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Reducing entrainment of sericite in fine flaky graphite flotation using polyaluminum chloride

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Abstract: Polyaluminum chloride (PACl) was introduced as a flocculant to reduce the entrainment of sericite gangue in fine flaky graphite flotation. The dispersion and aggregation behaviours of sericite and fine flaky graphite in the absence and presence of PACl were studied by settling experiments and verified by optical microscope images. Results of batch flotation tests for artificial mixtures indicated that the addition of PACl decreased the recovery of sericite gangue and the water recovery. Zeta potential measurements, FTIR and XPS analyses revealed that PACl selectively aggregated sericite particles in terms of charge neutralization and precipitate enmeshment.

Keywords: fine flaky graphite, sericite, polyaluminum chloride (PACl), dispersion, aggregation

1. Introduction

Graphite as an non-metallic material is widely used in many industries such as batteries, refractories, electrical products, and pencils (Li et al., 2003). Natural graphite is found in three commercial varieties that are crystalline flake, microcrystalline or amorphous, and crystalline vein or lump (Crossley, 2000). Several research projects about graphite beneficiation have been conducted; till now, flotation is one of the most common and cost-effective methods due to the natural floatability of graphite (Dong, 1997; Chehreh et al., 2016). However, during the conventional flotation process of flaky graphite, which makes up 49% of the world total graphite products (Chehreh et al., 2016), it is almost impossible to avoid the mechanical entrainment of hydrophilic gangue into the graphite concentrate (Li et al., 2015a).

Sericite, a fine-grained mica that belongs to the layered silicate minerals, is one of the main gangue minerals in graphite ore and it is also the main contaminant in graphite flotation concentrate (Li et al., 2015a). It possesses both mica and clay minerals characteristics and naturally interlocked with graphite (Li et al., 2015b). The separation efficiency between the graphite and the fully liberated and dispersed sericite depends on the degree of entrainment (Ross, 1990). Unlike true flotation, which is selective, both gangue and valuable minerals can be recovered by entrainment (Wang et al., 2015). As a result, the quality of the final graphite concentrate is often significantly reduced and this phenomenon is particularly severe when both graphite and sericite particles are ground fine or ultra-fine after multistage grinding and flotation. Therefore, reducing the entrainment of sericite will effectively improve the flotation selectivity of graphite ores.

Studies on the entrainment behaviour have been conducted for over 30 years. In graphite flotation, the entrainment of hydrophilic gangue exhibits detrimental effects, as it does in many other ores such as coal, Au/Cu sulphide ore (Gong et al., 2010), ultrafine sphalerite (Duarte and Grano, 2007), and base metal ores (Silvester et al., 2011). Many factors contribute to the entrainment of gangue, such as the properties of the gangue (Li et al., 2014), viscosity of the slurry, froth structure (Neethling and Cilliers, 2009), particle mass (Ata, 2012), and shape (Wang et al., 2015; Kirjavainen, 1996).

Thus far, there are two main methods to decrease the entrainment in froth flotation (Shi, 2001). The first method is ameliorating the flotation machine, such as optimizing the structure of the flotation cell or flotation column (Valderrama et al., 2011; Rubio, 1996; Li, 2013). For example, Mulleneers modified mechanical flotation cells by adding a counter current sedimentation zone to prevent the entrainment (Mulleneers et al., 2002). In addition, to encourage the drainage of gangues through the froth zone, technical solutions such as water spray to the froth layer (Kirjavainen, 1996), vibration of the froth zone, and a centrifugal-force-field flotation cell have been tested (Gong et al., 2010). The second method is optimizing the flotation technology and reagent system. Some researchers attempted to reduce the water recovery (consequently the entrainment) by increasing the flotation rate of hydrophobic particles (Akdemir et al., 2005). Weng et al. found that a three-stage grinding flotation circuit reduced the mechanical entrainment of gangue minerals in microcrystalline graphite flotation and the recovery and grade of final graphite product increased by 11.38% and 1.93% respectively compared with the onestage circuit (Weng et al., 2017). Furthermore, Qiu et al. (2016) originally introduced MF (formaldehyde condensate of methyl naphthalene sulfonic sodium salt) as a depressant and a surfactant for sericite and flaky graphite separation, and a high sericite recovery of 89.7% was obtained, indicating that sericite can be separated from graphite by reverse flotation separation.

In addition to the aforementioned methods, the entrainment of fine and ultrafine hydrophilic particles can be reduced by enlarging their particle sizes using either inorganic depressants or high-molecular-weight polymers (Gong et al., 2010; Cao and Liu, 2006), including aluminum-based, iron-based, inorganic-inorganic composite, inorganic-organic and multifunctional composite flocculants (Wang et al., 2004). Among them, Polyaluminum chloride (PACl) is the most commonly used inorganic polymer flocculant in the field of water treatment because of its high purification efficiency, excellent sedimentation performance and wide adaptability to pH (Wang et al., 2004). Theoretically, the introduction of PACl in flotation can increase the particle size and change the shape and surface charge of the sericite particles. Several recent studies have confirmed the possibility of introducing PACl as a flocculant in the flotation separation of carbonaceous minerals, such as coal and amorphous graphite (Liang et al., 2016; Li et al., 2015b). However, studies of PACl in fine flaky graphite flotation are scarcely found.

To investigate the effect of PACI on the dispersion and aggregation behaviours of sericite and fine flaky graphite, batch settling experiments of single minerals were performed in the first place. Consequently, batch flotation tests of artificial mixtures were performed to study the entrainment-reducing effect of PACI on sericite in the fine flaky graphite flotation. In addition, the surface charge of particles as a function of PACI were determined using zeta potential measurements. Fourier transform infrared spectroscopy (FTIR) and X-ray photoelectron spectroscopy (XPS) were also used to provided further information on PACI adsorption.

2. Materials and methods

2.1. Materials

In this study, the graphite and sericite single-mineral samples were obtained from Shangrao (Jiangxi, China) and Xianning (Hubei, China), respectively. The single mineral samples were processed using the method as follows. First, large lumps of minerals with high purity were picked out and crushed to -2.0 mm using a roll crusher, followed by wet grinding in a conical ball mill with zirconia balls as the grinding media. Finally, the ground samples were wet-screened using 0.037 mm US standard sieves and classified by sedimentation classification. The -38 +30 µm size fraction was collected and used in the tests. The fixed carbon (FC) and ash content of graphite single mineral were analyzed according to the standard method (GB/T 3521-2008) in China. The FC of the graphite single mineral was 95.75% with an ash content of 4.25%. Fig. 1a shows the X-ray diffraction pattern of the graphite single mineral, combining with the XRF analysis result, which indicates that the graphite content in the sample was higher than 95 wt %. The X-ray fluorescence (XRF) analysis of sericite indicates that it contained 9.17 wt % K₂O, 1.09 wt % Na₂O, 37.04 wt % Al₂O₃, and 48.51 wt % SiO₂. Fig. 1b shows the X-ray diffraction pattern of sericite, combining with the XRF analysis result, which indicates that the sericite sample contained more than 92 wt % sericite.

The flocculant PACI and other common chemical reagents in the experiment, such as sodium hydroxide, hydrochloric acid and ferron, were purchased from Sinopharm Chesericitel Reagent Co., Ltd. in Shanghai, China. Kerosene and octanol, which were used as the collector and frother, respectively, were purchased from Kermel Chemical Reagent Co., Ltd. in China. All of the reagents were of analytical grade and used directly without further purification.

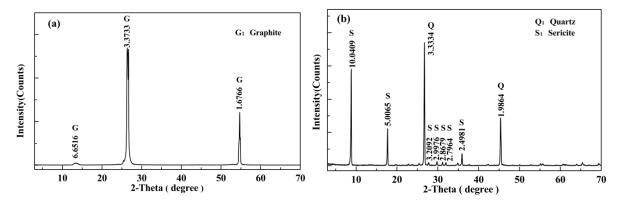


Fig. 1. X-ray diffraction (XRD) patterns of single minerals: (a) graphite single mineral; (b) sericite single mineral

2.2. Settling experiments

An amount of 2.5 g single mineral was added into 0.25 dm³ of PACl solution of a certain concentration. The pulp pH was adjusted to a desirable pH value using HCl or NaOH solutions before the pulp was stirred at 1800 r/min for 5 min. After settling for another 5 min, the sedimented particles were filtered, dried and weighed, the weigh is *Ms*. Finally, the sedimentation rate was obtained by equation (1).

$$S = \frac{M_S}{25} \times 100\% \tag{1}$$

where S is the sedimentation rate (%).

To determine the dispersion and aggregation behaviours of single minerals in the presence of PACl, batch settling experiments were performed. In addition, the dispersion and aggregation behaviours of sericite in the fine flaky graphite supernatant after the interaction with PACl were tested to simulate the effect of the PACl concentration on the actual graphite ore flotation. The supernatant was prepared by introducing fine flaky graphite into a solution of a certain PACl concentration. After 15 min of full interaction at a stirring speed of 2800 r/min, the supernatant was obtained through centrifugation.

2.3. Ferron method

A modified ferron method was applied to determine the speciation distribution of PACI (Wang et al., 2004). First, the mixed ferron solution was prepared by mixing 0.2% ferron, 20% NaAc solution and 10% HCl solution with a mass ratio of 2.5:2:1. Afterwards, 0.0055 dm³ of the mixed reagent was transferred into a graduated glass tube and diluted to a certain volume. Then, a certain amount of the PACI was added into the glass tube and quickly diluted to 0.025 dm³. The diluted PACI was transfered into a 1 cm glass container and mixed with diluted mixed ferron solution. A DU650 Beckman UV-Visible spectrophotometer were performed to measaure the timed absorbance at 366 nm for 2 h. It was operationally divided in to three stages: the first 1 min absorbance, Al_a; 1 min to 2 h, Al_b; Al_c was obtained by subtracting Al_a and Al_b from Al_t.

The aluminum components produced by the hydrolysis of PACl were classified into three categories on the basis of reacting kinetic difference with ferron : monomer Al_{a} , rapid reaction polymer Al_{b} and slow reaction colloid Al_{c} (Wang et al., 2004). The components of Al_{a} , Al_{b} and Al_{c} (Richens, 1997;Akitt et al., 1972; Wang and Tang, 2001) are shown in Table 1. It has been widely reconginized that Al_{b} (mainly including Al_{13} ($Al_{13}O_{4}$ (OH) $_{24}$ ⁷⁺) is considered as the active species of PACl for coagulation (Casey, 2006; Qu et al., 2004; Hu et al., 2012). In addition, Al_{c} was another high active species in solutions, the charge neutralization capacity of which is higher at pH under 6.5 and it obtaines the highest turbidity removal due to the strong flocs formation capacity (Chen et al., 2006).

Table 1. Components of Ala, Alb and Alc

Categories	Symbols	Ions
Monomer	Ala	Al ³⁺ , Al(OH) ²⁺ , Al(OH) ⁺ , Al(OH) ₃ , Al(OH) ₄ -
Rapid Reaction Polymer	Al _b	Al ₂ (OH) ₂ ⁴⁺ 、Al ₂ (OH) ₂ ⁵⁺ 、Al ₃ (OH) ₄ ⁵⁺ 、Al ₄ (OH) ₈ ⁴⁺ 、 Al ₆ (OH) ₁₂ ⁶⁺ 、Al ₁₃ O ₄ (OH) ₂₄ ⁷⁺ 、Al ₈ (OH) ₂₀ ⁴⁺ , etc.
Slow Reaction Colloid Al _c		Al(OH) _{3(s)}

2.4. Batch flotation tests

An XFD flotation cell (volume=0.14 dm³) was used in batch flotation tests. Sericite is one of the main gangue minerals in graphite ores (Li et al., 2015a). To investigate the effect of PACI on the entrainment of sericite in the flotation process, batch flotation tests of artificial mixtures composed of sericite and fine flaky graphite particles (the mass ratio of graphite to sericite was 4:1) were performed. The desired pH value was adjusted by HCl and NaOH solutions. First of all, the slurry was prepared by adding 2.5 g of the artificial mixture sample to 0.100 dm³ of solution. Secondly, the flocculant PACI was added and conditioned for 5 min at an agitation speed of 1000 rpm to enable the PACI adsorption. Followed the collector, kerosene and frother octanol were added to the pulp and conditioned for 3 min at an agitation speed of 1400 rpm. Then, flotation was performed for 3 min. Finally, the obtained froth products and unfloated products were filtered, dried, and weighed to calculate the flotation recovery. The sericite recovery and water recovery can be calculated by Equations (2) and (3).

$$R_{s} = \left[1 - \frac{2(m_{2} - m_{1}A_{g})}{A_{s} - A_{g}}\right] \times 100\%$$
⁽²⁾

where R_s is the recovery of sericite in the froth product (%); m_1 and m_2 are the weight and ash weight of the unfloated product, respectively (g); A_s and A_g are the ash content of sericite and graphite single mineral, respectively (%) (Li et al., 2015). A_s is the ash content of sericite sample that was analyzed according to the standard method (GB/T 3521-2008) in China.

$$R_w = \frac{M_1 - M_2}{M_w} \tag{3}$$

where R_w is the water recovery in the froth product (%); M_1 and M_2 are the wet weight and dry weight of the froth product, respectively (%); M_w is the weight of water consumed in the flotation process (%) (Liang et al., 2016).

3. Results and discussion

3.1. Speciation transformation of PACI

Fig. 2 shows the speciation characterization of PACl at various pH conditions using the modified ferron method. As observed, the pH value has a significant effect on further speciation transformation of PACl. In the acidic range, Al_a sharply decreases when the pH is increased and reaches a minimum in the nearly neutral pH region. The majority of Al_b occurs in the nearly neutral pH region, which suggests that the Al_a species largely transforms to Al_b . However, in the alkaline portion, a rapid decrease of Al_b and a sharp increase of Al_a occur when pH is increased. The distribution of Al_c is similar to that of Al_b .

3.2. Dispersion and aggregation behaviours of single minerals

The effects of pH and PACl on the dispersion and aggregation behaviours of sericite and fine flaky graphite single minerals were studied in the settling experiments. Accordingly, the chemical conditions for selective aggregating sericite were explored.

3.2.1. Dispersion and aggregation of single minerals in the aqueous solution

The variations of zeta potentials and sedimentation rate of sericite and fine flaky graphite based on various pHs in aqueous solutions are shown in Fig. 3

Fig. 3a shows that the points of zero charge for graphite and sericite are approximately 5.0 and 1.0, respectively, which are lower than the theoretical pH values of 7.0 and 2.0. This variation of iso-electric

point can be as a result of the crystal defect and the presence of impurities into the lattice of fine flaky graphite and sericite, which lead to a relatively constant negative charge on the particle surface (Qiao et al., 2011; Bergström, 1997). When the pH value is increased, the negative zeta potential values at the surface of these minerals contentiously increased.

In Fig. 3b, the sedimentation rate of sericite dramatically decreases with the increase in pH, whereas the sedimentation rate of graphite continuously remains high in the identical pH range. The dispersing/aggregating behaviours of sericite in aqueous solutions are consistent with the typical DLVO theory ($V_T=V_{ER}+V_{WA}$); where V_T is the total interaction energy among the particles; V_{ER} is the electrostatic repulsive energy; and V_{WA} is the Van der Waals attraction energy (Hunter, 2001).

As the pH increases, an increasing absolute value of the sericite particle surface potential is observed and the dispersing/aggregating behaviours are governed by the electrostatic repulsive energy, leading to better dispersion of sericite in aqueous solutions. However, the sedimentation rate suddenly increases when the pH is increased over 11 because the increase in ion concentration in the solution compresses the thickness of the electric double layer and deteriorates the dispersion between the particles.

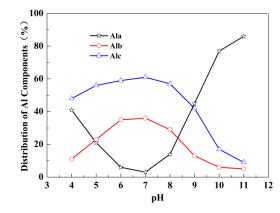


Fig. 2. Speciation characterization of PACl as a function of pH

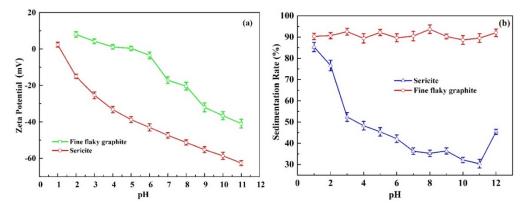


Fig. 3. Zeta potential (a) and sedimentation rate (b) of single minerals as a function of pH

Behaviours of fine flaky graphite, however, are consistent with the extended DLVO theory $(V_T=V_{ER}+V_{WA}+V_{HA})$, where V_{HA} is the hydrophobic attraction energy among the graphite particles and it is 1-2 magnitudes more intense than the electrostatic repulsion or van der Waals force (Song, 1993; Qiu et al., 2017). Therefore, the total interaction energy was negative over most of the distance range, and thus hydrophobic interactions dominate the aggregation of the hydrophobic graphite particles, leading to the high sendimentation rate (Qiu et al., 2017).

3.2.2. Dispersion and aggregation of single minerals in the presence of PACI

Fig. 4a shows the effect of pH on the aggregation behaviour of sericite at the PACl concentration of 300 mg/dm^3 . The sedimentation rate of sericite is the highest near the neutral pH condition. According to

the speciation characterization of PACl in Fig. 2, either Al_b or Al_c is the active ingredient to aggregate sericite particles. Moreover, the sedimentation rate of sericite in the aqueous solution is continuously higher than that in the fine flaky graphite supernatant, which indicates that the graphite surface also absorbs some PACl and reduces the actual concentration.

Fig. 4b illustrates the effect of the PACl concentration on the dispersion and aggregation behaviours of fine flaky graphite and sericite at the neutral pH condition. The sedimentation rate of fine flaky graphite in the aqueous solution is continuously maintained at approximately 95%, suggesting that the dispersion and aggregation of graphite were still dominated by hydrophobic interaction. However, the sedimentation rate of sericite exhibited a strong correlation with the PACl concentration both in aqueous solution and in fine flaky graphite supernatant. An initial increase of sedimentation rate was observed and then a decline tendency appeared as the PACl concentration was continued to increase. However, to achieve the greatest sedimentation rate in fine flaky graphite supernatant, a higher PACl concentration of 400 mg/dm³ was required. This distinction, once again, confirmed some PACl was adsorbed on graphite surface.

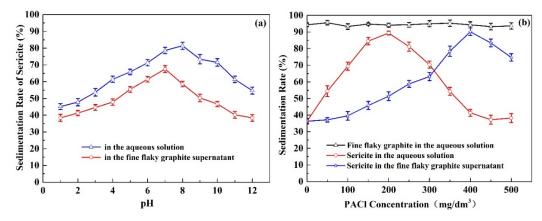


Fig. 4. Effects of the pH (a) and PACl concentration (b) on the sedimentation rate of single minerals

3.3. Batch flotation of artificial mixtures

The above results indicate that PACI can effectively aggregate sericite particles and improve the sedimentation rate. Theoretically, it can reduce the entrainment of sericite in flotation. In this section, the effects of pH and PACI concentration on the entrainment of sericite were investigated by batch flotation tests of artificial mixtures. The addition of PACI had little effect on the recovery of graphite, and the recovery of graphite was always greater than 95%.

3.3.1. Effect of pH on the entrainment of sericite

To determine the effect of pH on the entrainment of sericite, batch flotation tests were performed at the PACl concentration of 400 mg/dm^3 , and the results are shown in Fig. 5.

As seen, there is a direct correlation between the recovery of sericite and the water recovery, which is consistent with some studies on the correlation between the recovery by entrainment and the water recovery (Engelbrecht and Woodburn, 1975; Laplante et al., 1989). The lowest recovery of sericite and the water recovery at only 45.23% and 53%, respectively, were obtained at the pulp pH of 7. As expected, the highest sedimentation rate of sericite was observed at this pH condition. Consequently, the entrainment of sericite was significantly diminished because of the greatest aggregation effect of PACI on sericite particles under the neutral pH.

3.3.2. Effect of PACl concentration on the entrainment of sericite

Fig. 4 shows that a suitable dosage of PACl can aggregate sericite and improve its sedimentation rate, whereas excessive PACl contributes to the redispersion of sericite. To determine the preferable PACl concentration for actual graphite ore flotation, batch flotation tests were performed under the neutral pH condition, and the results are shown in Fig. 6.

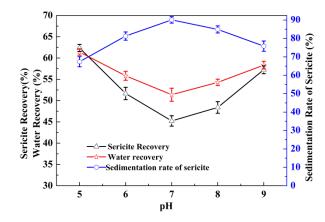


Fig. 5. Effect of pH on entrainment behaviour of sericite (PACl = 400 mg/dm^3)

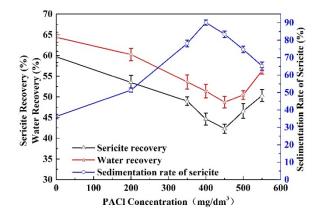


Fig. 6. Effect of the PACl concentration on the entrainment behaviour of sericite (pH = 7)

When the PACl concentration is increased, the sericite and water recoveries continuously decrease. Note that when the PACl concentration is higher than 450 mg/dm³, the sericite and water recoveries conversely increase because the excess PACl increases the electrolyte concentration in the solution and consequently enhances the froth stability (Craig et al., 1993). As a result, the optimum concentration of PACl in the actual fine flaky graphite flotation should be approximately 450 mg/dm³.

3.4. Mechanisms of PACl on the dispersion and aggregation behaviours of sericite and graphite

In general, it was reported that there are two main mechanisms for PACl aggregating fine/ultrafine particles and insoluble organic materials: charge neutralization (Jiang, 2015) and precipitate enmeshment. In charge neutralization, the aluminum species that PACl hydrolyzes are positively charged at neutral pH, whereas the dispersed mineral particles are often negatively charged and repulse one another. In precipitate enmeshment, amorphous hydroxide precipitates may form on the particle surfaces. The subsequent accumulation of these precipitates leads to the aggregation and settlement of the dispersed particles (Duan and Gregory, 2003; Xiao et al., 2008; Harif et al., 2012).

3.4.1. Zeta potential measurement

The zeta potential variations and sedimentation efficiency of sericite versus the PACl concentration in aqueous solutions and flaky graphite supernatant are shown in Fig. 7.

When the PACl concentration increases, the surface of sericite shifts from negatively charged to positively charged, which indicates the increasing adsorption of positively charged Al_b and Al_c onto the surface as a result of electrostatic attraction.

The sedimentation rate of sericite is closely related to the absolute value of the zeta potential on the sericite surface. A higher absolute value of the zeta potential corresponds to a greater sedimentation rate of sericite. Therefore, the sericite exhibits the greatest sedimentation rate at the PACl concentration

of approximately 200 mg/dm³ and 400 mg/dm³ in the aqueous solution and fine flaky graphite supernatant, respectively, where the absolute value of the zeta potential on the sericite surface is close to 0, as illustrated in Fig. 7. This strong consistency between the variations of sedimentation rate and surface zeta potential demonstrates that charge neutralization is one of the mechanisms of sericite dispersion and aggregation in the PACl solution.

The precipitate enmeshment of sericite from the amorphous precipitated hydroxide Al_c is another contributing factor. However, during the mixed mineral flotation process, an agitation speed up to 2800 r/min is required to maintain the dispersion of the flotation slurry. Strong agitation must damage the floc units and undermine the flocculation effect (Yukselen and Gregory, 2004).

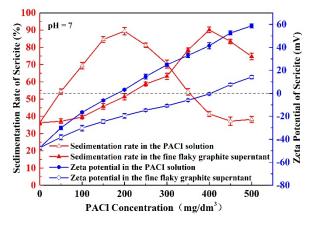


Fig. 7. Effect of the PACl concentration on the sedimentation efficiency and zeta potential of sericite (pH = 7)

3.4.2. Optical microscope images

Fig. 8 shows the optical microscopy images of fine flaky graphite particles and sericite particles in aqueous solutions at pH 7.0 in the absence and presence of PACl, respectively. Figs. 8a and 8c show that sericite particles are desultorily dispersive, and no aggregation is formed in the aqueous solution. The size of most particles in the image is under 40 μ m, which is consistent with the particle size distribution results as prepared. In Figs. 8b and 8d, the small sericite particles aggregate into a large and compact particle. The results indicate that PACl effectively aggregates sericite particles and enlarges the apparent size.

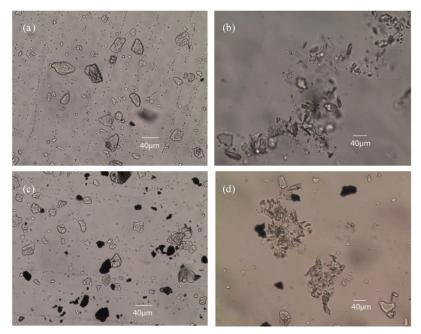


Fig. 8. Optical microscopy images of graphite and sericite in aqueous solutions. (a) Sericite without PACl; (b) sericite with PACl; (c) sericite and graphite without PACl; (d) sericite and graphite with PACl

3.4.3. FTIR spectra analysis and XPS analysis

To further verify the mechanisms of PACl in the flotation separation of sericite from graphite, the IR (infrared) spectra of these two minerals before and after interacting with reagents were measured, and the results are shown in Fig. 9.

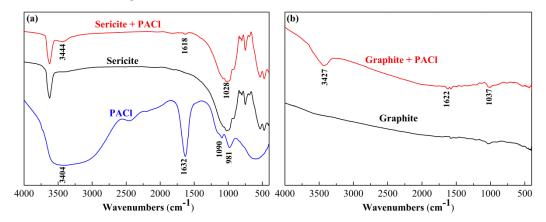


Fig. 9. FTIR spectra of (a) sericite and (b) graphite before and after the interaction with PACl

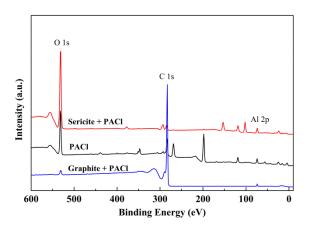


Fig. 10. X-ray photoelectron spectroscopy (XPS) survey spectra of graphite and sericite after the interaction with PACI

Sample —	Electron Binding Energy, E _B /eV					
	C 1s	O 1s	Al 2p	Si 2p	К 2р	
PACl	283.68	531.76	74.15	101.42	292.46	
Sericite+PACl	284.35	531.57	73.77	102.06	292.71	
$\Delta E_{\rm B}/{\rm eV}$	0.67	-0.19	-0.38	0.63	0.25	
Graphite+PACl	283.11	531.28	73.61	101.85	-	
$\Delta E_{\rm B} / {\rm eV}$	-0.57	-0.48	-0.54	0.43	-	

Table 2. XPS analysis results of graphite and sericite after the interaction with PACI

The FTIR spectrum of PACl presents an extremely wide stretching band of Al-OH-Al groups from approximately 3000 cm⁻¹ to 3500 cm⁻¹ and the intense vibration bends of hydrate water (H-O-H) at 1632 cm⁻¹, which indicates the vast existence of -OH groups in PACl molecules. The vibration bends of Al-OH-Al groups appear at 1090 and 981 cm⁻¹. After the interaction with PACl, the stretching bands of Al-OH-Al groups in the PACl molecules appear at approximately 3444 and 3427 cm⁻¹ on sericite and graphite surfaces, respectively. Moreover, the vibration bands of the hydrate water in PACl molecules appear at 1618 cm⁻¹ on sericite surfaces and 1622 cm⁻¹ on graphite surfaces. The FTIR spectrum results demonstrate that after the PACl treatment, no new adsorption peaks appear on the sericite and graphite

surfaces, except for the adsorption bands of PACl, which implies that PACl may adsorb onto the two minerals without the formation of new complexes.

As an alternative to FTIR, the XPS analysis of samples was performed. The XPS survey spectra of graphite and sericite after the interaction with PACl are shown in Fig. 10. Table 2 shows that Al and O elements appear on the surface of graphite and sericite. Meanwhile, the changes in electron binding energy (ΔE_B) of C, O and Al elements after the treatment with PACl are relatively small and less than the error value of the experiment equipment. Hence, the interaction between PACl and graphite or sericite is physical adsorption without the formation of new complexes. The XPS spectra are consistent with the FTIR results and confirm that physical adsorption occurred on the graphite and sericite surface.

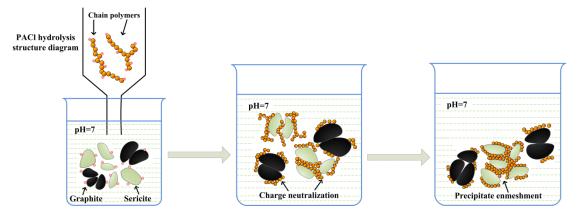


Fig. 11. Schematic representation for the selective aggregation of PACI on sericite and graphite minerals

The aforementioned mechanisms are schematically represented in Fig. 11. At the pH of 7, sericite particles are dispersed and negatively charged in the aqueous solution. PACl is hydrolyzed into fine particles and mutually aggregated into positively charged large chain polymers Al_b. As a function of the charge neutralization, the large chain polymers are favorably absorbed onto the negatively charged sericite surface. Aggregation of the dispersed sericite particles can be observed as a result of electrostatic attraction among sericite particles and the bridging of the precipitates. In the case of graphite particles, intense hydrophobic interaction dominates, and large and compact agglomerations can be observed throughout the process.

4. Conclusions

Batch settling experiments and zeta potential measurements of pure sericite and graphite indicate that the dispersion and aggregation behaviours of sericite are mainly governed by the electrostatic force among the particles. A lower absolute value of the zeta potential on the sericite surface corresponds to a better aggregation effect. However, the dispersion and aggregation of fine flaky graphite is dominated by the hydrophobic force. Moreover, PACI can effectively aggregate sericite by changing the zeta potential on the sericite surface. The results of batch flotation tests demonstrated that the addition of PACI can effectively decrease the recovery of sericite and water in fine flaky graphite flotation by increasing the apparent particle size of sericite and consequently reducing its entrainment, which was verified in the optical microscope images. The entrainment of sericite in the fine flaky graphite supernatant was significantly diminished at the neutral pH and PACI concentration of 400 mg/dm³.

The zeta potential measurements, FTIR and XPS analyses indicated that the charge neutralization and precipitate enmeshment are the main mechanisms for PACl aggregating sericite. The hydrolyzed aluminum species Al_b and Al_c were adsorbed onto sericite particles through electrostatic attraction and surface precipitation.

Acknowledgments

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