Dispersive effect of low molecular weight sodium polyacrylate on pyrite-serpentine flotation system

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Abstract: In this work, the dispersive effect of low molecular weight sodium polyacrylate (PAAS) on serpentine, and its dispersion mechanism were systematically investigated through zeta potential measurements, micro and batch flotation as well as adsorption tests. At pH 5, where flotation of iron sulphide was routinely performed, pyrite and serpentine minerals were oppositely charged, and therefore they were attracted to each other. Slime coatings of serpentine adhered to the surface of pyrite, decreasing the adsorption density of a collector on the pyrite surface, but also reducing the flotation recovery. PAAS increased the flotation recovery of pyrite by promoting dispersion between pyrite and serpentine. The effective flotation separation of pyrite from the refractory iron sulphide ore was possible by using PAAS as a dispersant. Anionic PAAS adjusted the surface potential of serpentine through adsorption on the serpentine surface and changed the interaction between pyrite and serpentine particles from attractive to repulsive, and then dispersed pyrite and serpentine.

Introduction

Dispersing modifiers are used to prevent fine particles from aggregating and for reducing the pulp viscosity in many cases. Either slime coating or aggregation phenomenon is common where slimes are presented in the pulp, and has a negative effect on flotation (Bulatovic, 2007; Wills and Finch, 2016).

The Shaopo concentrator (Yunnan, China) processes iron sulphide ores using froth flotation to produce a concentrate containing 48% S, 2.9% MgO, and 3.7% SiO_2. Then, the concentrate is smelted at the sulphuric acid fluidized bed roaster by burning off sulphur (to produce sulphuric acid) as a slag phase to produce an iron concentrate

containing 62% Fe and 0.2% S (Liu and Wen, 1996). Serpentine is the main magnesium-rich phyllosilicate mineral in the Shaopo iron sulphide ore. Phyllosilicate group minerals pose particular problems (Ndlovu et al., 2014), especially for serpentine, which can form tangled mats that entrap bubbles and float into the froth (Bhambhani et al., 2012). Moreover, serpentine is easily slimed due its low hardness in nature. The fine slimes possess a more positive charge than that of the coarse sulphide particles. This creates electrostatic attraction which results in the fine slimes forming coating over the surface of sulphide particles (Edwards et al., 1980; Pietrobon et al., 1997; Feng et al., 2015a). Coating of slime particles reduces both the flotation rate and recovery of the valuable sulphide minerals (Trahar, 1981; Learmont and Iwasaki, 1984).

The ultra-fine slimes, as well as other clay minerals, have different surface properties in comparison with coarse particles with respect to the surface energy and behaviour (Bankoff, 1943). In a number of investigations (Wellham et al., 1992; Bulatovic, 2007; Feng et al., 2012a,b), it is demonstrated that electrostatic forces are involved in slime clay adsorption on the desired mineral surfaces, where the oppositely charged target mineral and slime particles are attracted to each other. The pioneer work of Sun (1943) showed that the slime coating got dense when the zeta potential values of slimes and minerals were dissimilar in the magnitude and sign. The previous works (Gaudin et al., 1960; Smart, 1991) also demonstrated that an electrostatic mechanism was prominent in slime coating by showing that galena flotation was strongly depressed by ferric oxide and alumina particles which were positively charged at the pH range used in the study.

The action of dispersing agents in mineral processing is to improve floatability of mineral by preventing slime coating on mineral surfaces (Bulatovic, 2007). In order to limit the effect of ultra-fine slimes on sulphide flotation, sodium hexametaphosphate, sodium silicate, sodium carbonate and carboxymethyl cellulose have been used to disperse fine slimes and to remove slimes from sulphide surfaces (Bremmell et al., 2007; Kirjavainen and Heiskanen, 2007; Lu et al., 2012; Feng et al., 2012b,c, 2015b; Khraisheh et al., 2015; Wills and Finch, 2016). However, dispersion of slime particles requires more reagent dosages which increases production costs (Feng et al., 2012a; Zhou, 2015). Meanwhile, the conventional dispersants usually suffered the disadvantage of low selectivity and low performance (Pereira and Peres, 2005). Therefore, it is urgently demand to develop easily available, selective and high performance dispersants for dispersion of slime minerals, particularly for clays.

Technological innovations have been made, and a low molecular weight sodium polyacrylate (PAAS) dispersant into sulphide flotation has been introduced successfully. The low molecular weight PAAS has been widely used in dispersion of coating pigments and ceramic ferrous powder as well as used in the process of paperboard making (Wu et al., 2004; Huang et al., 2005). The previous work of Boisvert et al. (2000, 2001) showed that PAAS (with molecular mass MW 20000) can fully disperse the alumina-coated TiO_2 particles used as pigments in papers and paints.

However, PAAS, as a dispersant, has not been reported yet for dispersion of serpentine slimes in sulphide flotation, and the theoretical investigation on the dispersion mechanism between PAAS and serpentine has not been studied previously. In the previous micro-scale flotation research (Feng et al., 2012c, 2015a), pure minerals were used, while this study was based on a real FeS_2 ore for flotation separation.

In the present study, the low molecular weight PAAS was used to disperse serpentine slimes effectively and to limit the detrimental effect of serpentine on pyrite flotation. The dispersion mechanism of PAAS on serpentine was also investigated. The experimental part included flotation tests on single and mixed binary minerals as well as a real ore sample.

Materials and methods

Materials

The pure pyrite sample obtained from the Shaopo iron sulphide Mine of Yunnan Province, China was used for the experiments. The chemical analysis showed that the purity of pyrite was 93.7%, and it contained 50.1% S and 44.5% Fe. The samples were dry ground and screened to obtain the desired size fractions for the experiments. The -75+38 μm fraction was used for the flotation tests, and -25 μm for adsorption and zeta potential measurements. The pyrite sample was stored in a vacuum-desiccator.

The pure serpentine sample was obtained from Wenzhou, Zhejiang Province, China. The mineral composition of the serpentine sample was determined by XRD. The results showed that the sample contained 95.5% serpentine and 4.5% chlorite. The sample was dry ground and screened. The particle size distribution of the sample was determined using a Malvern Instruments Mastersizer. According to this analysis, the sample was 100% below 25 μm with an average diameter of 21.2 μm.

The ore for a batch flotation test was a refractory iron sulphide ore (skarn -type, containing serpentine, d_{80} size of feed from plant and ground material under 75 μm) from the Shaopo iron sulphide Mine of Yunnan Province, China. The main iron-bearing minerals were pyrite, and the liberation size of pyrite was below 75 μm. XRD spectra of the iron sulphide ore (Fig. 1) confirmed that the significant amounts of non-sulphide gangue minerals were serpentine, chlorite, calcite, dolomite, quartz and muscovite, especially for serpentine, it accounted for 30–35% of the total minerals. The result of multi-elemental chemical analysis of the ore is listed in Table 1 (the sulphur content was determined by a barium sulphate gravimetric method).

The sample of sodium polyacrylate PAAS, MW 800, $(C_3H_3NaO_2)_n$) was obtained from Tianjing Weiki Chemical Reagents Development Centre, Tianjing, China. Potassium amyl xanthate (PAX) and methyl isobutyl carbinol (MIBC) were used as a collector and a frother, respectively. Potassium nitrate (KNO_3) was used to maintain the ionic strength, and hydrochloric acid (HCl) and sodium hydroxide (NaOH) were

used as pH regulators. Deionized double distilled water was used for the micro-flotation tests, adsorption and zeta potential measurements.
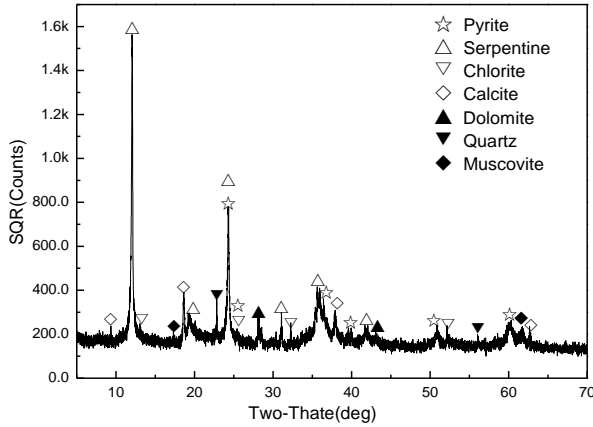

Fig. 1. XRD spectra of iron sulphide ore used for batch flotation tests

Table 1. Results of multi-elemental chemical analysis of ore for batch flotation tests

Element	S %	MgO %	SiO$_2$ %	Fe %	CaO %	Al$_2$O$_3$ %	TiO$_2$ %	Cu %	K$_2$O %	Na$_2$O %	TREO[*] (g/Mg)	Nb$_2$O$_5$ (g/Mg)
Grade	15.30	22.47	32.22	13.46	7.58	5.51	1.04	0.05	0.15	0.38	560	192

[*] content of rare earth metals

PAAS, sodium hexametaphosphate (SHMP) and sodium silicate (SS) were used as the dispersants, guar gum as a depressant, and oxalic acid as a pH regulator for the batch flotation tests. Tap water was used for the laboratory flotation tests. All reagents used in this study were of analytical grade.

Methods

Micro-flotation tests

Micro-flotation tests were carried out in an XFG-type flotation machine with a 40 cm^3 Plexiglas cell, and the impeller speed was set to 1800 rpm. The mineral suspension was prepared by adding 2.0 g of pyrite to 40 cm^3 of distilled water. When needed, serpentine was added at the beginning of the conditioning period. The pH of the mineral suspension was adjusted to the desired value by adding either HCl or NaOH stock solutions. Pulp pH was recorded by a LeiCi PHS-3 pH meter. Subsequently, the dispersant, collector and frother were added in the sequence to the Plexiglas cell, and having a 3 min conditioning period with air prior to the next reagent addition, respectively. The conditioned slurry was floated for 4 min. Both the froth product and tailing were collected, filtered and dried. The flotation recovery was calculated based on either solid weight distributions or S grade between two products. In order to assess

the accuracy of the flotation tests, the errors of the recovery were found to be within 2% after at least five tests under each condition, and the average values were reported.

Batch flotation tests

The bulk of laboratory test work was carried out in batch flotation cells (XFD-63) with 500 g sample. The cell with the volume of 1500 cm^3 for rougher and 1000 cm^3 for cleaning flotation, respectively, was mechanically agitated at the speed of 1500 rpm. After wet grinding, the desired amounts of reagents were added to the slurry and flotation was started with the injection of air into the flotation cell, and then the concentrates were collected. The concentrates and tailings products were filtered, dried, weighted, sampled and assayed for S. To assess the accuracy of the flotation tests, the calculated S grade of feed should be in the range of 15.3± 0.1%, otherwise the flotation tests were repeated (Zhao et al., 2015).
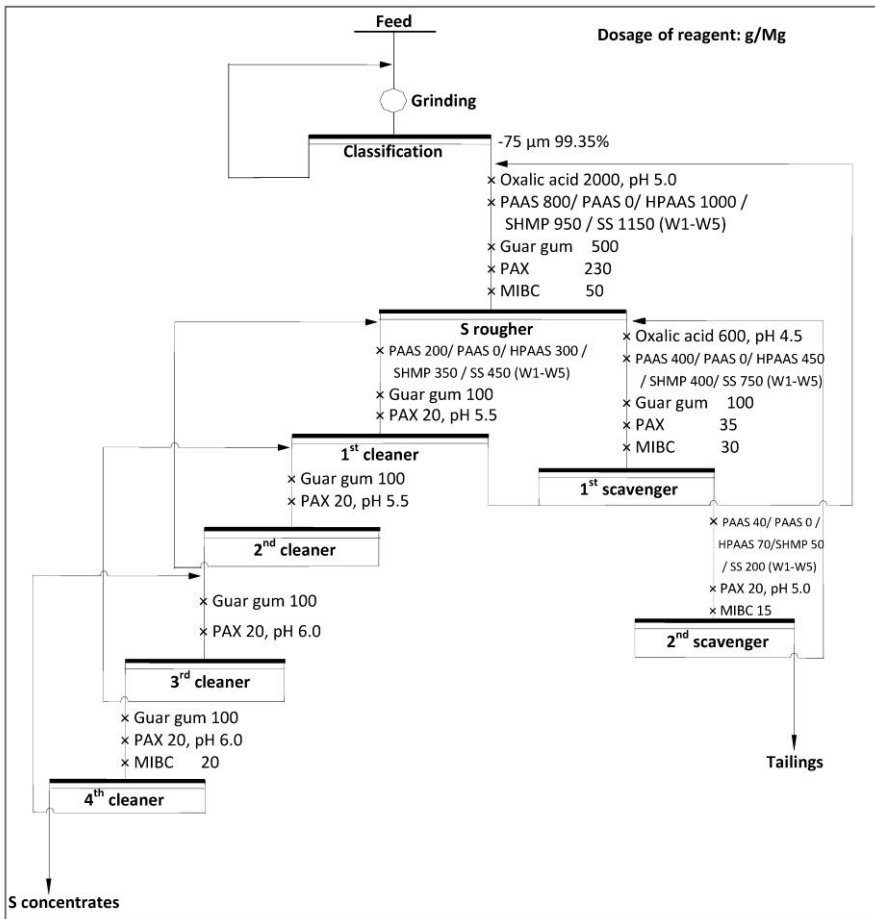

Fig. 2. Flowsheet and corresponding conditions of bench-scale locked cycle tests

The flowsheet and corresponding conditions of the locked cycle tests are shown in Fig. 2. The experimental system W1 used PAAS (MW 800) as the dispersant (the total dosage of PAAS was 1440 g/Mg, 800 g/Mg for rougher operation), PAAS was not added in the comparative system W2 (without PAAS), the comparative system W3 used high molecular weight PAAS (HPAAS, MW 1500) as the dispersant (the total dosage of PAAS was 1820 g/Mg, 1000 g/Mg for rougher operation), the comparative system W4 used SHMP as the dispersant (the total dosage of SHMP was 1750 g/Mg, 950 g/Mg for rougher operation), and the comparative system W5 used SS as the dispersant (the total dosage of SS was 2550 g/Mg, 1150 g/Mg for rougher operation).

Adsorption tests

In the adsorption tests, 1.0 g single mineral was added into a 300 cm^3 Erlenmeyer flask, and made up to 100 cm^3 after adding PAX solution at desired concentration. The suspension was mixed and placed on a rotator for 5 min. The sample was then centrifuged and the dosage of PAX left in the solution was analysed by a TU1810 UV–Vis spectrophotometer absorbance at 302 nm (Long, 2011). It was assumed that the concentration of PAX depleted from the solution adsorbed onto the mineral phase.

Zeta potential measurements

Zeta potential measurements for pyrite and serpentine were carried out using a Coulter DELSA440S II Type electro-kinetic instrument. KNO_3 was used to maintain the ionic strength at 0.01 mol/dm^3. A sample of 0.05 g single mineral was added to 50 cm^3 solution, and then ultrasonicated for 3 min, magnetically stirred for 10 min, and pH was adjusted using either HCl or NaOH. Then, the zeta potential of samples was measured. The measurement errors were found to be within 5 mV after at least three measurements under each condition, and the average values were reported.

Results and discussion

Effects of PAAS on flotation of pure pyrite sample in the presence of serpentine

The effect of PAX dosage on floatability of pyrite and serpentine at pH 5 is shown in Fig. 3. It indicates that upon collector addition, the pyrite recovery markedly increases up to the PAX dosage at $2 \cdot 10^{-4}$ mol/dm^3, with an increase of PAX dosage producing little gain in the recovery. Serpentine has little floatability, and PAX addition has slight effect on its flotation. The effect of pH on the flotation recovery of pyrite and serpentine is shown in Fig. 4. It is clear that the flotation recovery of serpentine is independent of pH, and the recovery of serpentine is low (20%) in the pH range studied, which is in the agreement with earlier observations (Feng et al., 2012a,b, 2015a). Inversely to serpentine, the flotation recovery of pyrite is very high under acidic and neutral conditions. As the pH value is increased, the pyrite recovery decreases sharply due to formation of hydrophilic iron oxy-hydroxy species on the pyrite surface (Bicak et al., 2007; Feng et al., 2012c).
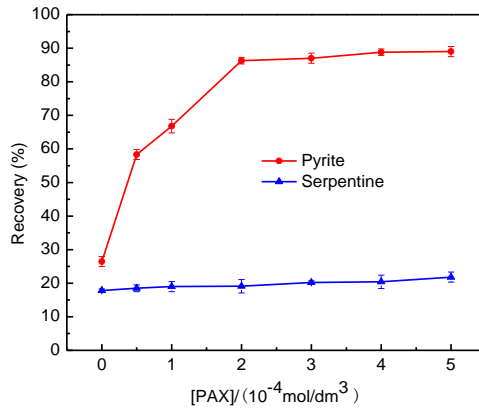
Fig. 3. Effect of PAX dosage on flotation of pyrite and serpentine.
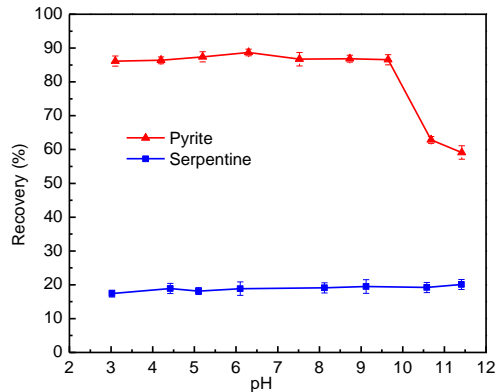Pyrite = $5 \cdot 10^4$ mg/dm^3, serpentine = $5 \cdot 10^4$ mg/dm^3, pH 5, MIBC = 9 mg/dm^3

Fig. 4. Effect of pH on flotation of pyrite and serpentine.
Pyrite = $5 \cdot 10^4$ mg/dm^3, serpentine = $5 \cdot 10^4$ mg/dm^3, PAX = $2 \cdot 10^{-4}$ mol/dm^3, MIBC = 9 mg/dm^3

The effect of serpentine/ PAAS on the flotation recovery of pyrite as a function of pH is shown in Fig. 5. It is clearly seen that serpentine particles interfere with flotation of pyrite, when fine serpentine particles were added prior to collector addition (mixed binary mineral system). The recovery of pyrite decreased with increasing pH from the maximum recovery of 87 to 20%. On the other hand, PAAS had little effect on the pyrite recovery in the whole range of pH values. Figure 6 also indicates that the addition of PAAS could restore the recovery of pyrite which was depressed by serpentine. At pH 5, where flotation of iron sulphide ore was routinely performed, the recovery of depressed pyrite increased from 32 to 83% when 25 mg/dm^3 PAAS was added.

Figure 6 presents the effect of PAAS dosage on flotation of pyrite depressed by serpentine at pH 5. It is interesting to note that the addition of PAAS could restore the

recovery of pyrite, and the maximum increase in the pyrite recovery was obtained with 25 mg/dm^3 PAAS, while higher PAAS dosages slightly reduced the pyrite recovery. Figure 7 also shows that the recovery of serpentine decreased slightly with the increasing of PAAS concentration, indicating that the PAAS had no depressed effect on serpentine flotation.
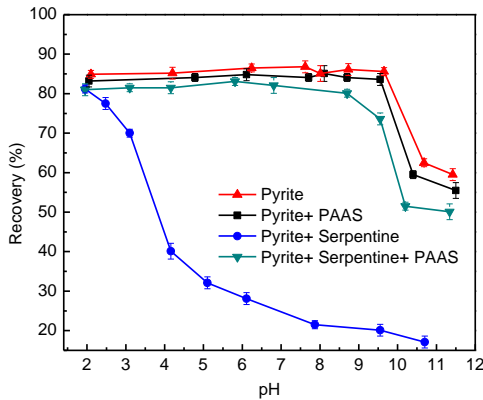
Fig. 5. Flotation recovery of pyrite as a function of pH under different conditions.
Pyrite = 5·10^4 mg/dm^3, serpentine = 1.25·10^4 mg/dm^3, PAAS = 25 mg/dm^3,
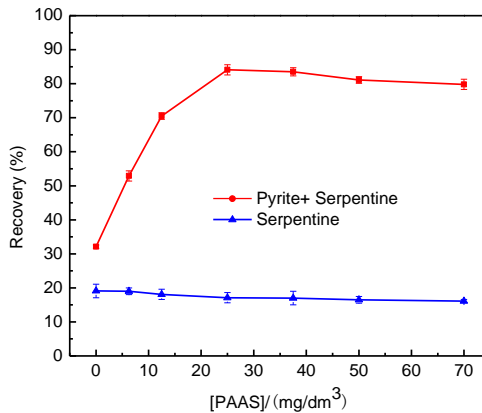PAX = 2·10^{-4} mol/dm^3, MIBC = 9 mg/dm^3

Fig. 6. The effect of PAAS dosage on flotation recovery of pyrite and serpentine.
Pyrite = 5·10^4 mg/dm^3, serpentine = 1.25·10^4 mg/dm^3, pH 5, PAX = 2·10^{-4} mol/dm^3, MIBC = 9 mg/dm^3

Batch flotation tests (locked cycle tests)

Because of the good flotation performance for pyrite, PAAS was selected as a slime dispersant in the batch scale tests. Based on the optimized flotation conditions (there are a large number tests that have been performed to get the comparison of dispersants such as different polyacrylates and dosages, etc., both high level grade and recovery

are selected), the locked cycle tests of pyrite flotation were carried out. The flowsheet and conditions are described in Fig. 2. The locked cycle tests results are shown in Table 2, Figs. 7 and 8.

The results presented in Fig. 7 indicate that when compared to the comparative system, the S grade and recovery increased dramatically when the locked cycle tests were carried out in the W1 system. The XRD spectrum (Fig. 8) confirmed that the S concentrates (pyrite) were over 93% pure. It is clear from Table 2 that when compared to the comparative system W2, the experimental system W1 with the PAAS scheme increased the grade and recovery of sulphur concentrates by 3.12% and 2.01%, respectively. For this complex iron sulphide ore (containing serpentine), PAAS showed an excellent dispersion for serpentine slimes, which is demonstrated by the high level grade (50.24%) and high level recovery (97.11%) of sulphur achieved with PAAS as the dispersant.

Table 2. Results of bench-scale locked cycle tests

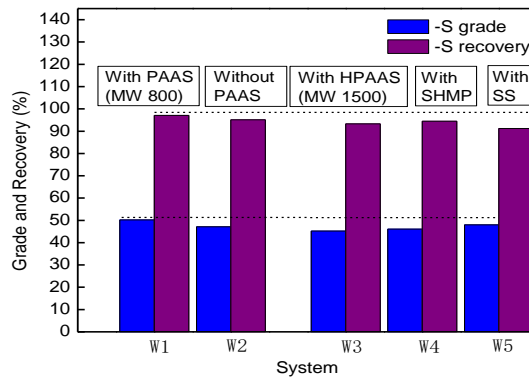| System	Products	Ratio (w/%)	Grade (%)		Recovery (%)	
			S	Fe	S	Fe
W_1 (with PAAS)	S concentrates	29.57	50.24	43.63	97.11	95.92
	Tailings	70.43	0.63	0.78	2.89	4.08
	Feed	100.00	15.35	13.45	100.00	100.00
W_2 (without PAAS)	S concentrates	30.84	47.12	40.95	95.10	93.69
	Tailings	69.16	1.08	1.23	4.90	6.31
	Feed	100.00	15.31	13.48	100.00	100.00

Fig. 7. Differential values of sulphur grade and recovery from locked cycle tests between experimental and comparative system

The mechanism of PAAS limits the detrimental effect of serpentine on pyrite flotation. Figure 10 shows the results of adsorption of PAX on the pyrite surface in the presence of serpentine and PAAS. It can be seen from Fig. 9 that the presence of

serpentine slimes decreased the adsorption density of PAX on the pyrite surface, especially in the weak acidic pH range. This may be the main reason of serpentine interference with pyrite flotation. With the addition of PAAS, the adsorption density of PAX on the pyrite surface increased, illustrating that PAAS could limit the detrimental effect of serpentine slimes in the decreasing collector adsorption on the pyrite surface.
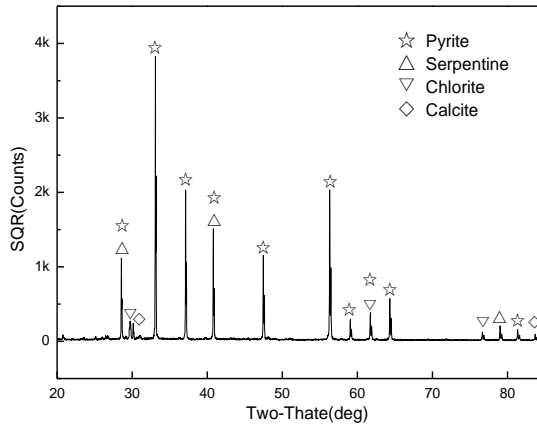

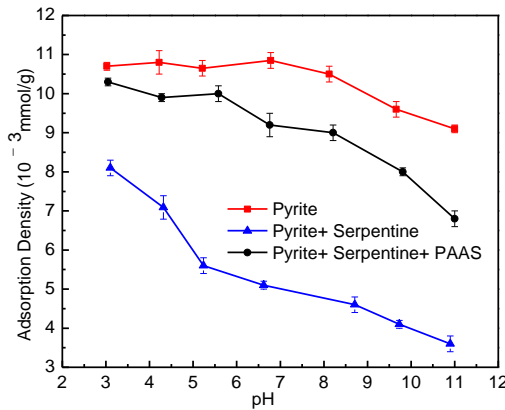Fig. 8. XRD spectrum of concentrate from locked cycle tests

Fig. 9. Adsorption of PAX on pyrite under different conditions.
Pyrite = $1 \cdot 10^4$ mg/dm^3, serpentine =$2.5 \cdot 10^3$ mg/dm^3, PAAS = 25 mg/dm^3, PAX = $2 \cdot 10^{-4}$ mol/dm^3

Electrostatic forces are involved in slime clay adsorption on the desired minerals surface, where oppositely charged mineral and slime particles are attracted to each other (Jewet and Pearse, 1956; Wellham et al., 1992). Formation of slime coatings is controlled by the interaction energies between the slime and mineral particles and involves the process of aggregation. Aggregation between sulphide mineral particles

and fine slime species is generally controlled by double layer interactions such as van der Waals forces. The forces existing between dissimilar particles are generally attractive (Li, 1993; Huynh et al., 2000). For the same flotation system, the presence of slimes on mineral surfaces is dependent on the surface charge of the particles (Feng et al., 2012a).

As a direct surface charge investigation of the different minerals, individual electrostatic studies of pyrite and serpentine particles as a function of pH are performed. The results presented in Fig. 10 indicate that serpentine has the isoelectric point (IEP) at pH 10. The zeta potential of serpentine is positive in the pH range of 2-10. This is consistent with previous work of Feng et al. (2012a,b). The IEP of pyrite is at pH 1.9, and the surface of pyrite is negatively charged in the pH range of 1.9-12 (Lu et al., 2011; Feng et al., 2015a). At pH values larger than 1.9, it is likely that the positively charged fine serpentine particles will attach to the negatively charged pyrite particle surface through the electrostatic attractions.

Figure 11 also indicates that after interacting with anionic PAAS, the zeta potential of original serpentine has a large negative shift, and the IEP of serpentine shifts from pH 10 to pH 3.8, which shows that PAAS could adsorb on the surface of serpentine and changes the surface potential of serpentine causing it to become more negative. On the other hand, PAAS has a little effect on the surface potential of pyrite across the entire pH range. Therefore, at pH 5, where flotation of iron sulphide ore is routinely performed, pyrite and serpentine minerals are all negatively charged, and therefore repulse each other through electrostatic exclusion, illustrating that PAAS could promote dispersion of serpentine particles, and therefore diminish the effect of serpentine slimes on pyrite flotation, which explains the flotation results.
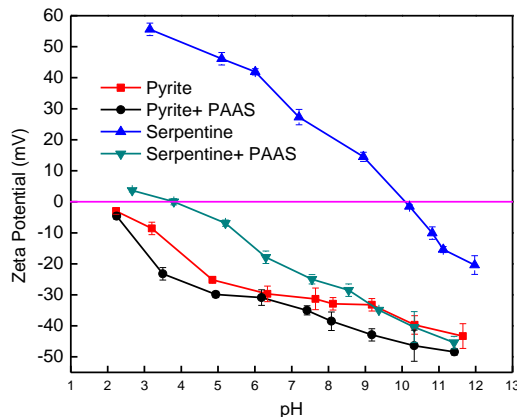

Fig. 10. Zeta potential of pyrite and serpentine as a function of pH
in the presence and absence of 25 mg/dm^3 PAAS with $KNO_3 = 0.01$ mol/dm^3

Based on the mechanism studies, it is concluded that anionic PAAS diminishes the effect of serpentine in decreasing collector adsorption on the pyrite surface and it

could adjust the surface charge of serpentine through adsorption on the serpentine surface and changes the interaction energy between pyrite and serpentine particles from attractive to repulsive energy, and then disperses pyrite and serpentine. Thus, the detrimental effect of serpentine slimes on flotation of pyrite is limited by the addition of PAAS.

Conclusions

1. As serpentine had a positive potential under weak acidic conditions, it could therefore attach through an electrostatic mechanism to the negatively charged pyrite surface. The presence of serpentine slimes on the pyrite surface reduced collector adsorption and resulted in the lower recovery of pyrite.

2. PAAS could diminish the effect of serpentine slimes in decreasing collector adsorption on the pyrite surface, and therefore increased the recovery of pyrite by promoting dispersion between pyrite and serpentine.

3. Effective flotation separation of pyrite from the refractory iron sulphide ore was realized by using PAAS as a dispersant, which was demonstrated by the high level grade (50.24%) and recovery (97.11%) of sulphur achieved in the PAAS system.

4. The surface potential played an important role in removal of serpentine slimes. Anionic PAAS adjusted the surface charge of serpentine through selective adsorption on the serpentine surface, and changed the interaction between pyrite and serpentine particles from attractive to repulsive, and then dispersed pyrite and serpentine.

Acknowledgements

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