Physicochem. Probl. Miner. Process., 61(1), 2025, 201418

http://www.journalssystem.com/ppmp

Physicochemical Problems of Mineral Processing

ISSN 1643-1049

The correlation between the pulp rheology and the flotation performance in a scheelite ore: from the flotation kinetic perspective

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Abstract: Pulp rheology plays a very important role in deciding the subtle process of flotation. Here the correlation between the flotation kinetics of a scheelite ore and the pulp rheology was studied through batch flotation test and rheology measurement with data fitting. The pulp rheology was controlled by pulp concentration, adding surface inert gangue mineral-andradite with different size and content. Afterwards, the cumulative flotation grade and recovery as a function of flotation time was recorded with further kinetic analysis corresponding to their rheological properties including the yield stress and the degree of deviation from Newtonian fluid (D). The results showed that adding the coarse andradite (+75~-100 μm) at the mass ratio of 20/6 could selectively increase the flotation rate co-efficient for the slow floating fractions (from 0.24 min^{-1} to 0.63 min^{-1}), which was responsible for the increased concentrate grade. In the meantime, the yield stress was in positive correlation with the degree of deviation from Newtonian fluid, indicating that the ideal Newtonian fluid type flotation slurry might exhibit high separation performance. The flotation kinetic for the fast floating fractions was hardly influenced by the D value, and was decided by $k_f = 0.00084D + 0.3911$. However, the flotation kinetic for the slow floating fraction was quite dependent of the D value, and was decided by $k_s = -0.0092D+0.72593$.

Keywords: flotation, rheology, flotation kinetics, yield stress, degree of deviation from Newtonian fluid

1. Introduction

Rheology is a science about the deformation and flow of fluid materials under applied shearing forces (Tadros, 2010). Mineral flotation slurry is a kind of typical and complicated non-Newtonian fluid material with solid, liquid and gas under dynamic shearing fields (Wang and Li, 2020). To most kinds of flotation slurry, rheological parameters such as apparent viscosity, extrapolated yield values or the flow index indicated the degree of inter-particle interaction or aggregation inside the slurry (Zeng et al., 2023). In this case, rheological behavior can be regarded as a flotation control parameter, just like pulp pH and pulp potential. To date, rheology has been used to offer a direct approach for investigating the microstructure of particulate suspensions and inter-relations between gangue and valuable minerals (Chen et al., 2022). This kind of work involved oxide minerals (Chen et al., 2019b, 2019a; Das et al., 2011; Otsuki et al., 2011; Tsujimoto et al., 2013), clays and clay minerals (Farrokhpay et al., 2016; Ndlovu et al., 2014, 2013; Zhang et al., 2015), sulphide minerals (Asamoah et al., 2019; Basnayaka et al., 2017; Chen et al., 2017; Farrokhpay et al., 2018; Liu et al., 2021, 2018), and saline minerals such as calcite and fluorite (Chen et al., 2019a, 2019b; Deng et al., 2010; Eriksson et al., 2007; Tan et al., 2013; Zhang et al., 2020).

As to scheelite ore flotation slurry, rheological behavior became more complicated due to its fine size distribution, adsorption of flotation reagents and enrichment of certain minerals such as phyllosilicate, tectosilicate and carbonate. Fine particles of scheelite as well as saline minerals were beneficiated and induced negative effect on pulp rheology, resulting in high apparent viscosity and low flotation rate in cleaning stages (Jeldres et al., 2019; Li et al., 2020). In addition, with adsorption of collectors on their particle surface, these fine particles may also form groups or net-work structures in slurry and weaken the reagent-particle and bubble-particle contact, which result in high gangue recovery and poor

DOI: 10.37190/ppmp/201418

selectivity (Farrokhpay, 2012; Somasundaran et al., 2012). This detrimental effect has been quantized in flotation of sulphide nickel-copper ores and chalcopyrite by studying the interactions between particles and the changes in cumulative recovery of valuables and flotation kinetics (Cruz et al., 2015, 2013; Forbes et al., 2014; Wang et al., 2016).

The objective of this study was to reveal the correlation between the pulp rheology and the flotation performance of a scheelite ore from the flotation kinetic perspective. The pulp rheology was manipulated by pulp concentration, additing surface inert gangue minerals-andradite with different size and content. The flotation kinetic was analyzed by fiiting the batch flotation test results of the cumulative concentrate grade/recovery as a function of the cumulative flotation time, and the correlation between the flotation index and the rheological properties was analyzed. Further, the correlation between the degree of deviation from the Newtonian fluid and the flotation kinetics for the fast (k_f) and slow (k_s) fractions were fitted, and their mathematic relation was given, on the basis of which the quantitative affecting mechanism of the pulp rheology was discussed.

2. Methods

2.1. Materials

2.1.1. Flotation feed

The flotation feed of this study was a scheelite rough concentrate obtained from Rucheng Tungsten Mining concentration in Hunan province, China. It was obtained by adopting a closed circuit of roughing with a flotation flow-sheet of single-stage roughing, two-stage scavenging and two-stage cleaning. The size distribution of the rough concentrate was estimated by means of light scattering, using a Mastersizer 2000 (manufactured by Malvern, United Kingdom), to obtain a P90 of 39.48μm and a P50 of 18.43μm. The mineral compositions of the flotation feed were obtained by using quantitative MLA analysis, as shown in the Table 1.

Table 1. The mineral composition of the flotation feed of this study (wt.%)

mineral	scheelite	calcite	fluorite	muscovite	kaolinite	andradite	other
content	5.10	78.23	7.34	3.56	3.25	1.49	1.03

2.1.2. Andradite powder

The andradite used in this study was obtained from Rucheng in Hunan province, China. The big chunks of the pure andradite were hand-picked and crushed to -2 mm with a laboratory jaw crusher and a laboratory roll crusher, then the crushed products were concentrated with a high gradient magnetic separator several times to remove the non-magnetic minerals. The magnetic fractions were ground in a porcelain mill and then wet-sieved to different size composition including -40 μm, +40~-75 μm, +75~-100 μm, +100~-150 μm. The purity of the andradite samples was 96.78%, as determined by the quantitative XRD analysis. In this study, the andradite sample was blended into the flotation feed during the conditioning operation.

2.2. Reagents

The sodium soap 733 ($C_{12\text{-}16}COONa$, commercial grade) was purchased from Zhuzhou Flotation Reagent Group. Co. Ltd, Hunan, China, and was used as the scheelite colletor. Water glass (modulus at 2.4, i.e., chemical composition of $Na_2O \cdot 2.4SiO_2$) was used as the depressant.

2.3. Flotation tests

The flotation test on the scheelite ore was conducted with an XFD-1.5 flotation cell (self-aeration). The volume of the cell was 1.5L, and the agitation rate was fixed at 2100 rpm. The flotation conditioning operation started as soon as the flotation feed was transferred into the flotation cell. Different amounts and size composition of andradite (if needed) was added as the agitation began. Then the collector and depressant were added sequentially at designed dosage. The reaction time for each reagent was 5 min.

The flotation started with the injection of air in the flotation cell and the air flow rate was kept at 0.08m^3/h which was monitored with a flow meter. The froth products-concentrates were collected after 30s of the air injection and were taken after 2, 4, 6, 8 and 10 min, as illustrated in the Fig. 1. Finally, each concentrate was filtered and dried for weighting, magnetic separation for the added andradite (if needed) and for W assay. The concentrate grade was presented by the scheelite ($CaWO_4$) content in the froth products, and was determined by the W assay results. Since the addition ratio maintained only the same quality of the andradite, the recovery was calculated on the basis of the scheelite distribution between the concentrate and tailing.

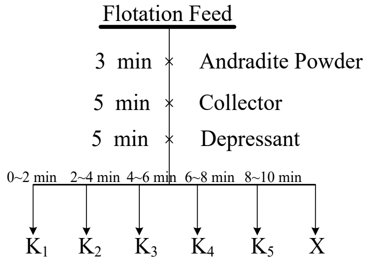

Fig. 1. The flotation flowsheet of the scheelite ore in this study

The batch flotation results were further analyzed by the flotation time-cumulative concentrate grade or recovery, cumulative recovery-cumulative concentrate grade and flotation rate coefficients calculation.

The first order kinetic model: $R = R_{max}\exp(-kt)$ was used to calculate the kinetic rate constant. A double exponential equation was adopted to calculate the rate constants for fast and slowly floating particles on the basis of the first order kinetic model (Chen et al., 2019a), as shown in the Eq. (1):

$$f(t) = \varphi \cdot e^{(-k_f t)} + (1-\varphi) \cdot e^{(-k_f t)} \tag{1}$$

where *f(t)* is the fraction of scheelite remaining in the cell after time *t*, φ is the fraction of fast-floating scheelite which has a flotation coefficient of k_f, k_s is the rate coefficient of the slow-floating scheelite.

2.4. Rheology measurement

The pulp rheology was measured using an Anton Paar MCR102 rheometer with a computation program to calculate and output the measurement parameters such as shear stress and shear rate. The flotation slurry was sampled with a polypropylene injection syringe dipped into the flotation cell just before the flotation started.

After sampling, a 20 mL of the flotation pulp was transferred into a 30 mm diameter cup. A 4-blade and 28 mm diameter vane impeller probe was placed on the cup and lowered until the gap between the impeller and the bottom of cup was 1 mm. Due to low viscosity of the slurry and turbulence created by high shear rate inside the cup, the shear rate range was limited to less than 400 rpm. The measurement procedure involved following steps: (1) pre-shearing of the slurry for 60 s at 120s^{-1}, (2) stabilization of the slurry for 30s, (3) measurement of shear stress in 72s with a shear rate ranging from 40s^{-1} to 240 s^{-1} with a step of 10s. For each rheological measurement, at least three repetitive operations were done at 20 °C with an accuracy of ±1°C. All rheology data were outputted and the mean values were counted as the final results (Chen et al., 2019b).

The rheology data was recorded by the curve data of the shear rate vs. shear stress real-time on line display of the rheometer. The rheology data was further analyzed using the Herschel Buckley rheology model. It has been widely acknowledged that this model fitted the rheological curves best in explaining the flotation slurry. The model is described by the following expression:

$$\tau = \tau_{HB} + \eta_{HB} \cdot \gamma^p \tag{2}$$

where τ is the shear stress (Pa), γ is the shear rate (s^{-1}), τ_{HB} is the Hershel Buckley yield stress (Pa), η_{HB} is the flow coefficient (Pa ·s^p) and p is the Herschel Buckley flow index. The τ_{HB} term is the extrapolated yield stress of the shear rate vs. shear stress curve. The flow coefficient (η_{HB}) describes the consistency of pulp, while the HB flow index (p) indicates the deviation from Newtonian behavior. In this study, rheograms and model parameters were mainly presented to show the rheological properties and changes in fluid types under different rheology manipulating conditions.

The degree of deviation from Newtonian fluid (D) was calculated by the following Eq. (3):

$$D = \frac{p-1}{p} \times 100\% \tag{3}$$

where p is the flow index calculated by Eq(2), and D is the degree of deviation from Newtonian fluid. The D value indicates the degree of the viscoelasticity of the flotation slurry under different conditions. For pure water which is an ideal Newtonian fluid, the p is 1.

3. Results and discussion

3.1. Batch flotation test

All of the flotation tests were performed at the vicinity of pH 9.5. The effect of the pulp concentration (solid mass concentration), andradite size and andradite content on the flotation index, i.e., the cumulative recovery vs cumulative grade was presented, coupled with the flotation kinetics calculating results of k_f and k_s.

3.1.1. Effect of pulp concentration

Fig. 2 showed the batch flotation data including the cumulative flotation time vs cumulative concentrate grade and recovery under different pulp concentration.

It was noted that these curves varied much as the pulp concentration changed in the 16-31 wt.% range. Generally, the cumulative concentrate grade declined and the cumulative concentrate recovery rose as the cumulative flotation time extended. However, as the pulp concentration increased, the cumulative concentrate grade lines move downwards as shown in the Fig. 2a, while the cumulative concentrate recovery moved upwards as shown in the Fig. 2b. When the cumulative concentrate recovery was fixed, the decreased pulp concentration yielded additional 0.8% grade, indicating that the low pulp concentration was beneficial for the enrichment ratio.

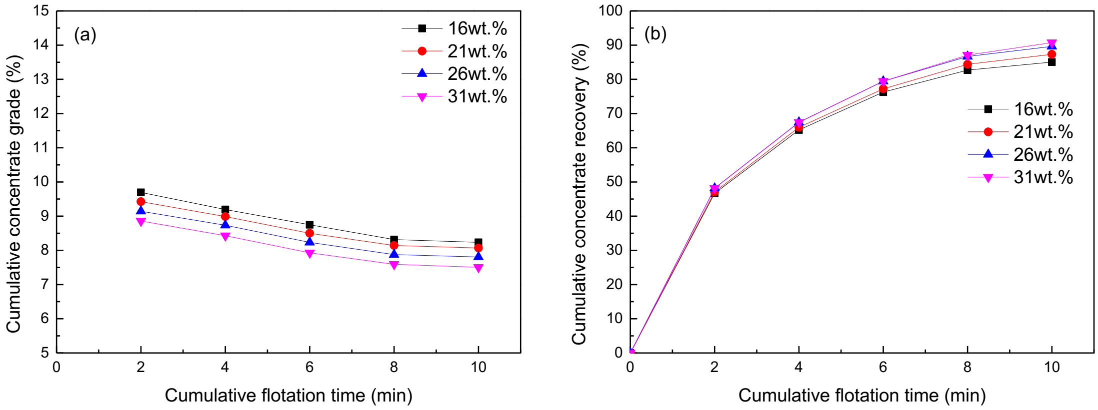

Fig. 2. Cumulative concentrate grade (a) and recovery (b) as a function of cumulative flotation time under different pulp concentration

On the basis of the cumulative concentrate grade and recovery data as a function of the flotation time, the flotation kinetics including the k_f and k_s was fitted and calculated, and was presented in Fig. 3. It was noted that as the pulp concentration increased, the flotation rate coefficient for the fast-floating fractions slightly increased while that for the slow-floating fractions nearly kept unchanged, indicating that the high pulp concentration promoted the floating of fast fractions but dragged that for the slow fractions, and further showing that the higher pulp concentration was beneficial for the floating of the coarse particles, which was regarded as the fast floating fractions.

3.1.2. Effect of the andradite size

Fig. 4 showed the batch flotation data including the cumulative flotation time vs cumulative concentrate grade and recovery under different additional surface inert andradite particles with different size.

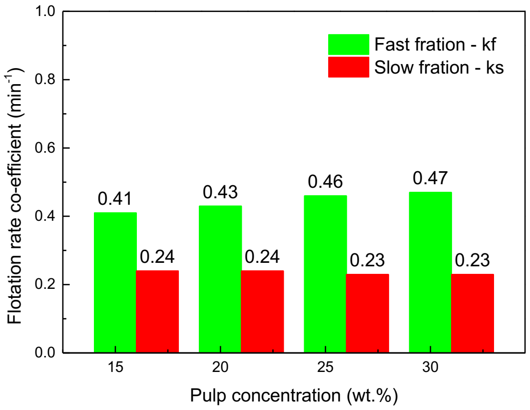

Fig. 3. Cumulative concentrate grade (a) and recovery (b) as a function of pulp concentration

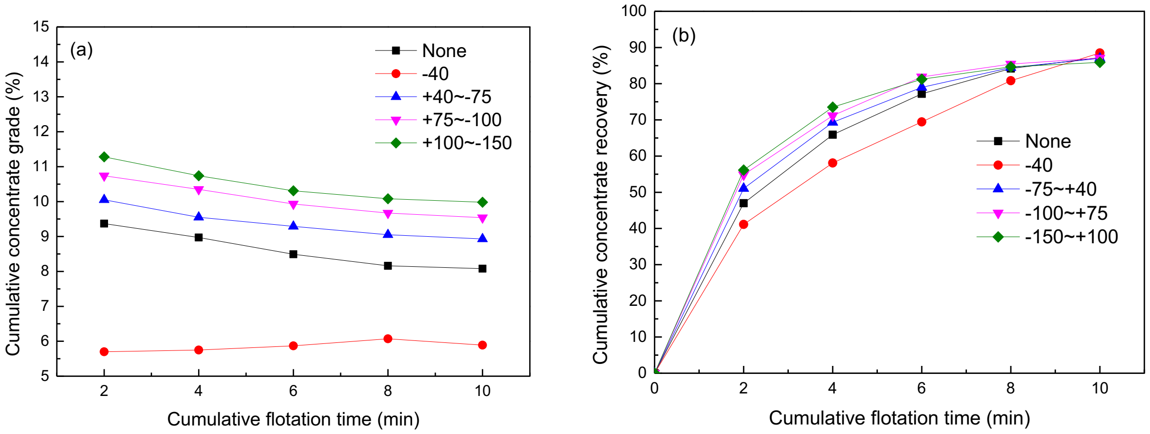

Fig. 4. Cumulative scheelite grade with time, as a function of cumulative flotation time under different size of andradite (5wt.%)

It was noted that the size of the added andradite exhibited quite different influences on the cumulative flotation time vs cumulative concentrate grade and recovery curves. The coarse andradite lifted the cumulative flotation time vs cumulative concentrate grade and recovery curves, compared with the blank contrast. When the size composition of andradite reached +100~-150 μm, a higher concentrate grade 9.98% (Fig. 4a) was obtained, compared to that of 8.08% with the baseline condition. On the contrary, flotation performance was deteriorated when blending fine fraction (-40 μm). This was consistent with the flotation result in section 3.1.1 that higher pulp concentration lead to a certain degree of decrease in cumulative grade.

Based on the remarkably changed flotation index shown in Fig. 4a and 4b, the flotation kinetics was analyzed as above, and the results were shown in Fig. 5.

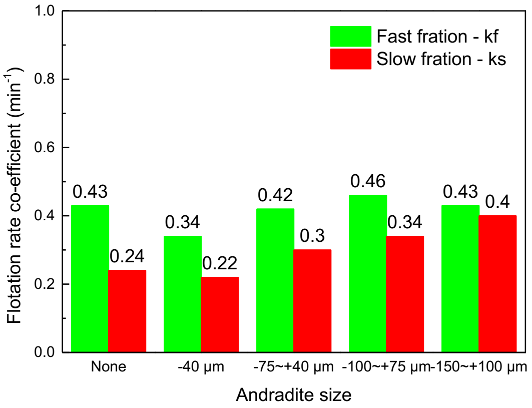

Fig. 5. Cumulative concentrate grade (a) and recovery (b) as a function of the andradite size

The results in Fig. 5 showed that the flotation rate co-efficient for the fast floating fractions basically remained unchanged in the 0.43 range except for the -40 μm situation, suggesting that the added coarse andradite particles had little influence on the floating processes on the fasting floating fractions. In addition, the fine particles, i.e., the -40 μm andradite significantly deteriorated the flotation rate for the fast floating fractions, indicating that the particle interactions or the slime coating was non-negligible for this situation. However, as the size of the andradite increased, the flotation rate co-efficient k_s witnessed considerable increase (from 0.24 min^{-1} to 0.40 min^{-1}), implying that the added coarse andradite could remarkably improve the flotation kinetic environment. In addition, a more careful observation of the fast flotation fraction also showed that the promotion effect of the andradite size was not as coarse as possible. This is the case that flotation fraction for fast-flotation scheelite tended to be invariable or even slightly declined when the andradite size increases till +100~-150 μm. Therefore, only moderate size composition of andradite could improve the flotation performance at the largest extent.

3.1.3. Effect of andradite content

Once the flotation performance of scheelite as a function of andradite size was established, batch flotation tests were performed to evaluate the positive influence of the fixed size composition (+75~-100 um) of andradite content on cumulative grade (Fig. 6a) and recovery (Fig. 6b).

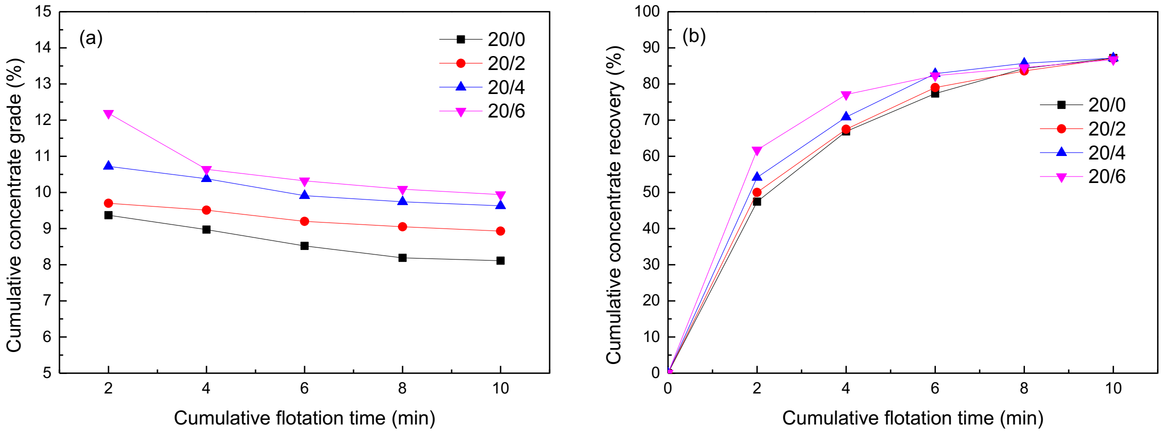

Fig. 6. Cumulative concentrate grade (a) and recovery (b) as a function of cumulative flotation time under different andradite content conditions

The flotation results in Fig. 6a showed that the increase of additional andradite in pulp moved the cumulative grade curves upwards. As andradite content in slurry reached 7%, the final cumulative scheelite grade increased from 8.11% to 9.94%, compared with the baseline balck condition. Fig. 6b exhibited the effect of the coarse andradite content on the cumulative scheelite recovery. As the content of coarse andradite in pulp increased, the partitioned recovery between 0~6min increased steadily. When 6% andradite was added, the end point of flotation arrived in advance at 6min, compared with that of blank test, which was at 8~10min, showing quite differences in the kinetics.

Thus, similar results could be seen when the cumulative recovery curves were analyzed for the flotation kinetics, as shown in Fig. 7. As more andradite was spiked with the flotation feed, the rate coefficient for slow-floating scheelite (k_s) increased stepwise, from 0.24 min^{-1} to 0.63 min^{-1}. It was worthy pointing out that too much blending coarse andradite would decrease the fast-floating fraction, as the kinetic value decreased from 0.43 min^{-1} to 0.41 min^{-1}. This conclusion revealed the similar law with the size composition experiment, namely only moderate blended andradite could effectively raise the concentrate grade with little loss in recovery.

3.2. Discussion

Among all the batch flotation tests, the added andradite was the only control variable. Therefore, the most likely explanation was that the improvement in flotation performance originated from the blended andradite particles. As the separation process was about particle-particle interaction, then the rheology property of the flotation slurry, which was the comprehensive evaluation of the inter-particle actions, must be taken into consideration (Li et al., 2018). To investigate the effect of pulp rheology on flotation performance caused by the added andradite, the flotation pulp samples corresponding to each batch flotation test before air injection were sampled and tested for the rhelogy parameters, and the yield stress, degree of deviation from Newtonian fluid (D) was presented.

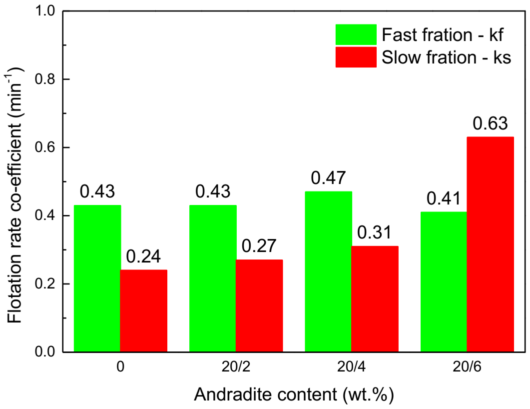

Fig. 7. Cumulative concentrate grade (a) and recovery (b) as a function of andradite content

3.2.1. Rheology behavior of flotation pulp under different concentration

Fig. 8 showed the yield stress and degree of deviation from Newtonian fluid (D) of the flotation of the pulp under different pulp concentration.

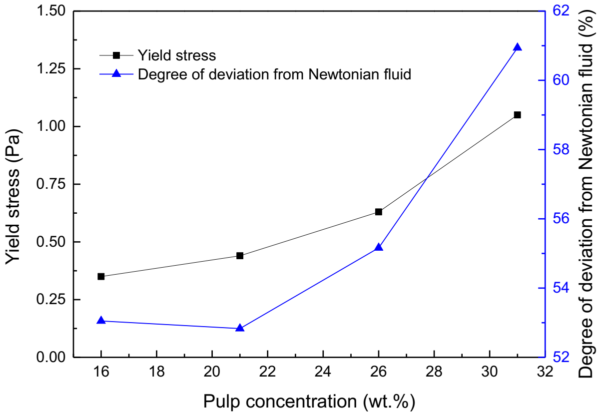

Fig. 8. Yield stress and degree of deviation from Newtonian fluid (D) of the flotation pulp, as a function of the pulp concentration

As expected in Fig. 8, the flotation pulp exhibited rheological behaviors far from the Newtonian fluid, with obvious yield stress and degree of deviation far from 1(Newtonian fluid), showing that there existed very complicated linking or aggregation among the particles. As the solid content in the flotation pulp increased from 16% to 31%, the yield stress increased and the slurry deviated more significantly from the Newtonian behavior. This was expected, as higher solid content corresponded to more interactions between mineral particles. In addition, the flotation feed of the fine dissemination ore with a P90 of 39.48μm formed easily into networks in slurry due to the agglomeration between particles or particle groups, and resulted in high yield stress and the subsequent high degree of deviation from Newtonian fluid.

3.2.2. Effect of the added andradite

Fig. 9 showed the yield stress and degree of deviation from Newtonian fluid (D) of the flotation of the pulp under different size and content of the added andradite.

It was noted that the yield stress and the D value exhibited the same changing trends as the andradite size and content changed except for the -40 μm situation. Generally, the low yield stress corresponded to the small D values, indicating that the aggregation or the formation of the net-work structures which increased the yield stress also represented the fluid type of the flotation slurry. On the whole, the flotation pulp tended to be Newtonian fluid as the coarse andradite and high content, showing that the flotation pulp was more dispersed, which showed further influence on the flotation kinetic environment, as presented in the Fig. 9a and 9b.

It was worth pointing out that the fine andradite (-40 μm) enlarged the pulp yield stress, but it decreased the D value. This was to say, blending fines into the flotation feed also help to make the flow behavior close to Newtonian fluid but failed to change the inter-particle interactions, probably due to its small momentum and size.

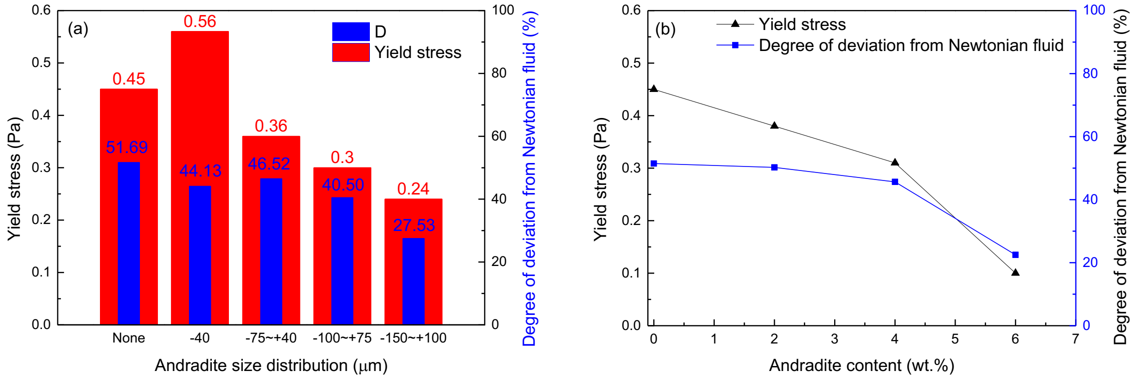

Fig. 9. Yield stress and degree of deviation from Newtonian fluid (D) of the flotation of the pulp, as a function of the size (a) and content (b) of the additional andradite

3.2.3. Correlation between the flotation index and the rheology

To further reveal the correlation between the enrichment process and the rheology, the cumulative concentrate grade and the yield under different flotation variables were counted, and the results were shown in the Figs. 10-11.

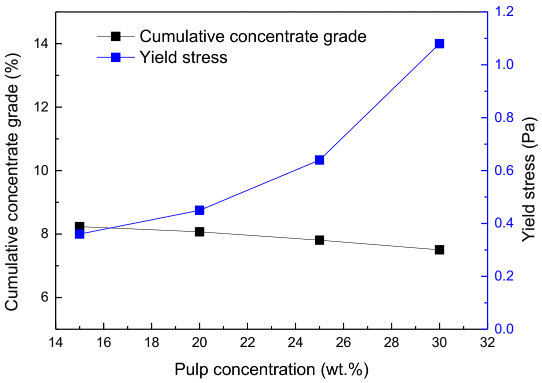

Fig. 10. The cumulative concentrate grade and the yield stress under different pulp concentration

Fig. 10 listed the cumulative grade of concentrate with the yield stress under different flotation pulp concentration. It was noted that as the yield stress increased, the cumulative concentrate grade gradually decreased, showing an obvious negative correlation. The results basically revealed that the high yield stress was not beneficial for the separation process.

Fig. 11 summarized the variation trends of the cumulative grade and yield stress under different andradite conditions. The results showed that the cumulative grade and yield stress was also in negative correlation. However, in this case, the key flotation index, the cumulative concentrate grade, was effectively improved by decreasing the yield stress using the methods of blending andradite with different size and content.

It should be also mentioned that even though the flotation slurry may not be noticeably changed in mineral composition, it was likely that subtle changes in rheological parameters of pulp could potentially affect the flotation behavior. It was to say, the small decrease in yield stress of pulp by blending coarse andradite could improve the grade of concentrate on a large extent

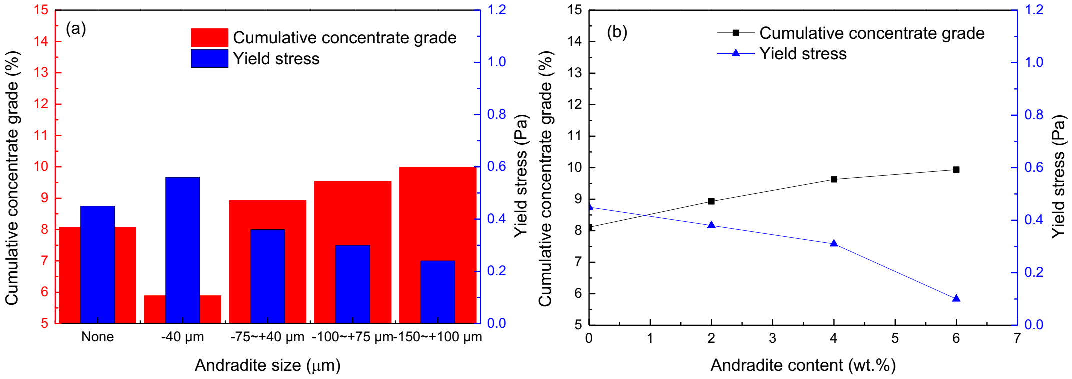

Fig. 11. The cumulative concentrate grade and the yield stress of the flotation pulp, as a function of the size (a) and content (b) of the andradite

3.2.4. Correlation between the degree of deviation from Newtonian fluid and the flotation kinetics

The flotation rate coefficient for both fast floating and slow floating fractions were selected to quantify the effect of the fluid type, i.e., the D value, on the flotation kinetic. The k_s and k_f as a function of the D values were calculated and linearly fitted, and the results were shown in Fig. 12.

It could be noted that the k_s decreased dramatically as the D value increased, showing that as the flotation slurry deviated more from the Newtonian fluid (D=0), the slow floating fractions of the scheelite faced with more difficult floating environment. The linear function between the D and the k_s was found to be: $k_s = -0.0092D+0.7259$. This was to say, as the flotation pulp was ideally dispersed as Newtonian fluid (D=0), the theoretical k_s was 0.7259 min^{-1}. However, for the fast floating fractions, the linear function between the D and the k_f was found to be: $k_f = 0.00084D+0.3911$, indicating that the D exhibited very little influence on the flotation rate. That was to say, the fast floating fractions could remain its high rate under high viscous flotation pulp.

It has been demonstrated that blending coarse andradite showed a strong effect on the flotation pulp rheology (Contreras et al., 2020; Wang and Li, 2020). Even small changes in pup rheological properties could lead to remarkable changes in the flotation behavior including the cumulative concentrate and flotation rate co-efficient. However, pulp rheology alone cannot explain all the differences seen in the test results. This meant that the reduction of entrainment and hetero-coagulation caused by the coarse andradite were also responsible, and were at least partially contributive for the improvement in flotation performance.

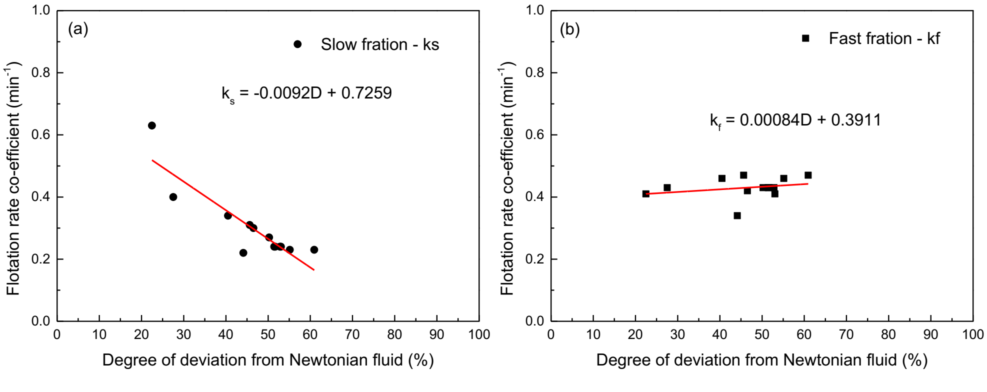

Fig. 12. Flotation rate coefficient for slow-floating (a) and fast-floating (b) fractions as a function of the D value

4. Conclusions

From all the results presented, including flotation, rheological measurements and data fitting, the following conclusions could be drawn:

1. For scheelite flotation, high pulp concentration exhibited big yield stress and typical non-Newtonian fluid. Increasing pulp concentration could improve the concentrate grade and the flotation rate co-efficient of the fast flotation fractions.
2. Adding coarse andradite (+75~-100 µm) at a mass ratio of 20/6 in the scheelite ore flotation slurry was found to increase the flotation rate co-efficient for the slow floating fractions from 0.24 min^{-1} to 0.63 min^{-1}.
3. The yield stress was in negative correlation with the cumulative concentrate grade for the scheelite flotation spiked with andradite. Weakening the yield stress, i.e., eliminating the aggregations in the slurry was beneficial for the enrichment ratio.
4. The flotation kinetic for the fast floating fractions was mainly independent of the degree of deviation from Newtonian fluid, and was determined by by kf = 0.00084D + 0.3911; However, the flotation kinetic for the slow floating fraction was quite dependent of the degree of deviation from Newtonian fluid, i.e., the flotation rate co-efficient ks was determined by ks = -0.0092D+0.72593. This might be ascribed to the transportation of these fractions under network structures with high yield stress.

Acknowledgements

The authors acknowledge the support of the Scientific Development Basic Research Foundation Project of the National Sintering and Pelletizing Equipment System Engineering Research Center, China (No. 2024JCYJ09) and the State Key Laboratory of Advanced Metallurgy for Non-ferrous Metals (No. YSQH-ZD-24012).

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