

# Effect of Fe(II) as assistant depressant on flotation separation of scheelite from calcite

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## ABSTRACT

The preparation of high-grade scheelite concentrates from calcite containing ores despite significant research progress remains an important commercial challenge. In this study, Fe(II) was selected as an assistant depressant and combined with sodium silicate (SS) to improve the flotation separation of scheelite from calcite. Micro-flotation experiments of the individual minerals and artificially mixed minerals using oleic acid as a collector were conducted to evaluate the selective depression effect of the SS/Fe(II) depressant (the mixture of SS and Fe(II)). The results showed that the depressant mixture had a better selective depression effect on calcite than SS alone, and the optimum mass ratio of SS to ferrous sulphate was 4:1. Flotation tests using ore from a commercial mine showed that a concentrate with a WO<sub>3</sub> grade of more than 62% and recovery greater than 89% could be achieved using SS/Fe(II) as the depressant. The depression mechanism of the SS/Fe(II) was investigated in detail using zeta potential measurements and X-ray photoelectron spectroscopy. The results showed that silicate species are more likely to adsorb on the surface of calcite and produce a selective depression effect. The improved depression effect could be attributed to the Fe(II), which not only increased the adsorption of the silicate species on the calcite surface, but also improved the formation of polymeric silicate species.

## 1. Introduction

Tungsten is a strategically important industrial metal. Scheelite (CaWO<sub>4</sub>) is an important tungsten-containing mineral, and calcite (CaCO<sub>3</sub>) is a significant gangue mineral in many scheelite deposits (Feng et al., 2015; Ilhan et al., 2013). The most common flotation technique commercially used for the treatment of scheelite is depressing the calcite with sodium silicate, followed by floating with conventional fatty acid collectors (Deng et al., 2016; Hu et al., 2012; Huang et al., 2010). However, the separation of scheelite from calcite is difficult to achieve due to similar hydrophobicity in the presence of fatty acid collectors. (Gao et al., 2016; Ozcan and Bulutcu, 1993).

Sodium silicate (SS) is the most widely used depressant among the several depressants that have been tested in the flotation of scheelite ores (Al-Thyabat, 2009; Feng et al., 2012; Pradip, 1981; Viswanathan et al., 1965). The depression mechanism of SS for different minerals has been widely investigated (Ding and Laskowski, 2006; Qi et al., 1993; Silva et al., 2012). However, in most practical processes, using a single type of depressant is inadequate to achieve adequate differential flotation selectivity between scheelite and calcite. To achieve more

effective separation, many investigations have studied other depressant systems to improve the differential separation between scheelite and calcite, such as acidified SS (Feng et al., 2015), sodium fluosilicate (Ren et al., 2017), and sodium hexametaphosphate (Ni and Liu, 2013). In addition, metal ions (such as Al(III), Fe(II), Pb(II), and Cu(II)) as assistant depressants could improve the depression ability of SS in flotation systems (Feng et al., 2017; Fuerstenau and Fitzgerald, 1989; Mercade, 1983). The depressant effects of different metal hydroxy silicate hydrosols in flotation were first reported by Belash and Pugina (1946). Since then, numerous studies have been conducted (Yang, 1978; Mercade, 1981; Naik and Reddy, 2006). Oliveira and Sampaio (1988) investigated the influence of SS added alone or as a hydrosol with FeSO<sub>4</sub>·7H<sub>2</sub>O on the bench scale for a scheelite ore that was assayed at 0.34% from the Currais Novos mining district of northeast Brazil. They showed that the addition of ferrous sulphate had a marked effect on the effectiveness of the flotation. Recently, Feng et al. (2017) used a depressant of saline SS (a mixture of SS and lead ions) to separate scheelite from calcite. They found that this depressant had a selective depression effect on calcite.

Despite the improved separation of scheelite from calcite using

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**Table 1**  
Chemical composition of scheelite sample.

Component	SnO <sub>2</sub>	WO <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe(T)	SiO <sub>2</sub>	S	CaO	CaF <sub>2</sub>	Loss	Total
Content, wt%	0.06	0.38	7.89	1.97	27.63	0.15	35.42	8.58	17.92	100.00

metal ions as an assistant depressant, the underpinning mechanism has rarely been studied and is still unclear. In this study, Fe(II) was considered as an assistant depressant, and attempts were made to enhance the selective separation of scheelite from calcite. Micro-flotation, zeta potential measurements, and X-ray photoelectron spectroscopy (XPS) studies were undertaken to better understand the depression mechanisms.

## 2. Materials and methods

### 2.1. Materials and reagents

The scheelite sample used in the study was obtained from the Malipo mine in Yunnan grading 0.38% WO<sub>3</sub>. The results of an X-ray fluorescence chemical analysis of the ore samples are listed in Table 1. The largest proportion of the tungsten minerals is scheelite. The primary gangue minerals in the material are calcite, quartz, fluorite, and tremolite.

The scheelite and calcite samples used in the experiments were prepared from handpicked crystals obtained from Malipo deposits in Yunnan, China. They were washed with distilled water, dried, and were then processed in a porcelain ball mill, sieved, and classified to obtain the desired particle size fractions. The particle size range of 19–120 μm was used for micro-flotation and XPS analyses, whereas finer particles (< 19 μm) were ground to under 5 μm in an agate mortar. This fine product was used for zeta potential measurements. Optical mineralogical and chemical analyses and the X-ray powder diffraction (XRD, Bruker AXS, Germany) spectra indicated that the purities of the particles of calcite and scheelite exceeded 96%.

The reagents used in the study are listed in Table 2. Deionized water was used throughout the experiments, and all the experiments were conducted at 26 °C ± 1 °C. The SS/Fe(II) depressant was prepared by adding an aqueous solution of ferrous sulphate (with a mass percentage concentration of 10%) to an aqueous solution of SS (with a mass percentage concentration of 10%) and subsequently agitating at a high speed for 10 min. The SS used in this study had a SiO<sub>2</sub> to Na<sub>2</sub>O ratio of approximately 2.4.

### 2.2. Methods

#### 2.2.1. Micro-flotation

Micro-flotation experiments of the individual minerals and artificially mixed minerals were conducted in a mechanical agitation flotation machine with a rotational speed of 1500 rpm. In each test, 4 g of the mineral sample was placed in 40 mL of deionized water. After 1 min of agitation, a freshly prepared NaOH (3 × 10<sup>-4</sup> mol/L) solution was

**Table 2**  
Reagents used in experiments.

Chemical	Conc. (%)	Supplier	Role
Sodium carbonate	99.5	Merck	pH adjuster
HCl	–	Merck	pH adjuster
Sodium hydroxide	96	Merck	pH adjuster
Sodium silicate	97	Merck	Depressant
Ferrous sulphate (FeSO <sub>4</sub> ·7H <sub>2</sub> O)	98	Akzo Nobel	Depressant
Sodium isobutyl xanthate	–	Penn Chemical	Collector
Oleic acid	99	Merck	Collector
Terpineol	99	Penn Chemical	Frother

**Table 3**  
Size distribution and chemical analysis of the flotation feed sample.

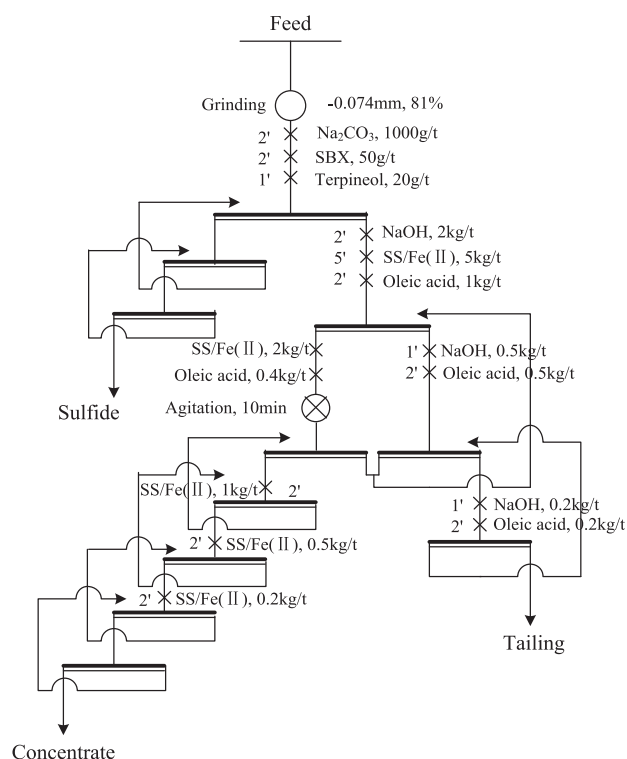
Size fraction (μm)	Mass recovery (%)	WO <sub>3</sub> Grade (%)
+74	19.00	0.35
–74 + 37	22.93	0.41
–37 + 19	22.98	0.44
–19 + 10	21.84	0.38
–10	13.25	0.28

added, and then the depressant with the desired concentration and oleic acid (2 × 10<sup>-4</sup> mol/L) were sequentially added. Their conditioning times were 2, 5, and 2 min, respectively. After adding the oleic acid, the flotation was performed for a period of 5 min.

For the flotation of individual minerals, the flotation recovery was calculated based on the solid weight of the floated mineral. For the flotation of the mixed minerals, the WO<sub>3</sub> grade of the concentrates and tailings were analyzed, and then the recoveries were calculated.

#### 2.2.2. Bench-scale flotation

The flotation feed (500 g, less than 2 mm) was ground to 81% passing 74 μm using a Φ200 × 400 mm XMB-type steel mill (China) at a pulp density of 60% (by weight). The size distribution and chemical analysis of the flotation feed samples are shown in Table 3. The liberation of scheelite (as determined by conventional optical mineralogy) is more than 87%. The pulp was conditioned at 35% solids with the reagents added in a 1.5 L XFD-type flotation machine (China). The primary sample flotation tests were conducted in accordance with the flowsheet shown in Fig. 1. After flotation, the floated products were



**Fig. 1.** Flowsheet for bench-scale flotation tests.

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