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Optimization of effective parameters in flotation of fluorite from lowgrade ore without frother

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Abstract: This study investigates the flotation behavior of low-grade fluorite ore from the Tuye Darvar Damghan mine. Characterization techniques, including XRD, optical microscopy, and SEM-EDX, were employed to analyze the mineralogical and textural properties of the ore. Flotation experiments were conducted using a Denver cell to evaluate the effect of various parameters, including particle size, solid percent, collector concentration (sodium oleate), depressant concentration (sodium silicate), and pH, on the recovery and grade of fluorite concentrate. The results demonstrate that optimal flotation conditions were achieved at a pH of 8, with a particle size of 75 μ m, a solid percent of 20%, a collector concentration of 600 g/t, and a depressant concentrate with a grade of 91.5% and a recovery of 16.75% was obtained. This study provides valuable insights into the flotation behavior of low-grade fluorite and can serve as a guide for optimizing industrial-scale flotation processes.

Keywords: fluorite, flotation, sodium oleate, sodium silicate, recovery

1. Introduction

Fluorite, with the chemical formula of CaF₂ mostly consists of calcium (Ca) and fluorine (F), with additional elements playing a minor role in its composition. The crystal has a cubic structure with a unit cell parameter of 0.5463 nm for all three dimensions (a, b, and c) and an angle of 90 degrees between them (α , β , and γ) (Klein and Dutrow, 2007). Each calcium atom is surrounded by fluorine atoms at a distance of 0.237 nm, and from another perspective, each fluorine atom is surrounded by four calcium atoms, resulting in the formation of an ideal tetrahedral shape, and with the merging of two tetrahedral forms results in the creation of the cubic crystal system with a body-centered structure in each fluorite single unit cell. The Ca-F bond type is classified as a strong ionic bond due to its dominance in the fluorite structure. This has resulted in 90% of the chemical bonds in fluorite being of the ionic type.

High fluorine content of the fluorite mineral makes it the main source of the fluorine element in the production of hydrofluoric acid. This mineral has found extensive applications in chemical engineering, construction materials, aircraft, cement, glass, ceramics, aluminum, and steel industries (Zhu et al., 2018; Bide et al., 2021; Gao et al., 2021).

Fluorite may be readily differentiated from other crystals with similar shape and colour based on its visual characteristics, hardness, and density. This material exhibits perfect cleavage in four directions, resulting in the formation of tiny octahedral pieces upon breakage. Due to its high density of 3.2 g/cm³, fluorite poses a challenge in terms of separating it from other associated gangue minerals. Fluorite mineral occurs naturally in a variety of colours, such as yellow, blue, purple, green, white, and black. Researchers found that impurities such as rare earth elements affect the colour of this mineral; for example, fluorite containing yttrium and cerium is usually green in nature (Sasmaz et al., 2018). The results indicated that the floatability of fluorite minerals varies depending on their respective colors. The floatability of colorless fluorite is better than green fluorite and green fluorite, is better than purple one, because there is a difference in the roughness of their surface (Zheng et al., 2017).

Fluorite exhibits chemical inertness and demonstrates a marked resistance to reaction with a majority of acidic substances or common chemicals. Fluorite, along with calcite, apatite, and francolite, is classified as a semi-soluble mineral. The equilibrium constant for the dissolution of fluorite in water at room temperature is 5.25×10^{-9} . The solubility of this mineral varies across its surfaces, perhaps due to the distinct crystal structure present on each side (Gao et al., 2014). The solubility levels of this material are presented in Table 1.

Table 1, the dissolution rate of diffrent fluorite surface(Godinho, Piazolo et al. 2012)

Fluorite surface	{111}	{100}	{110}	{310}
Dissolution rate	1.2±0.8	3.2±0.2	28±6	32 <u>±</u> 6

According to Table 1, it can be observed that the rate at which the {111} and {100} surfaces dissolve is lower compared to the {110} and {310} surfaces. Therefore, it can be inferred that the density of ions on the former surfaces is lower than that on the latter surfaces. Zhang et al. found that the surface of 111 exhibits a moderate level of hydrophobicity, with an average contact angle of 20 degrees (Zhang et al., 2015). Surfaces of 110 and 100 exhibit a high degree of hydrophilicity, resulting in a contact angle of zero. The studies conducted by Mielczarski and Zheng, along with their mathematical models, demonstrate that the {111} surface of fluorite is the most suitable surface for froth flotation of fluorite, specifically in terms of the adsorption of sodium oleate (Mielczarski et al., 2002; Zheng et al., 2018). Research has elucidated that the {111} surface emerges as the prevalent cleavage plane in 60% of occurrences subsequent to fragmentation, wherein fluorite demonstrates an intermediate level of hydrophobicity (Li and Gao, 2018).

Flotation is indeed the most common method to obtain concentrate through the selective adsorption of collectors on target minerals to make them hydrophobic and floatable. This process involves the use of collectors that selectively bind to the surface of target minerals, imparting hydrophobicity to those mineral particles, which is a necessary condition for air bubble attachment and subsequent flotation to the surface of the mineral slurry (Tao et al., 2024).

The zeta potential analysis performed by Lin et al. has demonstrated that fluorite possesses a superior flotation efficacy relative to calcite. This enhanced flotation capability is ascribed to the preferential chemical adsorption of sodium oleate and oleic acid on the surface of fluorite, which is more pronounced than on calcite (Lin et al., 2016). The reagents employed in the flotation of fluorite are differentiated into four distinct categories contingent upon the intrinsic nature and compositional characteristics of the polar group: anionic, cationic, amphoteric, and biogenic collectors (Gao et al., 2021). Among the various classes of collectors utilized in the flotation of fluorite, anionic collectors, particularly fatty acids, exhibit the most pronounced efficiency. The use of cationic and amphoteric collectors complicates the operation, and the smallest change in the system causes instability in the process and eventually recovery and grade of the final product. Microbial collectors also have little flexibility and efficiency on an industrial scale.

Gao et al. successfully separated fluorite from calcite by using a collector of 30 mg/L propyl gallate, at a pH of 9.5, with MIBC as a frother and without the use of any depressant. They found out that the amount and type of foaming agent have an effect on the flotation of fluorite. The presence of terpinol, which acts as a frother, reduces the selectivity of flotation by enhancing the capacity of calcite to floatability (Gao et al., 2019b).

The research conducted by Zhou et al. has elucidated that the application of acidified sodium silicate exerts a selective and efficacious influence on the suppression of calcite in the flotation of calcite-fluorite systems utilizing oleic acid as the collecting agent. Their findings indicate that sodium silicate not only impedes the flotation of calcite but concurrently serves as an activator for fluorite (Zhou and Lu, 1992).

Zhang et al. successfully achieved a concentrate with 98% fluorite at a low temperature by employing 600 g/t of sodium naphthenate (commercially known as GY-2) as a novel collector and 600 g/t of copper sulphate salt as a phosphate mineral inhibitor. The initial feed consisted of 60% fluorite and 32% silicate (Zhang and Song, 2003).

Gohar et al. achieved a recovery rate of 99.9% for fluorite with a grade of 98.7% from an ore containing 36.4% fluorite and 38% calcite. This was accomplished by using 100 g/t of oleic acid as a collector, 25 g/t of sodium dodecyl sulphate (SDS) as a frother, 200 g/t of sodium silicate, and 40 g/t of starch as a depressant (Gohar et al., 2002).

Column flotation, employed by Bhaskar et al., achieved a concentrate with higher grade and recovery compared to mechanical cells, while using fewer cleaning stages (Bhaskar Raju and Prabhakar, 2000).

Gao et al. discovered that the sequence of modifying chemical treatment has an impact on the flotation process. It was discovered that the addition of a collector before a depressant makes a reduction of calcite more difficult. According to their research, the addition of acid to the slurry decreases the amount of calcite in the concentrate. By employing this method, calcite was successfully isolated from fluorite, resulting in a recovery rate of 83% and a grade of 90.7%. The ore initially included 42% fluorite and 10% calcite. The optimal conditions for this technique included using 15 mg/l sodium oleate as a collector, maintaining a pH of 7, and adding 5 mg/l tannic acid as a depressant (Gao et al., 2019b).

Fatima et al. successfully employed a novel technique, devoid of mineral inhibitors, to achieve a significant reduction of 67% in carbonate and 75% in silica content. This was accomplished by treating the slurry feed to boiling prior to the flotation process. The process involved the use of 0.4 g/L of sodium silicate, 0.075 g/L of quebracho, 0.08 g/L of guar gum, 0.62 ml/L of oleic acid, and 0.25 g/L of palmitic acid (Fatima et al., 2022).

Regardless of the type of collector or flotation without a collector, the best pH for it has been recorded as 8 to 10. Empirical studies have indicated that the predominant pH level observed during the industrial and laboratory flotation processes of this mineral is approximately 9. The zero point charge (zpc) of fluorite depends on its origin and its occurrence; it varies from 6 to 10, and an optimal pH for its flotation should be found in each experiment. This study assessed the flotation of low-grade fluorite, characterized by a complex distribution of fluorite within the gangue matrix. In this study the flotation behaviour of low-grade fluorite ore from the Tuye Darvar Damghan mine was investigated. Characterization techniques, including XRD, optical microscopy, and SEM-EDX, were employed to analyze the mineralogical and textural properties of the ore. The key parameters, including solid percentage, particle size, collector concentration, depressant concentration, and pH, were evaluated in the absence of a frother. The study aimed to determine the feasibility of recovering a high-grade concentrate.

2. Materials and methods

2.1. Ore sample

A sample weighing 300 kg was procured from the Tuye Darvar Damghan mine. The sample was subjected to standard preparation techniques, which included crushing by jaw, cone, and roll crushers. This process effectively reduced the particle size to less than 2mm. The distribution of the crushed product is depicted in Fig. 1-a. The degree of freedom can be measured using polished sections and an electronic microscope for each fraction (Bissombolo and Mori, 1995). Data obtained from sample analyses revealed two-dimensional fractions with the highest degree of liberation. Subsequently, grinding operations were conducted using a laboratory ball mill. Fig. 1-b presents a dimensional analysis diagram detailing the grinding steps undertaken to achieve two-dimensional fractions of 75 and 37 microns.

2.2. Materials

The following reagents were used: Sodium oleate (from sigma aldrich), palmitic acid (from sigma aldrich), sodium silicate (from Merck) and MIBC (from Merck). To adjust the pH during flotation, analytical grade sulfuric acid and sodium hydroxide (from Merck) were employed. Tap water was used as the medium for all separation processes.

2.3. Methods

In this study, X-ray fluorescence (XRF) (Philips X Unique II) spectrometry was utilized to determine the chemical composition of the sample. This method is effective for quantifying both major and minor

elements within the sample. Additionally, the study employed X-ray diffraction (XRD) (X'Pert MPD, Philips, Holland) to analyze the mineralogical composition of the ore. This technique helped in identifying the various phases present in the fluorite and gangue minerals. The characterization of valuable and gangue minerals was further enhanced through the use of a scanning electron microscope (SEM) (Philips XL 30) coupled with wavelength dispersive X-ray (WDX) spectroscopy. This combination allowed for the detailed identification and analysis of the minerals present in the sample.



Fig. 1. Cumulative distributions of particle passed from sieves. a) crushing steps, b) milling with ball mill to reach the two liberation size

2.4. Flotation experiments

The flotation tests (Fig. 2) conducted in a 2-L Denver flotation cell were performed. The solid percent was certain amount, which is a suitable concentration for flotation processes and the pH was adjusted using sodium hydroxide. The conditioning time for all reagents used in the tests, including collectors, and dispersants, was set to 3 minutes, which is sufficient for the reagents to interact with the mineral particles and prepare them for flotation. The air flow rate was kept at a constant 3 L/min, providing a steady supply of air bubbles necessary for the flotation process. The froth collection time for each test has been considered as 8 minutes, based on the maximum froth production.

The results of the flotation tests included measurements of weight recovery and fluorite recovery for both the concentrate and tailings. The recovery (R) of the product in percent was calculated using Equation (1):

$$R = \frac{Cc}{Ff}$$
(1)

where, 'C' represents the weight of the product (kg), 'c' is the grade of the product (%), 'F' is the weight of the feed (kg), and 'f' is the grade of the feed (%) (Sobouti et al., 2020).

3. Result and discussion

3.1. Optical microscope analysis

An analysis was conducted using an optical microscope on the thin sections derived from the specified dimensional fractions. The mineral bedrock under investigation is an altered igneous rock. Observed alterations include sericite, argillic, carbonate, and chlorite. The alteration intensity ranges from moderate to severe, with a majority of the constituent minerals exhibiting alterations. The optical microscope analysis results demonstrate that a significant proportion of fluorite minerals, particularly those with dimensions less than 75 microns, achieve a satisfactory degree of liberation. Fig. 3 presents four selected images captured in parallel and crossed polar modes from the optical microscope. These images reveal that the fluorite and gangue minerals are intermingled within each other's matrices. The analysis indicates that achieving a concentrate of the highest purity would result in a lower recovery rate. The gangue minerals were mostly quartz and calcite.



Fig. 2. Flotation test for the beneficiation of low-grade fluorite



Fig. 3. Optical microscope analysis in the a,c) cross polar, b,d) paralell polar mode

3.2. SEM analysis

Studies using a SEM equipped with a WDX analyser (3PC Micro spec CO.) and preparation of X-Ray Mapping has been done to identify the morphology of the sample. Back scattered electron (BSE) detector

was used in the preparation of electron microscope images. In the images prepared with this detector, heavy minerals can be seen lighter than the others. By decreasing the average mass number of a mineral, its brightness in the image decreases. An example of these images is presented in Fig. 4. In this form, the light-grey particles are fluorite minerals and the dark-grey particles are gangue minerals such as quartz and calcite.





There are two types of fluorite in this sample in terms of texture and type of involvement. Part of them is fluorite that are released in large particle size (fraction size of +200 microns). However, these dimensions are also not suitable for gravity enrichment on an industrial scale unless it is possible to prepare a primary concentrate as flotation feed by removing part of the gangue minerals. Another type of fluorite is strongly involved with gangue minerals, which requires further crushing. This type of conflict can be seen in two ways: the distribution of fluorine inside the gangue minerals and, conversely, the distribution of gangue minerals of very small sizes inside the fluorite. These types of conflicts are also present in smaller size fractions, which seems to be more like the type of conflict that is the distribution of gangue minerals inside the fluorine as the dimensions become smaller.

3.3. XRD and chemical analysis

In order to determine the grades and type of minerals in the sample, chemical analysis has been done for 5 representative samples, which are presented in Table 2. The results show that the main gangue is

quartz and this deposit is of quartz-fluorite type. The XRD analysis (Fig. 5) showed that the sample mainly contains quartz (SiO₂), fluorite (CaF₂), hematite (Fe₂O₃) and calcite (CaCO₃).



Fig. 5. XRD pattern of sample

Sample	CaF ₂ (%)	CaCO ₃ (%)	SiO ₂ (%)	L.O.I(%)
1	13.57	0.6	63.12	2.77
2	12.8	0.5	70	2.68
3	16.77	1.5	58.68	4.17
4	17.47	1.6	52.64	4.65
5	18.17	1.7	52.26	4.82
Avg	16.32	1.18	59.4	3.8

Table 2. Chemical analysis report

4. Results and discussions

Flotation tests on the ore samples were conducted using a 2-L Denver cell. In this study, for low-grade fluorite ores, the collector chosen is critical in the high-grade recovery process of the flotation in the absence of any frothers.

4.1. Solid percent

In the process of fluorite flotation, the pulp solid percentage emerges as a significant parameter influencing both the grade and recovery. Fig. 6 shows the effect of pulp solids percent on grade and fluorine recovery. As depicted in Fig. 6, an examination of the recovery and grade demonstrates a clear correlation with the pulp solids percentage. The data indicates that an increase in the solid percentage coincides with an increase in recovery. However, this increment in solid percentage concurrently leads to a decrease in the concentrate grade, a phenomenon attributable to particle tailing. Lower solids percentage results in better drainage (Safari et al., 2022); therefore, given the fluorine concentrate grade, the optimal solid percent for continuous flotation tests is 20%.



Fig. 6. The effect of solid percent on the recovery and grade of fluorite (pH=8, collector concentration=300 g/t, depressant concentration=100 g/t)

4.2. Particle size

To investigate the effect of particle size, tests were conducted under identical conditions at three pHs: 8, 10, and 12, with two particle sizes of d_{80} =75 and d_{80} =37 microns. Fig. 7 (a and b) displays the grade and recovery results. Due to the fact that the most of the fluorite mineral in the ore is released below 75 microns, the flotation process was carried out in two dimensions of d_{80} , 75 and 37 microns. According to Fig. 7 (a and b), when the particle size goes from d_{80} =75 microns to d_{80} =37 microns, the grade of fluorine in the concentrate goes down at all three pH levels (8, 10, and 12). Also, as the particle size decreased from d_{80} =75 to d_{80} =37 microns, fluorine recovery also decreased. However, the grade of fluorine in the concentrate went down less at pH = 12 than at pH 8 or 10. While the highest reduction of fluorine recovery with decreasing particle size decreases. The phenomena that has been observed is thought to be caused by the reduced ability of fine-grained particles to float and select when subjected to processing in a conventional mechanical flotation cell (Liu et al., 2021).



Fig. 7. The effect of particle size on the recovery and grade of fluorite (solid percent=20%, collector concentration 300 g/t, depressant concentration=100 g/t)

4.3. Collector concentration

Sodium oleate, with its inherent foaming characteristics, served as a fatty acid collector in the conducted experiments. The foaming nature of sodium oleate is beneficial for flotation, as it helps stabilize the froth, allowing for better separation. Sodium oleate is used in the flotation separation of quartz-fluorite and calcite-fluorite due to its unique advantages, such as low cost, ready availability, and strong collection capabilities, effective in various conditions. Fig. 8 shows the effect of collector concentration on fluorine grade and recovery. As collector concentration increases from 300 to 800 g/t, the fluorine grade diminishes across all three levels of pH (8, 10, and 12), with a more pronounced drop observed at pH 8 compared to pH 10 and 12. On the other hand, as collector concentration increases, fluorine recovery increases, with the largest increase occurring at pH 8. This suggests that higher concentrations

of the collector enhance the attachment of desired minerals to air bubbles, leading to more efficient flotation. In other words, at concentrations of 300 g/t to 600 g/t, sodium oleate enhances the flotation recovery of fluorite by adsorbing onto its surface, so rendering it hydrophobic. This allows the fluorite particles to adhere to air bubbles and ascend to the surface, where they may be removed. Excessive collector concentration can lead to an inefficient flotation process due to over-saturation of the mineral surface or competition with other minerals. The optimal recovery was noted at a pH of 8. pH can significantly influence the ionization and solubility of collectors, as well as the surface charge of the minerals being processed. A pH of 8 may favor the effectiveness of sodium oleate in collecting hydrophobic minerals (Zheng et al., 2017).



Fig. 8. The effect of collector concentration on the recovery and grade of fluorite (solid percent 20%, d₈₀ particle size 75 μm, depressant concentration=100 g/t)

4.4. Sodium silicate concentration

The most common inorganic depressant is sodium silicate, which is a series of different compounds made up of different proportions of SiO_2 and Na_2O . It has excellent suppression for calcite and silicate gangue minerals. The suppression effect of sodium silicate on fluorite decreases when the amount of sodium silicate is substantial due to the changing proportion of SiO_2 and Na_2O . This phenomenon is attributed to the fact that the efficiency of sodium silicate as a depressant is influenced by its SiO_2 molar ratio. When the amount of sodium silicate is increased significantly, the molar ratio of SiO_2 to Na_2O can change, leading to a reduction in its effectiveness as a depressant for fluorite.

Fig. 9 illustrates that when the concentration of sodium silicate increases from 100 to 500 g/t, the fluorine grade decreases. This increase in sodium silicate concentration causes an increase in the depression of gangue minerals, such as calcite. Some gangue particles can get a layer of sodium silicate on their surface. This makes them hydrophilic, which means they like water. This makes them less likely to stick to air bubbles during flotation. On the other hand, as sodium silicate concentration increases, fluorine recovery also increases. This suggests that higher concentrations of the collector enhance the attachment of desired minerals to air bubbles, leading to more efficient flotation (Gao et al., 2021). The optimal concentrate of sodium silicate is 100 g/t.



Fig. 9. The effect of depresant concentration on the recovery and grade of fluorite (solid percent=20%, d₈₀ particle size 75 μm, collector concentration=600 g/t)

4.5. pH

In this process, the pH level plays a crucial role as it alters the surface charge and chemical conditions of the minerals and chemicals used, thereby enhancing the interaction between the collector and the mineral surface. At different pH levels, the surface charge of fluorite can change, making it more or less susceptible to flotation. Given that fatty acids function as anionic collectors, it is imperative for the target mineral surface to possess a positive charge to enable binding. The point of zero charge (PZC) of the fluorite mineral, owing to the presence of structural impurities, differs from other minerals and spans a broad pH range (Gao et al., 2021). Consequently, a comparative analysis was conducted with three distinct flotation pH values. Fig. 10 shows the effect of pH on the grade and recovery of fluorine flotation. The findings, as illustrated in Fig. 10, indicate that the optimal pH lies within the range of 8 to 10. The sodium oleate collector can selectively separate fluorite from other gangue minerals within the pH range of 8 to 10. This can maintain a higher grade of the fluorite concentrate.



Fig. 10. The effect of pH solution on the recovery and grade of fluorite (solid percent=20%, collector concentration=600 g/t, d₈₀ particle size 75 μm, depresant concentration=100 g/t)

4.6. Optimum test

Taking into account the outcomes derived from the conducted experiments, which were characterized by pH=8, particle size (d_{80}) = 75 µm, solid percentage=20%, collector concentration = 600 g/t and depressant concentration = 100 g/t, the design for the subsequent experiment can be conceptualized as depicted in Fig. 11. Table 3 shows the chemical analysis of the concentrate, tailings, middle and tailings of cleaning stages. Under optimum condition in the rougher stage, the grade and recovery of fluorite was obtained 55.2% and 45%, respectively. To improve the grade of fluorite in the process, the concentrate of rougher stage was processed for seven cleaner stages, as shown in Fig. 11. The obtained analysis for the final concentrate (after seven cleaner stages) revealed that the grade of fluorite was 91.5%. According to the values of fluorite and silica grades, the final recoveries of mentioned minerals in the flowsheet were calculated as 16.75% and 0.8%, respectively. It is noted that the tailing of the rougher stage was concentrated under scavenger stage to recover the fluorite remaining in the tail. The results indicated that the grade and recovery of fluorite in the concentrate of scavenger stage were 2.08% and 10%, respectively. Finally, the fluorite concentrate grade in the final product was obtained 91.5%, and its recovery against the initial feed was calculated to be 16.75%.

Sample	CaF2 (%)	CaCO3 (%)	SiO2 (%)	L.O.I (%)	Fluorite recovery (%)	Silica recovery (%)
Feed	17	1.6	66	5.56	100	100
Final tail	1.8	1.1	67.67	2.48	35.9	86.3
Middle	2.08	1.8	59.6	6.24	10	6
Concentrate	91.5	1	5.2	2.3	16.75	0.8
Tail of concentrate	2.19	1.68	57.66	6.8	37.35	6.9

Table 2. chemical analysis of the final completion test



Fig. 11. the flowsheet of the final completion test under optimum condition

5. Conclusions

This study comprehensively investigated the flotation behaviour of a low-grade fluorite ore from the Tuye Darvar Damghan mine. Characterization techniques, including XRD, SEM-EDX, and optical microscopy, revealed the mineralogical composition and textural characteristics of the ore, highlighting the presence of significant quartz and calcite as gangue minerals. A series of flotation experiments were conducted in a Denver cell to determine the optimal conditions for fluorite recovery. The effects of key parameters, including particle size, solid percent, sodium oleate concentration as a collector, sodium silicate concentration as a depressant, and pH, were systematically evaluated. Upon deriving the optimal parameters, a supplementary batch test was designed. The implementation of the designed flowsheet enabled the recovery of 16.75% of fluorite, exhibiting a grade of 91.5%, from an ore characterized by an average grade of 17%.

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