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# Comparative study on adsorption and depressant effects of carboxymethyl cellulose and sodium silicate in flotation



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#### ABSTRACT

Depressant effects of carboxymethyl cellulose (CMC) and sodium silicate on flotation of ilmenite and forsterite using sodium oleate (NaOL) as a collector were investigated by single mineral flotation, zeta-potential, Fourier transform infrared (FT-IR), X-ray photoelectron spectroscopy (XPS), and artificially mixed minerals flotation experiments. Single mineral flotation tests indicated that there was significant depressant effect for ilmenite and forsterite in the presence of CMC or sodium silicate, and the selective depressant using sodium silicate was more effective. FT-IR and XPS analysis suggested that CMC and sodium silicate were hardly interacted on the ilmenite surface. For forsterite, CMC adsorbed by hydrogen bonding and weakly chemical adsorption; however, sodium silicate chemically reacted with Mg on the forsterite surface, and made them hydrophilic. The depressant effect of forsterite could be attributed to that CMC and sodium silicate adsorbed on the forsterite surface, which inhibited the approach and adsorption of NaOL to the forsterite surface.

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#### 1. Introduction

The largest deposits of vanadium titanium magnetite resources in China are mainly distributed in the Panzhihua-Xichang area. The reserve of titanium reached  $8.7 \times 10^8$  t, as TiO<sub>2</sub>, occupying over 91% of the total reserve in China [1–4]. There are four mining sites including Panzhihua, Baima, Hongge, and Taibai in the Panzhihua-Xichang area. The Hongge possesses the largest reserve of vanadium titanium magnetite in Panzhihua-Xichang area [5]. With the increase of the exploration depth of vanadium titanium magnetite, the olivine content increased. The main silicon gangue mineral of the gabbro ore is titanaugite; however, for the onkilonite ore, the main gangue minerals are titanaugite and olivine [4, 6, 7]. Since the magnetic separation and the electrical separation require a large capital investment, and the gravity separation is an effective method only at coarse particle sizes, the flotation [8–12] is considered to be the most common method for separation of ilmenite and gangue minerals [13, 14].

The selective collectors [15–18] and depressants [19–22] are the key factors to achieve effective flotation separation [23–27] of ilmenite from gangue minerals. Currently, many collectors for the flotation of ilmenite have been investigated [13, 28, 29]. Sodium oleate (NaOL), a fatty acid collector, is widely used on flotation separation of ilmenite [13, 30]. For the case of selective depressants, the depressant effects on the

flotation of titanaugite are mainly on silicon gangue minerals, whereas the depressant effect on the flotation of olivine has been rarely investigated. An ilmenite concentrate of 47.4% TiO<sub>2</sub> was obtained [31] by flotation of an ilmenite ore containing 9% TiO<sub>2</sub> using acidified sodium silicate as a gangue depressant and benzyl arsonic acid as a collector. The acidified sodium silicate forms negatively charged colloidal silica that is selectively adsorbed on the gangue minerals, but does not affect the adsorption [32–34] of benzyl arsonic acid on the ilmenite surface.

Depressant SHQ (SHQ series are mixtures of quebracho and hexametaphosphate modified with surfactants) in combination with acidified silicate, showed the significant depressant effects of olivine, titanaugite, and other gangue minerals [13]. It was reported that the depressants of Na<sub>2</sub>SiO<sub>3</sub>, CMC, H<sub>2</sub>C<sub>2</sub>O<sub>3</sub>, Na<sub>2</sub>SiF<sub>6</sub>, (NaPO<sub>3</sub>)<sub>6</sub>, and sodium humate had certain depressing characteristic [14], but they had different selectivity to gangue minerals, CMC was shown to be one suitable depressant for the flotation of ilmenite. An ilmenite concentrate of 46.59% TiO<sub>2</sub> was obtained by flotation using CMC as a gangue depressant.

Feng et al. [35] reported the influence of copper ions and calcium ions on adsorption of CMC on chlorite. Calcium ions were not adsorbed onto the mineral surface, but they can interact with the CMC molecules, thus reducing the charge of the CMC. Copper ions were absorbed onto the mineral surface, which facilitated the CMC adsorption through chemical reaction. The effect of metal cations on the flotation behavior of CMC as the talc depressant was also studied previously [36]. Ca and Mg ions were found to be more effective than K ions promoting the

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depressant effect of CMC on the talc surface. It was suggested that iron recovery averaged above 90%, and the Fe concentrate of ~55% was obtained [37] by flotation of an ilmenite ore containing 42.8% Fe using sodium silicate as a gangue depressant and NaOL as the collector. The increase of sodium silicate modulus had a small influence on response variables. These cases indicate that CMC and sodium silicate have the selective depressant effects on the flotation separation of ilmenite and silicon gangue minerals.

To the best of our knowledge, the depressant effect on the flotation of olivine was rarely investigated in silicon gangue minerals. As the collectors may react with Fe and Mg ions on the olivine surface, and improve the flotation recovery of olivine, resulting in the poor flotation separation of ilmenite and olivine in the industrial production. Herein, it is beneficial to examine the depressant effects of ilmenite and forsterite, to improve the flotation separation of ilmenite and olivine. The objective of this study was to investigate the adsorption behavior also the depressant effects of CMC and sodium silicate on the flotation of ilmenite and olivine, using NaOL as the collector through singlemineral flotation experiments, zeta-potential measurements, FT-IR analyses, XPS analysis, and artificially mixed minerals flotation experiments.

#### 2. Experimental

#### 2.1. Materials and reagents

The ilmenite sample (FeTiO<sub>3</sub>, mainly FeO and TiO<sub>2</sub>) was obtained from vanadium titanium magnetite tailings through magnetic separation in the Hongge area. The ilmenite sample was repeatedly purified using low-intensity and high-intensity magnetic separation, and gravity separation. The purified ilmenite and forsterite samples (2MgO·SiO<sub>2</sub> or Mg<sub>2</sub>SiO<sub>4</sub>) were dry-ground in a ball mill. The samples were then dry sieved to obtain a particle size of  $-75 + 38 \,\mu\text{m}$ . The X-ray diffraction (XRD) patterns of the purified ilmenite and forsterite samples are shown in Fig. 1.

The XRD result indicated that the diffraction peaks of ilmenite and forsterite matched well with the pattern of standard diffraction peaks: ilmenite (PDF No. 3-781) and forsterite (PDF No. 7-74), respectively. The XRD patterns of ilmenite and forsterite showed that no other phases are found. The chemical compositions of the purified ilmenite and forsterite samples are presented in Table 1.

NaOL,  $CH_3(CH_2)_7CH$ — $CH(CH_2)_7COONa$ , was used as the anionic collector for the experiments. CMC,  $[C_6H_7O_2(OH)_2CH_2COOH]_n$ , and sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) were used as the depressants. Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) or sodium hydroxide (NaOH) was used for the pH adjustment. All of the chemicals were of analytical grade, and the water used for all the



Fig. 1. XRD patterns of the purified ilmenite (A) and forsterite (B).

#### Table 1

Chemical compositions of the purified minerals (mass fraction, %).

Sample	TFe	TiO <sub>2</sub>	SiO <sub>2</sub>	$Al_2O_3$	CaO	MgO	Others	Total
Ilmenite	31.07	50.4	2.37	0.51	0.28	5.24	10.13	100.00
Forsterite	6.62	0.03	44.4	1.00	0.13	47.56	0.26	100.00

experiments was deionized water (DI water) with a resistivity of 18.3 M  $\Omega$  cm.

#### 2.2. Flotation experiments with single minerals

The flotation experiments were conducted in a 200 mL flotation cell. The mineral particles (10.0 g) were placed in a cell, and then filled with 175 mL of pure water. The mixture of the mineral-water suspension was pre-conditioned for 1 min to obtain a well-dispersed suspension. The pH of the suspension was adjusted by  $H_2SO_4$  or NaOH for 3 min. The depressant, CMC or sodium silicate, was added and agitated for 3 min, and then, NaOL was added and again agitated for 3 min. The pH of the suspension was measured before the flotation. The flotation was conducted for 4 min. The concentrates were weighed after filtering and drying, and then, the flotation recoveries were calculated [(% recovery = weight of foam product / weight of feed) × 100]. Each test was measured no less than three times under the same experimental condition, and the mean value was taken as the final result, with the standard deviations as error bars.

#### 2.3. Zeta potential measurements

The zeta potentials measurements for the samples were carried out using a Zetasizer Nano Zs90 (England, Malvern Instrument Co.). The pH of the suspension was monitored continuously during the measurement at room temperature (25 °C). The purified mineral was ground to  $-2 \ \mu$ m using an agate mortar. The suspension was prepared by adding 30 mg of the purified mineral particles to 50 mL of DI water in a 100-mL Erlenmeyer flask. The collector or the depressant was added, and then, the prepared suspension was conditioned by magnetically stirring for 5 min, during which the pH of the suspension was measured. After settling for 10 min, the supernatant of the dilute fine particle suspension was obtained for zeta-potential measurements. The results presented in this paper are the mean of three independent measurements, with a typical variation of  $\pm 1.0 \ mV$ .

#### 2.4. FT-IR analysis

The FT-IR spectra were obtained using a Spectrum One (Version BM) FT-IR instrument (America, PerkinElmer Instrument Co.). Approximately 1% (mass fraction) of the solid sample was mixed with spectroscopic grade KBr. The wave number range of these spectra was 400–4000 cm<sup>-1</sup>. These spectra were recorded with 32 scans at 2 cm<sup>-1</sup> resolution. The mineral particles were placed in a cell with H<sub>2</sub>SO<sub>4</sub> or NaOH as the pH regulating reagent. Next, the purified samples were conditioned for another 3 min with NaOL, and the depressant was added and agitated for 3 min. Subsequently, the solid samples were washed three times using DI water at the same pH. The washed samples were vacuum dried at 55 °C prior to FT-IR analysis.

#### 2.5. XPS analysis

XPS tests for the samples were performed on a Kratos AXIS Ultra XPS system equipped with a monochromatic Al X-ray source operating at 150 W (England, Kratos). Each measurement started with a survey scan from 0 to 1350 eV, dwell time of 8 s, and band pass energy of 150 eV at steps of 1 eV. For the high-resolution measurement, the number of sweeps was increased, the band pass energy was lowered to

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