



## The separation of kyanite from quartz by flotation at acidic pH



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### ABSTRACT

An effective separation of kyanite from quartz by flotation is a challenge due to their similar surface properties and surface activation by metal ions. This study investigated the effect of pH, Fe<sup>3+</sup> and sodium silicate on the separation of kyanite from quartz using anionic collector, sodium petroleum sulfonate (SPS). Zeta potential measurement, infrared spectroscopic analysis and chemical speciation were conducted to identify the underpinning mechanisms. It was found that a good separation of kyanite from quartz was achieved at pH = 4.0 due to the selective adsorption of SPS on kyanite. However, the presence of Fe<sup>3+</sup> altered the electrical property on quartz which became attractive to SPS, enhanced quartz flotation and hence exacerbated the separation of kyanite from quartz. It is interesting that sodium silicate selectively depressed the flotation of iron-activated quartz by covering the activation sites and prohibiting collector adsorption. Although sodium silicate also interacted with kyanite, SPS still adsorbed on kyanite through reacting with Al atoms and therefore the depression of kyanite flotation by sodium silicate in the presence of Fe<sup>3+</sup> was limited.

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### 1. Introduction

Kyanite is an important metamorphic nesosilicate with a chemical formula of Al<sub>2</sub>O<sub>3</sub>·SiO<sub>2</sub> (Althaus, 1967). After calcination at temperatures higher than 1000 °C, kyanite can be transformed to mullite (3Al<sub>2</sub>O<sub>3</sub>·2SiO<sub>2</sub>) which is a high-grade refractory mineral with excellent heat and thermal shock resistance and particularly good creep resistance (McMichael, 1990). In addition, kyanite is also widely used to produce high performance lightweight aluminum-silicon alloys, fabricate metallic fibers and the leading edge of supersonic aircrafts and spaceships (Belogurova and Grishin, 2012; Lin et al., 2011; Skoog and Moore, 1988; Zhang et al., 2013).

The grade of most kyanite deposits in the world is between 10% and 25% and therefore a concentration process is necessary in order to produce kyanite products which can meet industrial needs (Lin et al., 2011). The traditional concentration methods are magnetic separation and gravity separation. Nowadays, flotation is becoming the dominant method for kyanite beneficiation due to decreased ore grades and increased industrial demands for higher grade products (Dong et al., 1993; Brandao and Mendes, 1998). In flotation plants, the selection of collector mainly depends on slurry pH. In general, fatty acid collectors are used at alkaline pH,

sulfonate collectors at acidic pH, and a mixture of both collector types at neutral pH (Dong et al., 1993; Sun and Yin, 2001).

The pH plays an important role in the selective flotation of kyanite from silicate gangue minerals. Amanullah et al. (1990) studied the flotation of a low grade kyanite ore containing mica and quartz and found that a better separation of kyanite from gangue minerals was achieved when using sulfonate collectors at acidic pH than using sodium oleate at alkaline pH. Xia et al. (1994) investigated the separation of kyanite from quartz by using three types of collector including sodium oleate at pH 8.5–9.5, sodium alkyl benzene sulfonate at pH 3.0–4.0 and an emulsion which is a mixture of distilled tall oil, saturated fatty acids, hydrocarbon oil and sodium hydroxide at pH 6.5–7.5. They found that sodium alkyl benzene sulfonate at acid pH showed the best selectivity. Wang (2010) also reported that the recovery and grade of kyanite flotation concentrate were improved by 2% and 8%, respectively, after changing the collector from sodium oleate at pH 9.0 to sodium petroleum sulfonate at pH 3.5. The poor separation of kyanite from quartz at alkaline pH is mainly associated with the undesirable mineral surface properties. Both kyanite and quartz are negatively charged at alkaline pH and their surface properties are also similar, limiting the selective adsorption of collector on kyanite. Despite the better separation of kyanite from quartz at acidic pH, the underpinning mechanism has been rarely studied. In this study, it is hypothesized that sulfonate collectors preferentially adsorb on kyanite and enhance the separation of kyanite from quartz.

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Another problem in kyanite flotation is the undesirable activation of silicate gangue minerals by metal ions. One of the common ions is  $\text{Fe}^{3+}$  released during grinding and flotation from iron oxides contained in kyanite ores. It has been found that iron ions exacerbate the separation of kyanite from quartz (Mao et al., 1995; Zhang et al., 2014). It seems that iron hydroxides precipitate on mineral surfaces and consume the collector, which is the subject of this study. Fuerstenau et al. (1963) found that quartz could be activated by  $\text{Fe}^{3+}$  at acidic pH, increasing the collector adsorption on quartz and therefore quartz flotation. However, how  $\text{Fe}^{3+}$  affects kyanite flotation is not well understood, which is also the subject of this study.

The depression of activated quartz is necessary in order to achieve efficient separation of kyanite from quartz. Sodium silicate is used to depress the flotation of quartz activated by metal ions. Mao et al. (1995) studied the effect of  $\text{AlCl}_3$  and sodium silicate on the separation of kyanite from quartz at alkaline pH in the presence of sodium oleate. They found that the addition of sodium silicate strongly depressed the flotation of  $\text{Al}^{3+}$  activated quartz. The depression effect was mainly caused by the adsorption of sodium silicate on quartz, prohibiting the reaction of sodium oleate with quartz. Liu and Sun (2011) also indicated that sodium silicate was an effective depressant for iron-activated quartz. They proposed that the hydrolysis of sodium silicate produced negatively charged colloidal silicate and  $\text{SiO}(\text{OH})_2^-$  which adsorbed on quartz and then reduced the surface hydrophobicity. Again, it is not clear how sodium silicate affects kyanite flotation.

In this study, the flotation behavior of both kyanite and quartz and their separation were studied at acidic pH in the presence of sodium petroleum sulfonate.  $\text{FeCl}_3$  was added to introduce  $\text{Fe}^{3+}$  ions to activate quartz and kyanite. Sodium silicate was used as a depressant to achieve the separation of kyanite from quartz in the presence of  $\text{Fe}^{3+}$  ions. The interaction of sulfonate collector,  $\text{Fe}^{3+}$  ions and sodium silicate with both minerals was studied by zeta potential measurement, FTIR analysis and chemical speciation. The purpose of this study was not to identify the optimal flotation conditions to achieve the separation of kyanite from quartz, the significant factors and the interaction of individual factors. Instead, the underpinning mechanisms responsible for the separation of kyanite from quartz under conditions that were hypothesized to reflect the variation were studied.

## 2. Experimental

### 2.1. Materials and reagents

Kyanite and quartz single minerals were supplied from a kyanite mine in Nanyang, Henan, China. High grade kyanite and quartz were handpicked, crushed in a roll crusher, dry ground in a porcelain mill and then screened to collect  $-0.10$  mm  $+0.074$  mm particle size fractions. The mineral surfaces were cleaned by immersing minerals in 10% HCl for 24 h and then rinsed in DI water. The samples were dried in a vacuum oven. Table 1 shows the chemical composition of the samples analyzed by XRF.

Sodium petroleum sulfonate (SPS) was used as the collector. It is of industrial grade and has an average molecular weight of 480. Sodium metasilicate ( $\text{Na}_2\text{SiO}_3$ ) and iron(III) chloride ( $\text{FeCl}_3$ ) used as the depressant and activator, respectively, are of AR grade.

**Table 1**  
Chemical components of kyanite and quartz (%).

	$\text{Al}_2\text{O}_3$	$\text{SiO}_2$	$\text{Fe}_2\text{O}_3$	$\text{K}_2\text{O}$	CaO	Ignition loss
Kyanite	62.76	35.65	0.15	0.02	0.08	1.04
Quartz		99.82	0.09			

HCl and NaOH were used to adjust slurry pH and both of them are of AR grade. De-ionized (DI) water was used in all experiments.

### 2.2. Flotation tests

A laboratory mechanical flotation cell with a volume of 35 mL was used in this study. The impeller speed was 2000 rpm and the air rate was  $20 \text{ mL min}^{-1}$ . 2.0 g of single mineral was firstly mixed with 35 mL DI water in the flotation cell for 1 min. pH modifier was then added and conditioned for 2 min to adjust the pulp pH. Activator or depressant was added and conditioned for 2 min. Collector was then added with a conditioning time of 3 min. The total flotation time was 3 min and the concentrate was collected by manually scraping. Both the concentrate collected and the tailing remained in the cell were dried and weighed for calculating flotation recovery. Repeat tests were conducted throughout this study and the standard deviation was less than 2% recovery.

Experimental design was conducted to identify the collector dosage and pH to allow the effective flotation of kyanite,  $\text{Fe}^{3+}$  dosage for the activation of  $\text{Fe}^{3+}$  on quartz and sodium silicate dosage for the prevention of flotation of  $\text{Fe}^{3+}$ -activated quartz to introduce conditions that directly affected the variation. Under these conditions, zeta potential measurement, infrared spectroscopic analysis and chemical speciation were conducted.

### 2.3. Zeta potential measurement

For zeta potential measurement, the samples were ground in an agate mortar. The particle size of the ground powder was below  $2 \mu\text{m}$  as measured by the Leica DMLP Microscope. 30.0 mg of ground minerals was mixed with 50 mL DI water in a beaker. After stirring for 2 min, HCl or NaOH was added to adjust the slurry pH. A certain amount of collector was added into the slurry and stirred for another 6 min. The slurry was then injected into a Tiselius cell in a ZetaPlus Zeta Potential measurement unit. Each sample was measured three times and the average zeta potential with standard deviation was reported.

### 2.4. Infrared spectroscopic analysis

Fourier transform infrared spectroscopy (FTIR) analysis was conducted by a Nicolet iS10 FTIR Spectrometer. Slurry samples were collected directly from the flotation cell after conditioning with the reagents and then dried in a vacuum oven. A small amount of mineral samples was mixed with KBr and ground in an agate mortar. The ground powder was pressed into a pellet and then transferred into the spectrometer for measurement.

## 3. Results and discussion

### 3.1. Flotation behavior of kyanite and quartz

Flotation tests were conducted by using kyanite and quartz single minerals. The effect of pH on the flotation recovery when using  $3.0 \times 10^{-4} \text{ mol L}^{-1}$  sodium petroleum sulfonate (SPS) as the collector is shown in Fig. 1. Kyanite recovery increased significantly with pH and reached a maximum of 58% at pH 4 and then started to decrease. A similar trend was found when floating quartz, however, quartz recovery was much lower than kyanite recovery across the pH range and the highest quartz recovery was only about 12%.

The effect of SPS concentration on the flotation recovery of kyanite and quartz at pH 4.0 is shown in Fig. 2. Kyanite recovery increased with SPS concentration and reached the maximum of 92% at  $5 \times 10^{-4} \text{ mol L}^{-1}$  SPS. Kyanite recovery remained unchanged after further increasing the SPS concentration. Quartz

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