation through particles interaction, and interactions between ascharite, serpentine and quartz were systematically studied at pH 9.0. Besides, AFM was employed to probe the particles interaction forces between mineral particles and different substrates in aqueous solutions for a better understanding of flotation mechanism.

## Experimental

### Samples and reagents

The serpentine and ascharite samples used in the study were obtained from Dandong, Liaoning province, China. The chemical analysis results were shown in Table 1. The images of X-ray diffraction (PANalytical B.V, Netherlands) were listed in Fig. 1. The results of XRD and chemical analysis confirmed that ascharite sample was of a high purity with trace serpentine, and the purity of serpentine sample was 96.5%. The quartz sample was obtained from Anshan, Liaoning province, China. The results of XRD and chemical analysis showed the purity of quartz was 97.8%. The samples were ground and screened respectively, then the particle size distribution was analysed by Malvern Instruments Mastersizer (Mastersizer2000, England) as shown in Table 2.

Table 1. Chemical analysis of serpentine, ascharite and quartz (%)

Samples	B <sub>2</sub> O <sub>3</sub>	MgO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	Fe
Serpentine	0.10	42.09	45.54	0.90	-	2.18
Ascharite	39.16	46.21	0.74	0.25	-	1.87
Quartz	-	-	97.83	0.02	0.03	-

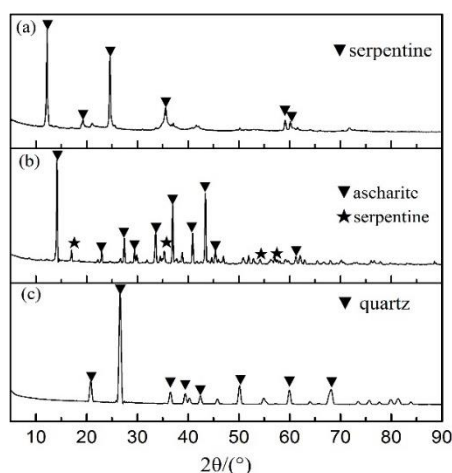


Fig. 1. XRD patterns (a: serpentine, b: ascharite, c: quartz)

Table 2. Particles size distribution of samples

Samples	$D_{10}$ , $\mu\text{m}$	$D_{50}$ , $\mu\text{m}$	$D_{90}$ , $\mu\text{m}$	Average diameter, $\mu\text{m}$
Ascharite	1.8	12.1	43.8	27.8
Serpentine	1.5	11.0	42.3	25.1
Quartz	3.6	41.9	114.1	82.4

Sodium oleate and methyl isobutyl carbinol (MIBC) were used as a collector and a frother, respectively. NaOH and HCl were used as pH regulators. Potassium nitrate was used to maintain the ionic strength in zeta potential measurements. Deionized water was used for all experiments. All the reagents used were of analytical grade.

### Flotation tests

Flotation tests were carried out in a mechanical agitation micro-flotation machine with impeller speed of 1920 r/min. In a single mineral flotation test, the mineral suspension was prepared by adding 2.0 g of single mineral to 30 cm<sup>3</sup> of deionized water. In the flotation tests of mixed minerals, the usage of ascharite was 1.0 g. The reagents addition scheme involved the collector, and then the frother with 3 min conditioning period prior to next reagent addition. The flotation concentrates were collected for 5 min. Then, the concentrates and unfloated particles (tailings) were filtered, dried and analysed.

### Measurements

Zeta potentials of quartz, serpentine and ascharite were measured by a Malvern Zetasizer Nano potential meter. The mineral samples were first ground to less than 2  $\mu\text{m}$ , and then 20 mg of sample was added to 50 cm<sup>3</sup> deionized water. Potassium nitrate was used to maintain the ionic strength at 10<sup>-3</sup> mol/dm<sup>3</sup>. The suspension was magnetically stirred for 10 min and the pH value was adjusted by using HCl and NaOH. The pH values of the suspension were recorded using a pHS-3C pH meter. After that, the supernatant was measured using the potential meter 20 min later to ensure the uniformity of particle size.

The surfaces of serpentine and quartz were polished to meet requirements of AFM measurement at first. Then, slices were cut into pieces of 1×1 cm<sup>2</sup> used as substrates. Before slices were used, they were cleaned by a UV Ozone Cleaner for 10 min and rinsed with deionized water, then dried with ultra-high purity N<sub>2</sub> gas. A standard liquid cell was employed throughout. The cell was cleaned by deionized water and dry with clean compressed air (Karamath et al., 2015). The pH value of solution was 9.0 for measurements.

AFM experiments were conducted by using a MFP-3D AFM (Asy-lum Research, Santa Barabaram, CA). Triangular silicon nitride AFM probes with spring constant of 0.15 N/m were used for force measurements. The spring constant was determined after all force measurements finished. Serpentine and ascharite particle suspensions in water

were dropped on a glass slide and dried in air under an infrared lamp. Epoxy adhesive was placed on a glass slide and a trace amount of epoxy adhesive was picked up by lowering a tipless AFM cantilever (Wang et al., 2016). Then, serpentine and ascharite particles were picked up by the tipless AFM cantilevers with epoxy adhesive, respectively. Then, the resulting assemblies were dried in air for at least 48 h. The force-distance curves were obtained by approaching and retracting the colloid probe against a desired substrate in water. In this research, interaction forces between the particle and substrate were measured to approximately simulate the forces between particles.

The mixed minerals (serpentine and quartz, serpentine and ascharite, ascharite and quartz) were placed in a flotation cell with 30 cm<sup>3</sup> deionized water, respectively. They were stirred for 5 min at pH 9, and the products were dried for detection. The particles surfaces interaction was studied using a SSX-550 scanning electron microscope (Shima-dzu, Japan) and energy dispersive spectroscopy embedded onto the SEM with an Inca X-ray spectrometer (Oxford, England).

## Results and discussion

### Flotation tests

The influence of sodium oleate dosage on floatability of quartz, serpentine and ascharite is shown in Fig. 2. Without collector addition, ascharite had a good natural floatability at pH 9.0. Upon the addition of collector, the ascharite recovery increased remarkably and reached the maximum at 60 mg/dm<sup>3</sup>. The species distribution of sodium oleate in the solution (60 mg/dm<sup>3</sup>) is shown in Fig. 3. The main species of sodium oleate were RCOO<sup>-</sup> and (RCOO<sup>-</sup>)<sub>2</sub><sup>2-</sup> at pH of 9.0. With further sodium oleate addition, there was no significant change in the ascharite recovery. Moreover, quartz and serpentine showed a poor natural floatability and sodium oleate had little effect on their recovery.

The effect of serpentine and quartz on the ascharite flotation recovery is shown in Fig. 4. It can be seen that ascharite recovery without serpentine addition remained at a high level at pH range of 4-11. After addition of 1.0 g of serpentine particles prior collector, the ascharite recovery sharply decreased with increasing of pH values. At pH of 9.0 the ascharite recovery decreased from 88 to 55%, and the recovery of ascharite was less than 40% when pH>10. The results indicated that ascharite recovery could be depressed by serpentine. However, the recovery of ascharite obviously increased at the whole pH range after addition of 0.5g quartz to the pulp. This may be caused by particles interaction between quartz and serpentine.

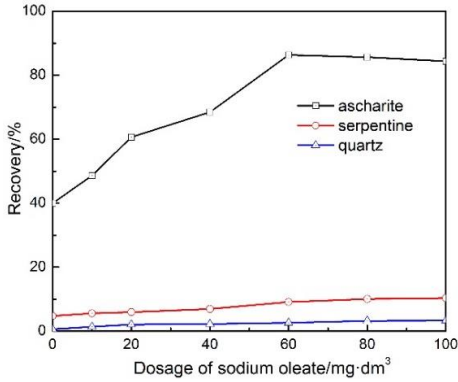


Fig. 2. Effect of sodium oleate dosage on flotation at pH 9

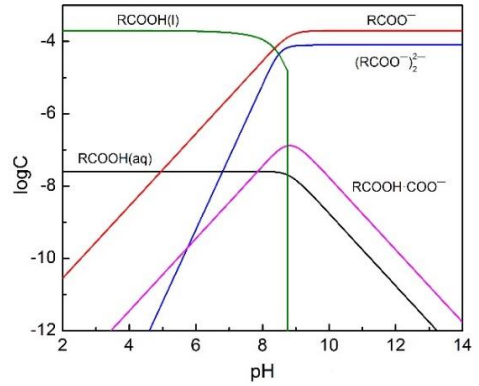


Fig. 3. Species distribution of sodium oleate at a concentration of 60 mg/dm<sup>3</sup>

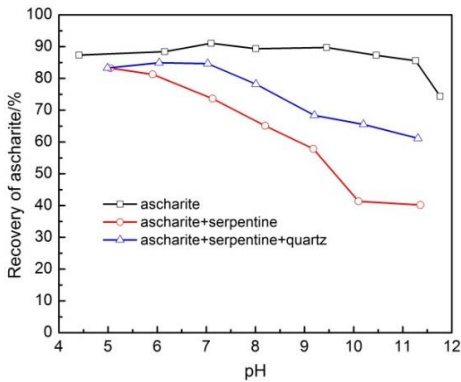


Fig. 4. Effect of serpentine and quartz on ascharite recovery

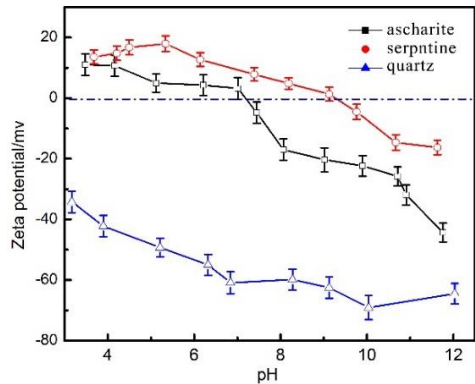


Fig. 5. Zeta potential of ascharite, serpentine and quartz

**Zeta potential**

Zeta potential of quartz, serpentine and ascharite, as a function of pH, is shown in Fig. 5. The results show that serpentine had an isoelectric point (IEP) at pH 9.2 and the zeta potential of serpentine remained positive in the pH range of 4–9.2. The IEP of ascharite was about pH 7.2 and the surface of ascharite was negatively charged in pH range of 7.2–12. In the pH range of 7.2–9.2, positively charged serpentine was likely to attach to the negatively charged ascharite surface through electrostatic attraction (Feng et al., 2012). It can be also seen that the zeta potential of quartz was negative at whole pH range measured and more negative than that of ascharite. At pH > 7.2, both ascharite and quartz were negative charged, so electrostatic repulsive force existed between ascharite and quartz. At pH 9.0, positively charged serpentine particles were more likely to attach to more negatively charged quartz rather than onto the surface of ascharite through electrostatic interaction.

### DLVO theory analysis

Formation of slime coating was controlled by the interaction energy between slime and mineral particles and involved the aggregation process (Feng et al., 2012). Interactions between mineral particles were generally controlled by van der Waals forces and electrostatic interaction, which could be quantitatively predicted by DLVO theory (Sinha et al., 2013; Wang et al., 2016). In addition, quartz was used to improve the recovery of ascharite depressed by serpentine, this is may be caused by interaction between quartz and serpentine particles. References showed that van der Waals forces are generally attractive forces existing between mineral particles (Feng et al., 2015). For serpentine minerals, most studies on formation of slime coating confirmed that the electrostatic force was the main reason leading to slime adhesion to mineral surfaces (Bremmell et al., 2005). Based on the DLVO theory, interactions between slime and mineral particles were dependent on the surface charge of mineral particles. Constant charge boundary condition was applied in DLVO theory analysis in this research.

The particles interaction energy in aqueous solution is described by the DLVO theory, which can make prediction of particles interactions. The total interaction energy is (Feng et al., 2012; Wang et al., 2015):

$$V_T = V_W + V_E \quad (1)$$

where  $V_T$ ,  $V_W$ ,  $V_E$  are the total, van der Waals and electrostatic energies, respectively.

The van der Waals energy can be described as:

$$V_W = -\frac{AR_1R_2}{6H(R_1 + R_2)} \quad (2)$$

where  $R$  is the radius of mineral particle,  $H$  represents the separation distance between particles,  $A$  is the Hamaker constant of two different minerals in an aqueous solution and it can be described as follow:

$$A = (\sqrt{A_{11}} - \sqrt{A_{33}})(\sqrt{A_{22}} - \sqrt{A_{33}}) \quad (3)$$

where  $A_{11}$ ,  $A_{22}$  represent Hamaker constants of minerals in the vacuum. The Hamaker constants of quartz and serpentine are  $5.0 \times 10^{-20}$  and  $10.3 \times 10^{-20}$  J, respectively (Lu et al., 2015).  $A_{33}$  represents the Hamaker constant of water in the vacuum and is equal to  $A_{33} = 4.15 \times 10^{-20}$  J (Feng et al., 2012). However, the Hamaker constant of ascharite is unknown. The relationship between the Hamaker constant and the surface free energy is (Yin et al., 2014):

$$A = \frac{4\pi}{1.2} \gamma_{sv}^d D^2 \quad (4)$$

where  $\gamma_{SV}^d$  is the non-polar component of surface free energy,  $D$  is equilibrium separation between particles. The value of  $\gamma_{SV}^d$  can be obtained by measuring the contact angle of ascharite in two different test liquids (Yin et al., 2014). The relationship between the contact angle and  $\gamma_{SV}^d$  is:

$$\gamma_{LV}(1 + \cos \theta) = 2(\sqrt{\gamma_{SV}^d \gamma_{LV}^d} + \sqrt{\gamma_{SV}^p \gamma_{LV}^p}) \quad (5)$$

where  $\theta$  is the contact angle,  $\gamma_{LV}$  represent the surface free energy between solid and liquid surface,  $\gamma_{SV}^p, \gamma_{LV}^p$  are polar components of surface free energies of ascharite and the test liquid, respectively;  $\gamma_{SV}^d, \gamma_{LV}^d$  are the non-polar components of surface free energies of ascharite surface and the test liquid, respectively. The surface free energy parameters of test liquids are listed in Table.3. Contact angles of ascharite in water and ethanediol are 24.8 and 31.7°, respectively. Replacing data into Eq.(4) and (5), the Hamaker constant of ascharite in vacuum was calculated to be  $A_{44}=19.3 \times 10^{-20}$  J. the Hamaker constant for quartz and serpentine in aqueous solution was  $A_1 = 0.24 \times 10^{-20}$  J; the Hamaker constant for quartz and ascharite in aqueous solution was  $A_2 = 0.47 \times 10^{-20}$  J, while the Hamaker constant for serpentine and ascharite in aqueous solution was  $A_3 = 2.76 \times 10^{-20}$  J.

Table 3. The surface free energy parameters of test liquids

Liquid	$\gamma_{LV}, \text{mJ} \cdot \text{m}^{-2}$	$\gamma_{LV}^d, \text{mJ} \cdot \text{m}^{-2}$	$\gamma_{LV}^p, \text{mJ} \cdot \text{m}^{-2}$
Water	72.8	21.8	51.0
Ethanediol	58.2	29.3	19

The electrostatic energy can be calculated as

$$V_E = \frac{\pi \varepsilon_a R_1 R_2}{R_1 + R_2} (\varphi_1^2 + \varphi_2^2) \left( \frac{2\varphi_1 \varphi_2}{\varphi_1^2 + \varphi_2^2} \times \ln \frac{1 + e^{-\kappa H}}{1 - e^{-\kappa H}} + \ln(1 - e^{-\kappa H}) \right) \quad (6)$$

where  $\kappa$  is the thickness of electric double-layer,  $\kappa = 0.180 \text{ nm}^{-1}$  (Lu et al., 2011);  $\varepsilon_a$  is the relative dielectric constant of the continuous phase,  $\varepsilon_a = 6.95 \times 10^{-10} \text{ C}^2 \text{ J} \cdot \text{m}$  (Wang et al., 2015);  $\varphi_1$  and  $\varphi_2$  are the surface potentials (mV). The zeta potential of quartz, serpentine and ascharite were based on the experimental data, and they were -62.5, 1.9 and -20.4 mV at pH of 9, respectively.

The curves of total interaction energy are shown in Fig. 6. The total energy between serpentine and ascharite was negative, which indicated that the force between serpentine and ascharite particles was attractive, and serpentine easily adhered to the surface of ascharite.



Figure 6 also shows that the total interaction energy between serpentine and quartz was negative. It reveals that serpentine particles were prone to attach to the quartz surface. In addition, the total interaction energy between quartz and ascharite was positive, which reflected the disperse state between quartz and ascharite in water. Therefore, after the addition of quartz to the pulp, quartz particles adhered to serpentine and inhibited the adhesion of serpentine on the ascharite surface. As a result, addition of quartz was benefit for improvement of ascharite recovery, which was consistent with the flotation results.

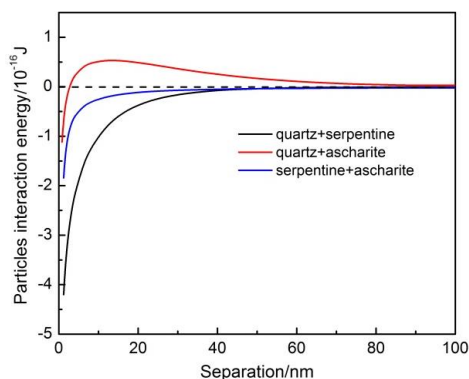


Fig. 6. Particles interaction energy-separation curves

## Results of AFM measurements

Although AFM is a powerful tool for measuring and calculating the particle surface interaction, there are also some limitations. Owing to the irregular geometry and surface heterogeneity of particle surface, the measurement results cannot be explained quantitatively with the standard DLVO theory of colloid stability. Consequently, the force curves are only qualitatively compared, and we restrict our discussion to qualitative speculations. This procedure has previously been successfully used to study the coal particle–bubble interaction and the presence of the nanobubbles and/or slime removal on ZnS surface after methanol treatment (Holuszko et al., 2008; Xing et al., 2016; Gui et al., 2016).

The force-distance curves between ascharite and serpentine are listed in Fig. 7. It reveals that the force between ascharite particle and serpentine was attractive, and it became weak gradually as the distance increased. Therefore, after adding serpentine into the ascharite pulp, serpentine particles attached to the surface of ascharite leading to decrease in the ascharite recovery.

Figure 8 shows the approaching force-distance curves between ascharite particle and quartz substrate. It indicates that the force between ascharite particle and quartz was repulsive. With the distance increasing, the repulsive force decreased gradually. Therefore, quartz would not attach to ascharite and would be well dispersed with ascharite in the aqueous solution.

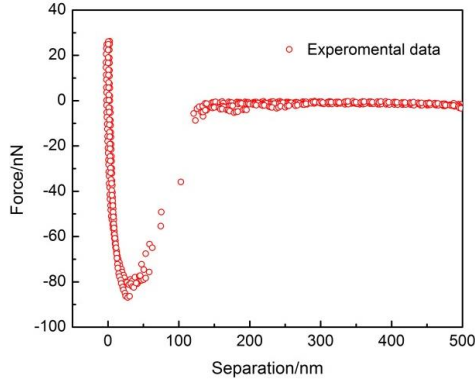


Fig. 7. Force-distance curves for ascharite particle approaching serpentine substrate

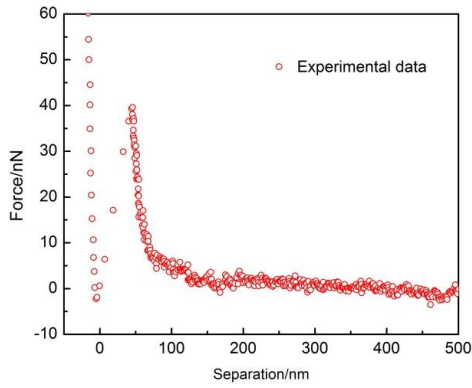


Fig. 8. Force-distance curves for ascharite particle approaching quartz substrate

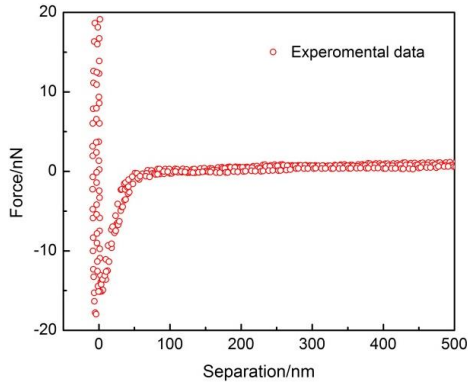


Fig. 9. Force-distance curves for serpentine particle approaching quartz substrate

The force-distance curves between serpentine particle and quartz substrate are shown in Fig. 9. The force between serpentine and quartz was attractive and it

gradually decreased with the increase of separation distance. Therefore, quartz would adhere to serpentine easily.

It is well known that the van der Waals force is a short-range attraction force that is widespread. In this research, the charges on the surface of serpentine, ascharite and quartz are opposite at pH 9.0, so electrostatic forces between serpentine and ascharite as well as serpentine and quartz are attraction forces. The ascharite and quartz are both negative charged in solution at pH 9.0, so electrostatic repulsive force was a predominate role between these two minerals.

Based on the results of AFM measurements, we know that serpentine can attach to the surface of ascharite and quartz, but quartz is dispersed with ascharite. After addition of quartz into the pulp, serpentine interacts with quartz and attaches to its surface, which inhibits aggregation between serpentine and ascharite. In this way, the adverse effect of serpentine on ascharite is reduced and results in a remarkable improvement of ascharite recoveries. The AFM experiments results are coincident with flotation results and DLVO theory analysis.

### SEM and EDS

The SEM and EDS images of particle surfaces are shown in Fig. 10. Figure 10(a) shows the interaction between serpentine and ascharite, as well as that the serpentine particles were detected on the surface of quartz. The attachment of serpentine on the ascharite surface was also observed in Fig. 10(b). However, no quartz particles were found on the ascharite surface, as shown in Fig.10 (c). It means that quartz would not attach to ascharite. The images demonstrated the validity of the results of DLVO theory and AFM force measurements.

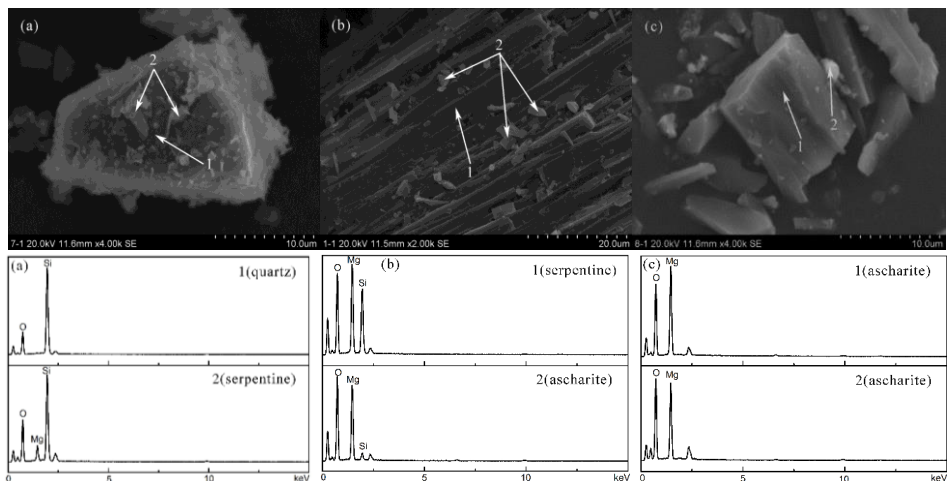


Fig. 10. SEM and EDS images of interactions between (a) serpentine and quartz, (b) ascharite and serpentine, and (c) ascharite and quartz

## Conclusions

Ascharite recovery decreased seriously in the presence of serpentine. Quartz can be used to inhibit the adverse effect of serpentine on ascharite flotation by particles interaction, leading to an improvement of ascharite recovery.

The results of DLVO theory analysis showed that the particle interaction energy between serpentine and ascharite, as well as serpentine and quartz was negative. However, the interaction energy between ascharite and quartz was positive. The AFM force measurements and SEM analysis results were consistent with the DLVO theory. Attraction forces between ascharite and serpentine, quartz and serpentine led to aggregation. The electrostatic repulsive force prevented the attachment of quartz on ascharite, and the particles had the tendency to disperse in water. Therefore, a part of serpentine particles attached to the quartz surface after the addition of quartz to the pulp. Then, the adverse effect of serpentine on ascharite flotation was weakened, which led to increase in the ascharite recovery.

## Acknowledgment

This work was supported by the National Natural Science Foundation of China under grant number 51204033.

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