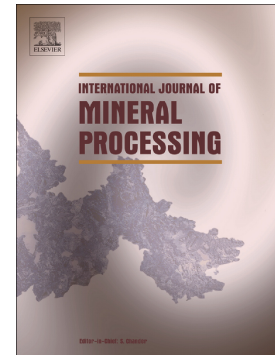


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Flotation separation of scheelite from calcite using mixed collectors

Weiping Yan^a, Cheng Liu^{b1*}, Qiming Feng^b, Wencai Zhang^c

^a School of Mineral Processing and Bioengineering, Central South University, Changsha 410083, China

^b Institute of Multipurpose Utilization of Mineral Resources, Chinese Academy of Geological Sciences, Chengdu, 610041 Sichuan, China

^c Department of Mining Engineering, University of Kentucky, 504 Rose Street, 230 Mining & Mineral Resources Building, Lexington, KY 40506-0107, United States

Abstract: Flotation separation of scheelite from calcite is difficult due to the similarities in their surface properties. In this work, the flotation behavior of scheelite and calcite using oxidized paraffin soap(OPS), benzohydroxamic acid(BHA) and the mixed OPS/BHA collectors was investigated through micro-flotation experiments. The flotation results of single mineral experiments demonstrated a higher selectivity for the flotation of scheelite from calcite at pH 9 than individual OPS and BHA when using water glass as depressant. In order to probe the validity of the findings, mixed binary minerals experiments, contact angle and zeta potential experiments were also carried out successfully.

Keywords: Scheelite, Calcite, mixed collectors, Flotation separation

1 Introduction

Tungsten is an important non-ferrous metals that used in the electron, alloys, chemical engineering, as well as other industries (Bar et al., 2000; Ilhan et al., 2013; Rao, 1996). The global tungsten resources with industrial utilization value are mainly derived from wolframite and scheelite. China is a country with abundant wolframite and scheelite resource with huge reserves on top of the world, and scheelite usually coexists with other Ca-bearing minerals in ore deposits, such as fluorite (CaF_2), calcite (CaCO_3), fluorapatite [$\text{Ca}_{10}(\text{PO}_4)_6\text{F}_2$](Deng et al., 2016), however, the most common calcium containing gangue in scheelite ores is calcite(Filippova et al., 2014; Filippova et al., 2012; Somasundaran et al., 1985). In practice, the commonly used method for recovering scheelite minerals from calcite is flotation. However, it is difficult to separate scheelite from calcite minerals due to the same Ca site on their surfaces, resulting similar flotation performance when using fatty acid as collectors(Miller and Misra, 1984; Marinakis et al., 1985; Feng et al., 2015; Antti and Forssberg, 1989; Rao et al., 1991; Miller et al., 1995; Kou et al., 2010), hence the separation is not always satisfactory in industry (Houot, 1982; Mishra, 1982; Pugh and Stenius, 1985; Zheng and Smith, 1997). To achieve excellent separation performance, many investigations indicated that using collector mixtures can have a synergistic advantage over the use of single collector on the flotation of scheelite when using water glass for depressing calcite. Gao et al (2015) discovered that the mixed anionic/anionic(733/MES) collectors exhibited a higher selectivity for the flotation separation of scheelite from calcite and fluorite than the 733-collector alone.. Huang et al (2016) found that there is a high selectivity and recovery for the flotation of scheelite from calcite at pH 7 using

Corresponding author.

E-mail address: liucheng309@sina.com (C.Liu)

mixed anionic/cationic(DDA/NaOL) collectors. However, very little research has been conducted concerning the separation of scheelite from calcite in the presence mixed anionic/chelate collectors.

Collector oxidized paraffin soap(OPS), a anionic collector, is widely used industrially for scheelite flotation (Huang et al., 2010; Li et al., 2010; Meng et al., 2007), always has a high reactive but low selective performance. Previous research has shown that using water glass as depressant and oxidized paraffin soap as collector can achieve selective flotation of scheelite from calcite, however, the water glass need to be acidized (Liu et al.,2016). Hydroxamate collectors have been widely utilized in flotation due to its distinct chelating abilities with different metal ions (Zhang and Honaker, 2017, Zhang et al., 2017). Benzylhydroxamic acid(BHA), a chelate collector, has a relatively poor collecting performance for scheelite. Feng used benzohydroxamic acid as collector and saline(lead ions) water glass as a depressant for the separation of scheelite from calcite and shown that the reagent scheme exhibited a excellent selecting power(Feng et al., 2016), but the use of lead ions brings about water pollution, soil pollution, etc. Up to now, mixed OPS/BHA collectors have not been reported for the separation of scheelite from calcite.

Hence the aim of this study is to investigate the separation of scheelite from calcite. This work using mixed OPS/BHA as collector and water glass as a depressant. Single and mixed minerals micro-flotation tests were conducted to examine the selective separation behaviour, and the zeta potential measurement and contact angle analyses were performed to investigate the mechanisms of the selective separation.

2 Materials and methods

2.1. Pure minerals and reagents

The pure minerals of scheelite and calcite were obtained from Luanchuan of Henan Province and Ganzhou of Jiangxi Province, China, respectively. The samples were crushed to -1mm and ground using an agate mortar. The products were then dry sieved and the $-74\ \mu\text{m}$ size materials were collected and used for micro-flotation tests. The X-ray diffraction analyses chemical compositions of sample were shown in Fig 1 and Table 1, respectively. X-ray diffraction analysis demonstrated that the purities of samples were very high.

Technical grade oxidized paraffin soap($\text{C}_{12-16}\text{COONa}$) and water glass were obtained from the Zhuzhou Flotation Reagents Factory(Hunan, China), and water glass with the ratio of $\text{SiO}_2:\text{Na}_2\text{O}$ is 2.4. AR grade benzohydroxamic acid was obtained from Shanghai EKEAR Bio@Tech Co.LTD. HCl (hydrochloric acid) and NaOH (Sodium hydroxide) were used as pH regulators. Distilled water was used for all tests.

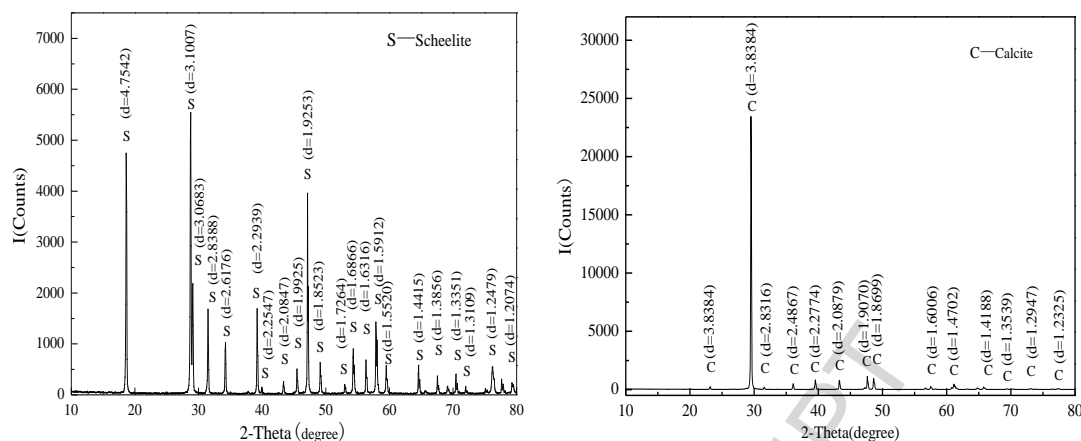


Fig. 1. XRD diagrams of single scheelite and calcite samples.

Table 1. Chemical analysis results of single scheelite and calcite samples

Sample	WO ₃	CaO	MgO	Fe	SiO ₂
scheelite	79.72	19.89	/	0.01	/
calcite	/	55.36	0.05	/	0.27

2.2 Micro-flotation tests

The micro-flotation tests were carried out in an XFGC-1600 flotation machine (see Fig.2) with a 50 mL plexiglass cell, and the impeller speed of fixed at 1800 rpm. Pure mineral particles (2 g) and deionized water (40 ml) were both placed in the plexiglass cell. The pH of the was adjusted by adding NaOH or HCl. After the desired amount of reagents was added, the pulp was stirred for 5 min. Recording the pH of the suspension before flotation, and the flotation was conducted for 4 min. For single mineral tests, the forth and tails were collected, dried, and weighed. and the recovery was calculated based on the dry weight of the product. For mineral mixture, the concentrates and tails were assayed for WO₃ and used to calculate the recovery. Each micro-flotation test were measured three times, the average reported as the final value.

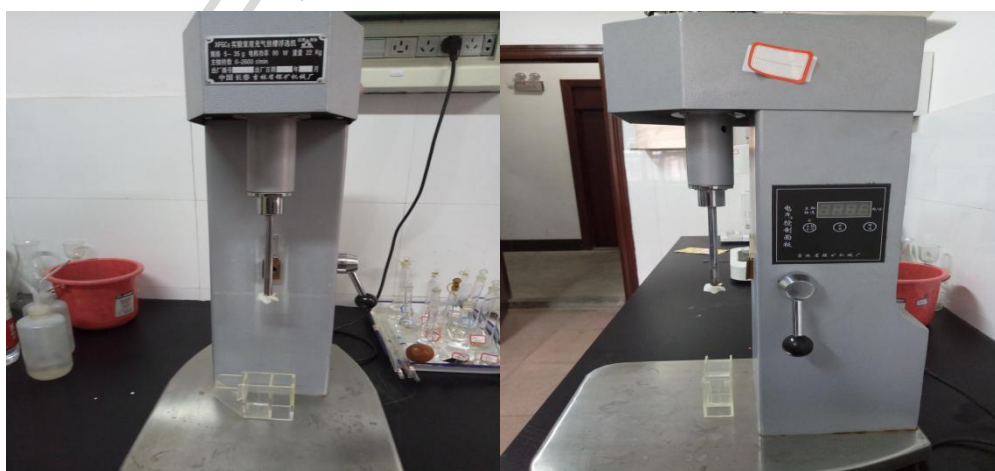


Fig. 2. XFGC flotation machine for pure mineral flotation tests.

2.3 Contact angle measurements

Contact angle measurements were carried out using a MiniLab ILMS analyzer

instrument in a desired reagent solution. A hand-picked blocky pure single sample was cut by a portable abrasive cutter, and the exposed cut surface were polished by alumina powder. After the polished sample was washed by distilled water, conditioned in the desired reagent solution at a given pH for 10 min, and subjected to the contact angle measurement. The average of five readings was reported for each measurement. The last process was repeated for repolishing before each measurement.

2.4 Zeta potential tests

Zeta potential measurements were performed using a Coulter Delsa440sx Zeta analyzer instrument. The suspensions (0.01% mass fraction) with small amount of minerals in a $1 \times 10^{-3} \text{M}$ KCl background electrolyte solution were dispersed in a beaker magnetically stirred for 6 min at a desired pH. After 20 min of settling, the pH value of the suspension was measured and the supernatant was collected for zeta-potential measurements. The zeta-potential of each sample was measured three times, and the average value was reported and the standard deviation were calculated.

3 Results and discussions

3.1 Flotation experiment results

Fig. 3 presents the flotation recoveries of scheelite and calcite as a function of pH in the presence of 50 mg/L oxidized paraffin soap and 200 mg/L BHA, respectively. Both scheelite and calcite showed good flotation in the pH region from 7 to 12 when using OPS as collector. The high recovery of both minerals in the basic region is attributed to interaction between RCOO^- and Ca ions on mineral surface (Gao, 2014). Regarding BHA alone, its collecting power is significantly weaker than OPS. the flotation recovery of scheelite increased with the increasing pH and reached maximum values at approximately pH 9, and about 60% was obtained, and the calcite recovery (about 20%) does not change across the entire pH range. The interaction mechanism of minerals with BHA is due to $-\text{CONHOH}$ group exhibits a chelating ability with metal ions exposed on the mineral surfaces (Deng et al., 2015). Fig.3 demonstrated that it is impossible to separate scheelite from calcite without depressants using OPS as collector. Although the flotation recovery of scheelite is about 40% higher than calcite is about 40% using BHA as collector, the collecting performance is poor for scheelite.

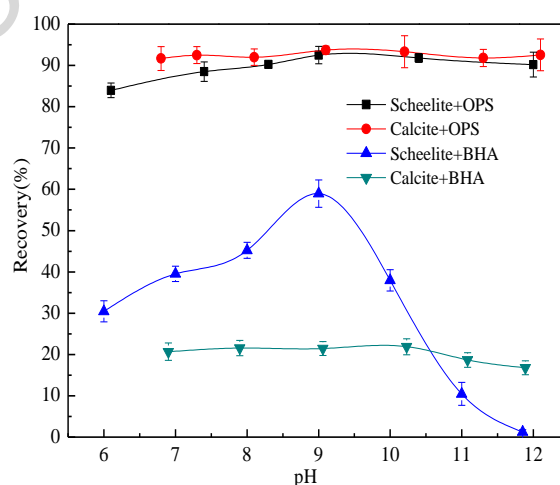


Fig.3 Effect of pH on the flotation of scheelite and calcite with 50 mg/L OPS and 300 mg/L BHA, respectively

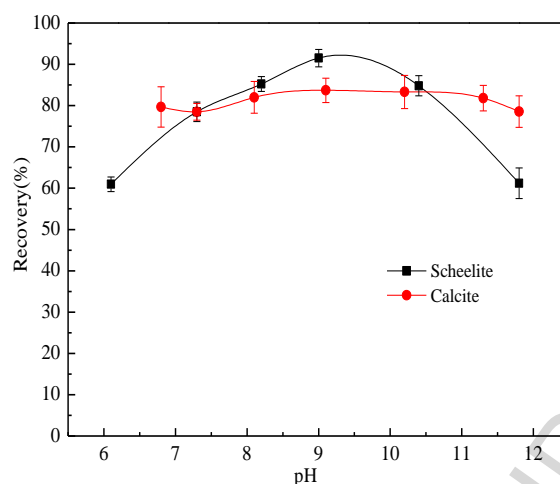


Fig.4 Effect of pH on the flotation of scheelite and calcite with mixed OPS/BHA collectors (OPS:25mg BHA:150mg/L)

The effect of mixed OPS/BHA collectors on the flotation recovery of scheelite and calcite as a function of pH are plotted in Fig.4. As shown in Fig.4, the recovery of both scheelite and calcite presented good flotability. The maximum flotation recovery of scheelite occurred at approximately pH 9, and the calcite recovery is slightly lower than scheelite. The results in Fig. 4 also demonstrate that it is difficult to separate scheelite from calcite in the absence of depressant.

Fig.5 shows the flotation recoveries of scheelite and calcite as a function of water glass concentration in the presence of OPS at pH 9. As can be seen in Fig. 5, flotation recovery of scheelite calcite decreased slowly with the increases in water glass initial concentration. The recovery of calcite was more than 60%, although the water glass concentration was 400 mg/L, indicating that the flotation separation of scheelite from calcite can not be realized. Fig 5 also shows the effect of water glass concentration on the flotation of scheelite and calcite with 300 mg/L BHA, the recovery of calcite is below 10% when the water glass concentration is 150mg/L, however, the recovery of scheelite is below 50%, it is not conducive to the scheelite resource recycling.

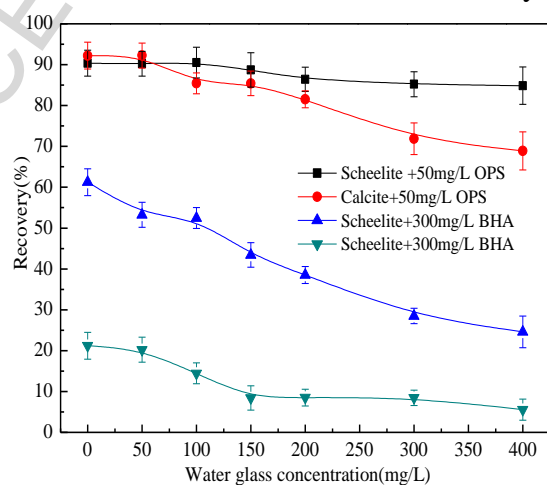


Fig. 5 Effect of water glass concentration on the flotation of scheelite and calcite with OPS and BHA, respectively

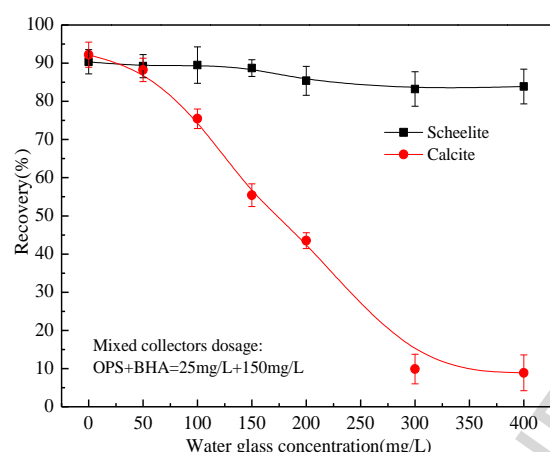


Fig. 6 Effect of water glass concentration on the flotation of scheelite and calcite with mixed OPS/BHA collectors

In order to study the selective flotation performance of scheelite and calcite with the mixed OPS/BHA collectors, also using water glass as depressant, the effect of water glass concentration on the flotation performances of scheelite and calcite are plotted in Fig.6. The calcite recovery was less than 10% when the concentration of water glass is 300mg/L. Interestingly, by increasing water glass concentration the recovery of scheelite decreased slightly, which suggested that mixed OPS/BHA is a suitable collector on selective flotation of scheelite and calcite using water glass as a depressant.

The single mineral flotation results indicate that that it may be possible to depress calcite on scheelite flotation with different collectors when using water glass as depressant. Mixed scheelite and calcite flotation tests have been conducted were conducted with mixed OPS/BHA collectors at pH 9 using 300mg/L water glass and the result is shown in Table 2. It can be seen from Table 2 that the recovery of scheelite is 76.28% and the grade of WO_3 is 61.41%. For comparison, the mixed ore flotation results of using single OPS as collector is also presented in Table 2. As shown in Table 2, the WO_3 grade is only 52.49% and is significantly lower than that using mixed OPS/BHA when the scheelite recovery is similar. The results suggest that an effective separation of scheelite from calcite can be achieved with the reagent scheme of mixed OPS/BHA as collector and water glass as depressant.

Table.2 Flotation results of mixed scheelite/calcite

Collector	Products	Yield (w/%)	WO_3 grade (%)	WO_3 recovery(%)
Mixed OPS/BHA	Concentrate	32.96	61.41	76.32
	Tailings	67.04	9.37	23.68
	Feed	100.00	26.52	100.00
Single OPS	Concentrate	39.23	52.49	77.64
	Tailings	60.77	9.76	22.36
	Feed	100.00	26.52	100.00

3.2 Contact angle results

The hydrophobicity of scheelite and limonite with mixed OPS/BHA collectors in the absence and presence of water glass was characterized by contact angle and the results are presented in Table 3. It can be seen from Table 3 that the contact angle of scheelite

increases from 23.4° to 76.8° and calcite from 37.2° to 72.4° in addition of mixed collectors at pH 9, respectively, indicating the increase of hydrophobicity for both minerals under mixed collectors. For comparison purposes, the table also shows the contact angle of water glass-treated scheelite and calcite, further conditioned with mixed collectors at pH 9, in this latter case the contact angle of scheelite is 73.5°. however, the the contact angle of calcite decreased to 30.4°, which indicates that calcite has a relatively lower hydrophobicity than scheelite after the same sequential treatment by water glass and mixed collectors. The contact angle results illustrated that the mixed collectors OPS/BHA can improve the surface hydrophobicity of scheelite, but no calcite in the presence of water glass, which agreed with the single mineral and mixed mineral flotation results(see Fig.6 and Table.2).

Table 3 Contact angle results of scheelite and calcite

Reagent	Contact angle(degrees)	
	Scheelite	Calcite
Distilled water	23.4	37.2
OPS+BHA:25mg/L+150mg/L	76.8	72.4
Water glass:300mg/L OPS+BHA:25mg/L+150mg/L	73.5	30.4

3. 4 Zeta potential results

Fig.6 and Fig.7 presents the zeta potential of scheelite and calcite with and without reagents as a function of pH, respectively. The IEP(isoelectric point) of calcite occurred at pH 9.5, and scheelite zeta potential are negative in the pH range of 6-11, which agrees with the previous investigations of Gao and Yang, etc (Gao et al., 2015; Yang et al., 2015;Hu and Xu, 2003). It can be seen from Fig.6 that the addition of OPS alone causes a decrease by 14 mV in zeta potential of scheelite around pH 9, indicating a strong interaction of OPS species at Ca site of scheelite surface(Liu et al, 2016). In the presence of mixed OPS/BHA, the zeta potential of scheelite is more negative (decreased from -23 mV to -42 mV) compared to using OPS alone, suggesting a much stronger interaction between mixed collectors and scheelite surface. Fig.6 also shows that water glass reduced the scheelite zeta potential slightly(decreased from -23mV to -29mV), suggesting a weak interaction of water glass species on negatively charged scheelite, after the addition of mixed OPS/BHA tended to produce more negative surface charge of scheelite than with water glass alone, illustrating that conditioning of scheelite with water glass prior to mixed collectors addition can not prevent the adsorption of mixed collectors on scheelite surface.

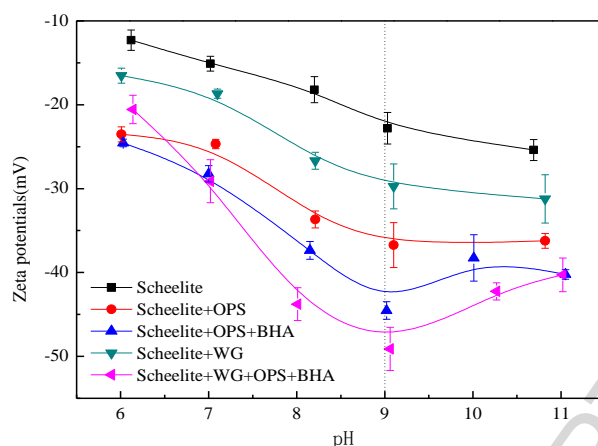


Fig.6 Effect of reagent adding on the zeta potential of scheelite as a function of pH
(WG(water glass)=300mg/L, OPS=25mg/L, BHA=150mg/L)

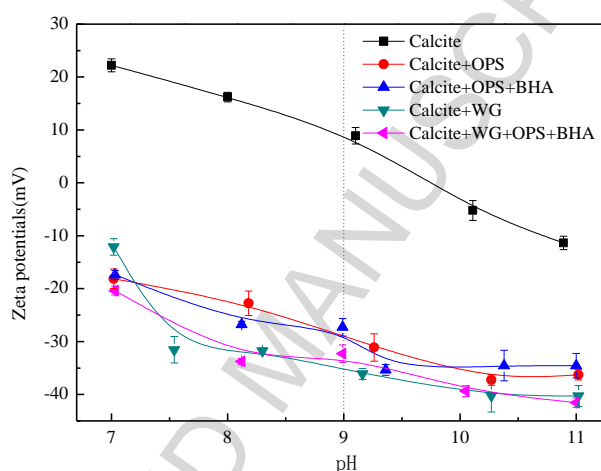


Fig.7 Effect of reagent adding on the zeta potential of scheelite as a function of pH
(WG(water glass)=300mg/L, OPS=25mg/L, BHA=150mg/L)

Fig.7 shows that the zeta potential of calcite decreased significantly in the presence of OPS, indicating a similar adsorption behavior of OPS species on calcite surface, after the addition of mixed OPS/BHA, the surface charge of calcite changed slightly compare to the result of the addition OPS alone around pH 9, suggesting a weak interaction of BHA species on the calcite surface. Compare calcite with scheelite, this weak adsorption may be attributed to the formation of O-Ca bonds between BHA and surface Ca sites. For the O-O distance in WO_4^{2-} group of scheelite (2.899\AA) matched well with that in BHA group (2.842\AA), but the O-O distance in CO_3^{2-} group of calcite was 2.224\AA , which was far shorter than that in BHA group (2.842\AA) (Deng et al., 2016), causing to a much stronger interaction of BHA with scheelite in the presence of OPS. By adding water glass alone. The addition of water tended to produce more negative surface charge for calcite (see Fig.6) than scheelite (see Fig.7). This could be attributed to the production of $\text{SiO}(\text{OH})_3^-$ is the predominant species of water glass in the pulp at pH 9 that adsorbed on positively charged calcite surfaces by electrostatic attraction (Mishra, 1982, Feng et al., 2015). The addition of mixed OPS/BHA after water glass, calcite surface charge changed slightly, indicating that the presence of water prevents the mixed collectors adsorption on calcite surfaces.

4. Conclusion

Through the micro-flotation tests of pure scheelite and calcite, mixed OPS/BHA collectors present higher selectivity and stronger collecting power to scheelite than individual collector. The mixed collectors could achieve flotation separation of scheelite from calcite, and the water glass need not to be acidized. Zeta potential implied that water glass adsorbed more strongly on calcite surface by electrostatic attraction. Mixed collectors OPS/BHA adsorbed more strongly on scheelite surface than on calcite in the presence of water glass, illustrating that the addition of water glass before mixed OPS/BHA does not prevent mixed collector to absorb on scheelite surface while it prevents mixed collectors to adsorb on calcite surface. The results of contact angle measurements are matched well with the micro-flotation results. Under these circumstances, it can be indicated that mixed OPS/BHA is an appropriate collector in selective separation of scheelite from calcite when using water glass as depressant

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Highlights

Effect of mixed OPS/BHA on selective flotation of scheelite from calcite was attempted.

Single mineral and mixed minerals flotation tests were conducted at pH 9.

Scheelite/calcite separation was achieved using mixed OPS/BHA collectors.

Zeta potential and contact angle experiments confirmed the flotation results in this study.