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Evaluation of sodium petroleum sulfonates with different molecular weights for flotation of kyanite ore

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Abstract: The flotation performance of sodium petroleum sulfonates with different molecular weights was evaluated for flotation of a kyanite ore, by investigating valuable mineral recovery-grade, flotation kinetics and gangue entrainment. The results indicated that the higher molecular weight of agent, the higher final cumulative kyanite recovery was, with the maximum value of 72% being obtained with KY-3 with the molecular weight of 438. The final cumulative kyanite grade initially increased, and then decreased with the molecular weight increasing. In other words, the maximum final cumulative kyanite grade (i.e. 89.05%) was obtained with KY-2 with the molecular weight of 392. The kyanite flotation kinetics followed the first order kinetics well, while the modified flotation rate constant showed a decreasing trend after the initial increase as the molecular weight increased. In addition, the overall entrainment degree decreased with decreasing molecular weight of sodium petroleum sulfonates. The use of KY-2 in kyanite flotation was an attractive option in comparison with KY-1 and KY-3.

Keywords: *evaluation, sodium petroleum sulfonate, kyanite, flotation kinetics, entrainment*

Introduction

Kyanite, as a kind of high-alumina silicate mineral with a theoretical composition of 37.10% SiO_2 and 62.90% Al_2O_3, is widely used in industry as a raw material for refractory production and ceramic synthesis (Lin, 2011; Zhang et al., 2013; Kustov, 2014; Guo et al., 2015).

Currently, the most efficient way to beneficiate low grade and complicate kyanite ore is flotation due to the difficult sorting features of this mineral (Sweet, 1994; Bulut and Yurtsever, 2004). Flotation of kyanite can be conducted by alkaline, neutral or acidic environment, with the last being more widely used in industry due to its better selective properties and simple reagent system (Jamerson et al., 2001). For instance,

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Yang et al. (2003) found that the grade of concentrate obtained from acid condition was greater than that from alkaline one by 6.97%, although the recovery was similar. The flotation results of Bitlis kyanite ore using different collectors showed that the collecting capability of cationic collector dodecylamine hydrochloride was inferior to anionic sulfonate collector AERO series (sodium petroleum sulfonates), with a Al_2O_3 concentration of 58–60% and a recovery of over 80% (Bulut and Yurtsever, 2004).

As a collector in flotation of kyanite, sodium petroleum sulfonate is extensively used in industry. This reagent normally contains molecules with different weights and polarities (Hou et al., 2000). The main component (active substance) of sodium petroleum sulfonate is sulfonate, which has a highly-hydrophilic sulfogroup connected with alkyl, giving rise to a structural formula of RSO_3Na, where R is an alkyl group (Zhu et al., 2015). Other important parts included neutral oil and water etc. which also affect the collection performance of sodium petroleum sulfonate (Wang, 1992).

There are many successful practices in kyanite beneficiation with sodium petroleum sulfonate as a collector. A mica-quartz-bearing kyanite ore in India was treated by reverse flotation to remove mica firstly, which was then floated using M-500 (a kind of sodium petroleum sulfonate with molecular weight 500–550, sulfonate 60–62%, mineral oil 30–35% and water 4–5% etc.), resulting in a Al_2O_3 grade of 59.18% and a kyanite recovery of 72.7% in the concentrate (Amanullah et al., 1990). Shi et al. (2011) conducted flotation tests with sodium petroleum sulfonate (molecular weight 450) to separate kyanite and quartz, increasing the kyanite grade from 50.00% to 94.26% and giving a recovery of 96.11%. Na (1998) analyzed three sodium petroleum sulfonates with different molecular weight ranges (250–350, 350–500 and greater than 500) as collectors, and concluded that the sodium petroleum sulfonate with a molecular weight of 350–500 was considered to be a good flotation agent for oxide ore separation, including kyanite. Therefore, molecular weight of sodium petroleum sulfonates has an influence on their flotation performance, in line with the results of Na (1998).

The evaluation of flotation reagents is normally carried out in a rather arbitrary manner in industry. The most common way is the valuable mineral recovery-grade way, evaluating reagents through comparing the recovery and grade of valuable mineral, which ignores the flotation rate and other reactions in flotation process (Bhattacharya and Shobhana, 2008). The flotation process is a rate phenomenon, so it is important to determine the flotation recovery as a function of time, which is an effective comparison criterion when evaluating the reagents for mineral flotation (Agar and Barrett, 1983; Oliveira et al., 2001; Bhattacharya and Shobhana, 2008). Apart from the recovery, the grade is another important index in flotation. The change of grade as a function of different reagents needs analysis to assess the selectivity of reagents (Bhattacharya and Shobhana, 2008). The non-selective process of gangue recovery has an obvious influence on the concentrates grade, in which entrainment has a preponderant action, especially in the flotation process of fine particles (Pita, 2015). Reagents with different properties show different entrainment behaviors in a flotation process (Melo and Laskowski, 2006; Gong et al., 2010; Wang et al., 2015), so it is also vital to evaluate the flotation performance of reagents by researching the influence of different flotation agents on gangue entrainment.

Sodium petroleum sulfonate has been widely used in flotation, but the evaluation of sodium petroleum sulfonate as the collector has not attracted sufficient attention. Therefore, this study evaluates flotation performance of sodium petroleum sulfonates with different molecular weights for flotation of the kyanite ore through valuable mineral recovery-grade, flotation kinetics and gangue entrainment analysis.

Experimental

Materials

The kyanite samples used in this work were obtained from Nanyang, Henan Province, China. The particle size of the samples is shown in Table 1, which was determined by screening. The liberation size of kyanite was 150μm.

Table1. Particle size distribution of the samples

Particle size (μm)	Yield (%)	Cumulative undersize yield (%)
+100	2.86	100.00
-100+74	8.48	97.14
-74+45	16.78	88.66
-45	71.88	71.88

The chemical composition of the samples was determined by an X-ray fluorescence spectrometer (XRF) (Axios advanced, PAN Alytical B.V., Netherlands) and the mineralogical analysis indicated by X-ray diffraction (D/ Max-IIIA, RIGAKU, Japan) with monochromatic Cu Kα (λ = 0.15418 nm) radiation at 35 kV, 30 mA in 5–70° (2θ) at a rate of 0.02°/s. The results are presented in Table 2 and Fig. 1, respectively.

Table 2. Chemical composition of the samples

Chemical composition	Al_2O_3	SiO_2	TiO_2	P_2O_5	CaO	K_2O	Fe_2O_3
Percentage, %	47.41	46.55	1.22	0.54	0.34	0.24	0.19
Chemical composition	Na_2O	SO_3	BaO	ZrO_2	SrO	Ignition loss	
Percentage, %	0.10	0.04	0.02	0.02	0.01	3.33	

According to the XRF analysis and XRD pattern, the approximate mineral composition of samples is summarised in Table 3.

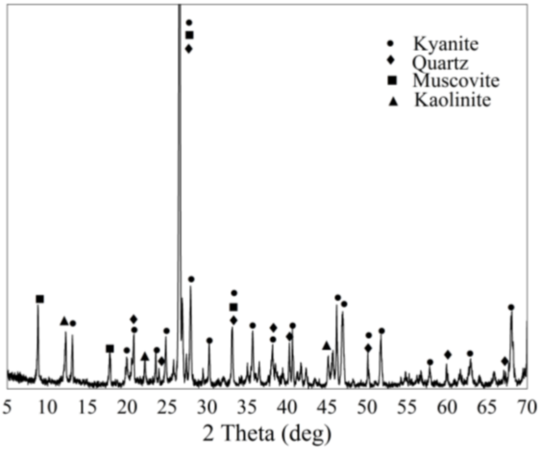

Fig.1. The XRD pattern of the samples

Table 3. Mineral composition of the samples

Mineral composition	Kyanite	Quartz	Muscovite	Kaolinite	Others
Content, %	68	27	2	2	1

The linear chain sodium petroleum sulfonates synthesized using the same raw materials were named as KY-1, KY-2, KY-3, with the molecular weights of 326, 392 and 438, respectively, and the active substance content of 42.17%, 45.00% and 40.02%, successively. Analytical grade sulfuric acid was used to adjust the pH value of the flotation system.

Flotation tests

The flotation tests were conducted in a RK/ FD-type laboratory flotation machine (1.0 dm^3) with a constant impeller speed of 2000 r/min at 25 °C. For each test, 200 g samples were used, with water being subsequently added to a marked level, and then the pH regulator and collector were added to the pulp successively with each conditioning time of 3 minutes. During the flotation process, acid liquor was replenished to keep the liquid level to the constant level, with pH being controlled within 3.0–3.5 (Sun and Yin, 2001). A total of six concentrates were skimmed off automatically at 0.5, 1, 1.5, 2.5, 4 and 6 min into six basins.

After 6 min, the six concentrates were dried at 100 °C in oven for 6 hours, and then weighed. In addition, the amount of water used during the flotation test was recorded. All concentrates and tailings were assayed by GKF-VI Rapid Multi Element Analyzer, whilst the water recovery calculated followed the methods reported in Li et al. (2014).

Analysis of the kinetics data

It is well known that flotation kinetics can be studied similarly to chemical kinetics and many models can express it correctly (Polat and Chander, 2000; Vinnettet al., 2015; Asghar et al., 2015). These models have been widely used during the last decades, and the concentration in a flotation cell can be modeled by Eq.(1):

$$\frac{\mathrm{d}C}{\mathrm{d}t} = -K \cdot C^{n} \tag{1}$$

where C is the concentration of mineral in the flotation cell, t is flotation time, K is the flotation rate constant, n is the kinetic order (Vinnett et al., 2015).

The first order model is the most commonly used in flotation (Yekeler, 1997; Melo and Laskowski, 2006; Uçurum M, 2009; Luo et al., 2015; Asghar A et al., 2015; Trumic and Antonijevic, 2016). Zhang (2014) analyzed the flotation kinetic of kyanite using Matlab, and concluded that the flotation process can be simulated by a classical first order flotation kinetic model. In this study, we assumed that n=1 in Eq. (1) which therefore can be transferred to:

$$R = R_{\infty} \cdot [1 - \exp(-K \cdot t)] \tag{2}$$

where K is the first-order rate constant, R_{∞} is the maximum recovery after prolonged flotation time. R_{∞} can be calculated based on the methodology presented elsewhere (Ding, 1991; Luo et al., 2015):

$$R_{\infty} = R_{m} + \frac{R_{m} - R_{m-1}}{t_{m} - t_{m-1}} \cdot t_{m-1} \tag{3}$$

where m is the time of skimming the concentrate. Equation (2) can further result in the following form shown in Eq. (4):

$$K \cdot t = \ln R_{\infty} - \ln(R_{\infty} - R) \,. \tag{4}$$

Generally, when a test condition is changed, both R_{∞} and K will change at the same time. Therefore, the modified flotation rate constant was used to deal with the problem (Xu, 1998; Uçurum, 2007):

$$K_{m} = R_{\infty} \cdot K. \tag{5}$$

The selectivity index of mineral I over mineral II in this flotation system is defined as the ratio of the K_m of kyanite to the K_m of gangue (Xu, 1998):

$$SI = \frac{\text{Km of kyanite}}{\text{Km of gangue}} \,. \tag{6}$$

Results and discussion

Flotation

Figure 2 shows the flotation cumulative grade of kyanite as a function of flotation time using sodium petroleum sulfonates as collectors with a dosage of 0.3 g/ dm^3. The results indicate that the grades of kyanite floated with KY-2 and KY-3 decreased slightly with the flotation time, while it remained almost constant for KY-1 as the collector. It should be noted that the best cumulative grade of kyanite equl to 89% was obtained with KY-2, and it was close to KY-3, while the grade of kyanite in flotation with KY-1 was significantly lower.

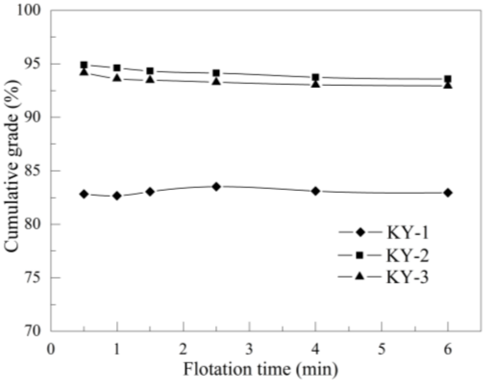

Fig. 2. The influence of KY-1, KY-2 and KY-3 on kyanite grade

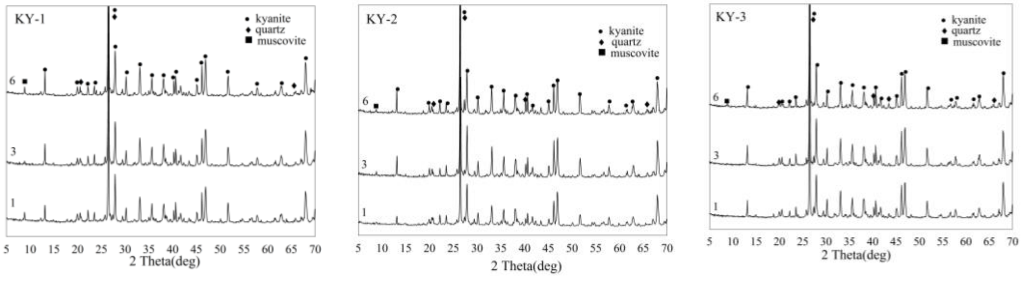

Fig. 3. XRD patterns of concentrates obtained with KY-1, KY-2 and KY-3 as collectors (1 and 3, 6 represent the first, third and sixth concentrates, respectively)

The XRD patterns of the first, third and sixth concentrates are shown in Fig. 3. As seen the main mineral in the concentrates was kyanite, with gangue being quartz with small amount of muscovite. It indicates that the gangue such as quartz, muscovite and kaolinite can be removed significantly from the kyanite samples, resulting in a high concentrate grade of kyanite. The peaks of kyanite are strong and sharp, reflecting an ordered and high-crystallinity structure, showing that the floatation process did not change the crystallinity structure of kyanite (Li et al., 2015). For KY-1, the peaks of gangue in the third concentrate are weaker than that of the first and the sixth concentrates, which is in line with the results presented in Fig. 2. The peaks of gangue became stronger as the flotation proceeded with KY-2 and KY-3, especially for muscovite, while almost not observed in the first concentrates.

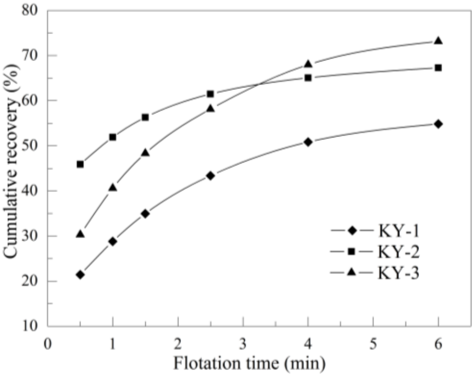

Fig. 4.The influence of KY-1, KY-2 and KY-3 on kyanite recovery

Figure 4 shows the cumulative recovery of kyanite as a function of flotation time using sodium petroleum sulfonates as collectors. The cumulative recovery of kyanite increased as flotation proceeded. In addition, the greater molecular weight of sodium petroleum sulfonate, the greater kyanite recovery equal to 72, 67 and 54% for KY-3, KY-2 and KY-1, respectively.

According to the grade and recovery of concentrates, the selectivity index of the three sodium petroleum sulfonates followed the order of KY-2 (2.031) > KY-3 (1.852) > KY-1 (1.704), which was calculated with Eq. (6). However, the collecting capacity in the descending order was: KY-3, KY-2 and KY-1 as the recovery of kyanite were 72.36%, 67.09% and 54.07%, respectively, illustrating that with the increase of molecular weight of sodium petroleum sulfonates from 326 to 438, the collecting capacity of sulfonates increased correspondingly. Therefore, it is hard to identify the best collector on the basis of grade and recovery. Many researchers investigated the influence of reagents on the flotation kinetics of minerals (Jowett and Ghosh, 1964; Agar and Barrett, 1983; Hosten and Tezcan, 1990; Adkins and Pearse, 1992; Bhattacharya and Shobhana, 2008; Kowalczuk et al., 2016) and entrainment of gangue (Melo and Laskowski, 2006; Gong et al., 2010). Also in this paper the influence of three different sodium petroleum sulfonates on kyanite flotation behaviour is discussed.

Kinetics

According to Eq. (4), the relationship between $\ln R_\infty - \ln(R_\infty - R)$ and flotation time is illustrated in Fig. 5 where kinetics parameters such as the flotation rate constant and R^2

are shown. The flotation kinetics of kyanite was fitted with Origin software and the results are showed in Table 4. As noted, R^2 is close to 1, indicating that flotation behavior of kyanite with three sodium petroleum sulfonates as collectors follows the first order model. It is obvious that R_∞ increases as the molecular weight increases, which may be due to better hydrophobicity derived from longer carbon chain (Na, 1998; Zhao and Zhu, 2003). The flotation rate constant K increases when the molecular weight of sulfonates increases from 326 to 396, which however declines apparently when the molecular weight increases from 396 to 438. K has an obvious relation with active substance content, the higher active substance content, the higher flotation rate constant K. Similarly, K_m increases initially and then decreases as molecular weight of sodium petroleum sulfonates increases, with the minimum K_m for KY-1. Therefore, as compared to KY-1 and KY-3, KY-2 is the best collector to obtain a fast flotation process.

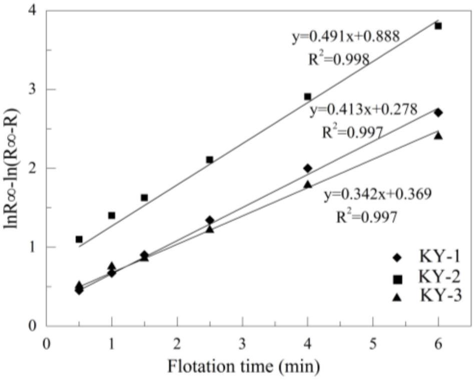

Fig. 5. Kinetic models of three collectors in kyanite flotation

Table 4. Effect of three collectors on the flotation rate of kyanite

Collector	MW, g/mol	R_∞, %	K, min^{-1}	K_m, %·min^{-1}	R^2
KY-1	326	58.77	0.413	24.242	0.997
KY-2	392	68.81	0.491	33.786	0.998
KY-3	438	80.42	0.342	27.504	0.997

Entrainment

Much attention has been paid to understand the mechanisms how gangue was floated into the concentrates, while entrainment was supposed to be a primary reason (Kirjavainen, 1996; Neethling and Cilliers, 2002; Melo and Laskowski, 2006; Wang et al., 2015). It is of great significance to analyse the entrainment of gangue when separate material with fine grain size, especially the mineral particles with a size less than 50 μm that are more easily to be recovered by entrainment (Savassi et al., 1998; Wang et al., 2015).

In this study, the main gangue was quartz, others like muscovite, kaolinite and etc. All could hardly be recovered by true flotation under the fixed flotation condition (Sun and Yin, 2001; Shi et al., 2011), and it was assumed that the gangue was recovered to the concentrates by entrainment only. In addition, the particles with a size less than 50 μm accounted for more than 71.88%, so it was necessary to investigate the gangue recovery by entrainment of fine mineral particles with different sodium petroleum sulfonates as the collectors. As entrainment is strongly associated with the water recovery and influences both mineral recovery and concentrate grade, the water recovery is one of the key parameters in analyzing entrainment (Zheng et al., 2006; Wang et al., 2015). Many attempts have been tried to study the relationship between water recovery and entrainment in the last decades (Neethling and Cilliers, 2002; Zheng et al., 2006; Wang et al., 2015). The relationship between the cumulative water gangue recoveries is shown in Fig. 6.

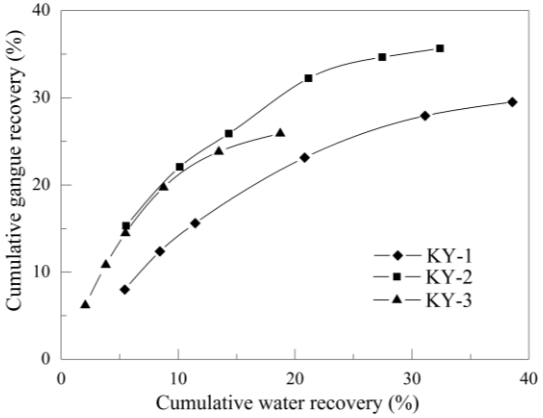

Fig. 6. Relationship between water recovery and gangue recoveries

Figure 6 shows that the cumulative gangue recovery increased when the cumulative water recovery increased. As indicated in Sun and Yin (2001) and Zhao and Zhu (2003), sodium petroleum sulfonate had both collecting and frothing capacities. When molecular weight reduced the frothing capacity (i.e. foam output) increased (Zhang and Que, 2008), resulting in increase in the water recovery. Therefore, it is possible to control the frothing capacity of reagents to decrease the entrainment of gangue. The degree of entrainment can be calculated by using formula:

$$R_{S,i} = ENT_i \cdot R_{W,i} \quad (7)$$

where $R_{S,i}$ is the recovery of the particles in the i^{th} flotation time by entrainment, while $R_{W,i}$ is the water recovery during the same time. ENT_i is the total of entrainment and entrapment factors (Zheng et al., 2006; Li et al., 2015; Wang et al., 2016). The relation between *ENT* of three collectors and flotation time is shown in Fig. 7.

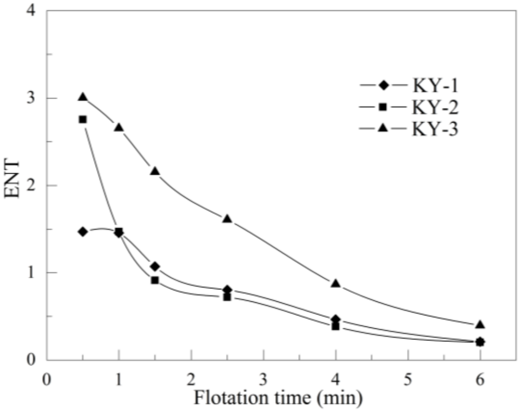

Fig. 7. The influence of flotation time on *ENT*

According to Figure 7, *ENT* dramatically decreases with flotation time, what is in line with the results of Wang et al. (2016). As fine particles tend to behave as a part of liquid phase, the particles with smaller sizes can be recovered firstly in water constituting the froth phase with bubble (Güler and Akdemir, 2012). In contrast, coarse particles are less likely to be entrained in the initial stage compared with fine particles (Zheng et al., 2006; Wang et al., 2016), and the ratio of coarse particles in concentrates increased with the flotation time, indicating that coarse gangue particles were more likely recovered in the latter stage.

Compared to KY-2, the overall *ENT* of KY-3 was greater during the whole flotation period, which was consistent with that shown in Fig. 2. The low *ENT* of KY-1 and the greatest water recovery may result from the strongest frothing capacity of KY-1. As the molecular weight of sodium petroleum sulfonates increased, the surface activity increased, resulting in the decline of the water recovery. The overall *ENT* of KY-1, KY-2 and KY-3 were 0.76, 1.10 and 1.38, respectively, indicating that *ENT* increased with the increased molecular weight of the collectors. It agrees well with the research of Kracht et al. (2016) and Lu et al. (2015). Zheng et al. (2006) and Li et al. (2015) concluded that if entrapment was an additional mechanism for the recovery of liberated gangue particles, it was possible in practice for the calculated entrainment factor value to be greater than 1. Apart from entrainment, entrapment may be an additional mechanism for gangue recovery in this study, which may be due to the occlusion of fine gangue minerals within the flocs (Polat et al., 2003; Wang and Peng, 2013).

Conclusions

Sodium petroleum sulfonates with different molecular weights as collectors for flotation of kyanite were evaluated through flotation tests, flotation kinetics analysis and gangue entrainment analysis. Greater cumulative recovery of kyanite was achieved when the sodium petroleum sulfonates with greater molecular weight was applied as collectors, e.g. the maximum recovery of 72.36% was observed when KY-3 (molecular weight is 438) was used. The cumulative grade of kyanite increased when the molecular weight of sulfonate increased from 326 to 396, but decreased afterwards, resulting in the maximum value of 89.05% for KY-2. The kyanite flotation followed well the first order kinetics model. With decrease in the molecular weight of sodium petroleum sulfonates, the water recovery increased, but the overall ENT decreased. The use of KY-2 in froth flotation processes is an attractive option in comparison with KY-1 and KY-3.

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