COMPARATIVE ANALYSIS OF FLOTATION EFFICIENCIES BETWEEN MICROFLOTATION CELL AND BATCH FLOTATION CELL TESTS

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MİKROFLOTASYON VE FLOTASYON HÜCRESİ İLE YAPILAN TESTLERİN FLOTASYON VERİMLİLİKLERİNİN KARŞILAŞTIRMALI ANALİZİ

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1. Introduction

Froth flotation is a widely used metallurgical separation method used for mineral processing, wastewater treatment and plastic recycling (Takoungsakdakun and Pongstabodee, 2007., Wang, Wang, H., Fu and Liu, 2015, Negari, Ostad Movahed and Ahmadpour, 2018). Flotation is essentially a separation technique relies on the wettability of minerals. It is a physical-chemical process based on utilizing the gas bubbles to selectively adhere to particle surfaces in a solid/water pulp. The

particles attached to bubbles are separated from the pulp depending on its hydrophobic or hydrophilic properties. Chemicals are used to alter the surface hydrophobicity of particles, thereby increasing the effectiveness of bubble collection. The fact that mineral surface properties can be changed with the use of chemicals enables the flotation process to be used in the wide range of mineral separation. This method is especially useful for treating fine grained ores that cannot be effectively processed using traditional gravity concentration techniques.

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Separation takes place in a flotation cell (Figure 1) in which a slurry consisting of particles and water is added and air is continuously introduced into the system. The bubble capture process consists of three steps. The initial phase involves collision, where a particle encounters a bubble. Subsequently, during the attachment stage, the particle might adhere to the bubble, rebound into the pulp, or slide off the bubble's surface. In the final detachment stage, external forces can cause adhered particles to separate from the bubble. Ultimately, only particles successfully attached to the bubble will be gathered in the froth phase and recovered as the floated product. Hydrophillic particles settle within the pulp and generate the non-floated product (tailings). In flotation columns, gas bubbles with diameters typically around 0.5 mm are generated. These bubbles selectively adhere to hydrophobic particles, transporting them to the liquid surface, creating a froth zone for separation. Meanwhile, hydrophilic particles are discharged from the bottom outlet.

Figure 1. Flotation Cell

In conventional flotation cells, the establishment of three distinct hydrodynamic regions: turbulent, quiescent, and froth zones are fundamental to ensuring efficient flotation processes (Savassi, 2005). Figure 1 illustrates the presence of these three distinct hydrodynamic regions within a conventional flotation cell. The turbulent zone nearest to the impeller exhibits the highest turbulence intensity, facilitating the dispersion of injected gas into bubbles that interact with suspended solids. The turbulent zone influences the suspension of solids, the dispersion of gas, and the interactions between bubbles and particles. A quiescent zone situated in the central layer of the cell promotes the ascent of bubble-particle aggregates into reduced turbulence zone which aiding in their stabilization and reducing the gangue mineral entrainment. At top of the cell, the froth zone acts as an additional cleaning stage, facilitating drainage and further enhancing concentrate grade (Gorain, Franzidis and Manlapig, 2000).

The challenge of flotation processing fine (15-5 μm) and ultrafine (<5 μm) mineral particles remains a significant technical obstacle in mineral processing. Losses

resulted from the inherent characteristics of these particles, such as their limited mass, high interfacial free energy, and primary flotation efficiencies, which contribute to their low likelihood of being captured by bubble particles (Dai, Fornasiero and Ralston, 1999; Wiese and Harris, 2012; Ahmadi, Khodadadi, Abdollahy and Fan, 2014).

The large bubbles (1-3 mm) generated in conventional flotation cells are inefficient for the flotation of these small particles. As a result, both the flotation time and collector usage rise significantly. An encouraging solution to this issue involves the utilization of small microbubbles, each less than 0.1 mm in diameter through microflotation. The microflotation process involves the extraction of fine suspended minerals from water by employing microscopic gas bubbles, typically 100 μm or smaller in size. These small bubbles have a higher efficiency in capturing smaller particles compared to the millimeter-sized bubbles utilized in traditional flotation techniques (Loewenberg and Davis, 1994).

Figure 2. Microflotation Cell

Froth flotation fundamentally involves collecting minerals through ascending bubbles. To achieve effective mineral recovery via flotation, crucial steps like bubble-particle collision and particle adhesion to bubbles are necessary. When an external force surpasses the internal attachment force within a particle-bubble aggregate, the particle initially adhered to the bubble will detach. The stability of these aggregates depend on the balance of forces exerted on them. Frothers are introduced to generate small bubbles and enhance froth stability. The stability of the froth is not only influenced by the frother, but also by the properties of the suspended particles (Johansson and Pugh, 1992; Schwarz and Grano, 2005).

The number of particles present in the system is thought to influence viscosity directly. Additionally, the viscosity

of the flotation medium could impact the recovery achieved through flotation. According to Xu, Ametov and Grano (2011), bubble-particle aggregates tend to exhibit greater stability in suspending mediums with higher viscosity levels.

Since flotation separation is employed in various systems, the equipment utilized for the procedure differs. Classification can be based on how gas bubbles are generated and introduced, the degree of turbulence, and the direction of phase movement relative to each other. Machine capacities may range from small fractions of a cubic centimeter to several hundred cubic meters.

The complexities outlined above pose significant challenges in scaling up flotation techniques. Furthermore, unlike numerous other separation methods, extending the duration of flotation separation does not always enhance its metallurgical outcomes. Hence, accurately determining the arrangement and dimensions of flotation cells based on batch and pilot test outcomes (scaling up) is paramount for achieving favorable overall results.

Despite all factors taken into account, the usual method involves identifying the optimal parameters through laboratory-scale tests (such as conditioning and separation time, etc.) and subsequently applying them to industrial-scale operations using an empirical "scaleup factor". The utilization of data from small-scale column flotation cells in designing large industrial flotation columns is highly significant and has been thoroughly addressed in literature. (Dobby and Finch, 1990; Dobby and Finch, 1991; del Villar, Finch, Gomez and Espinoza-Gomez, 1992; Luttrell, Mankosa and Yoon, 1993).

Pyrite is the most common sulphide gangue mineral in ore deposits. Flotation of pyrite mineral is a critical step in the ore preparation process aimed at separating valuable minerals from gangue. For effective separation, the factors affecting the pyrite flotation process must be fully understood. As a result, extensive studies on pyrite depression and flotation behavior are still ongoing (Zhang, F., Zhang, C., Zhang, H., Chen, Wang, Chen, D., Chen, J., Tian and Sun, 2023; Jiang, K., Han, Y., Liu, J., Wang, Y., Ge, W. and Zhang, D. , 2023; Zhang, Peng, Wang, Cao, Qi and Huang, 2024). This study aimed to investigate the existence of correlation between microflotation and batch flotation experiments of pyrite with different collector usage at varied pH range.

3. Material and Method

Pure pyrite samples from Kütahya region collected by hand were crushed and ground in a mortar, dry sieved to produce a size fraction less than 425 µm, and then air tightly packaged and stored at -18 °C to prevent oxidation. 2 g of the prepared pyrite samples were

weighed and ground to d_{80} :76 μ m. Grinding operations were carried out in the mill shown in the Figure 3a. The grinding medium is designed for the use of 30 steel balls of size 6 mm in the glass mill $(\emptyset 40x75$ mm). Wet grinding is carried out by adding 2 ml of distilled water to the mill. The grinding process is carried out by placing the mill with the grinding media and pyrite sample on the Retsch sieve shaker (Figure 3b). Based on the data obtained from the preliminary tests, the grinding times were determined as 2.5 minutes. The particle size distributions of the wet ground pyrite samples are analyzed with a Malvern Mastersizer Hydro 2000 MU Particle Size Analyzer using the laser diffractometer method (Figure 4).

Figure 3. a) Glass Mill Used for Grinding b) Sieve Shaker

Figure 4. Particle Size Distribution of Pyrite

Batch flotation tests are carried out in a 1,25-liter volume flotation cell using a Denver D12 type with an impeller speed of 1300 rpm at an air flow rate of 6 dm³ min-1 (Figure 5a). Flotation experiments were carried out with the artificial mixture obtained by wet ground pyrite sample (2 g) and quartz (300 g). The ground pyrite samples were artificially mixed with the - 212+150 µm quartz samples to ensure proper pulp rheology during the batch flotation tests. All batch flotation tests were conducted at about 30% solids.

Following the flotation process, the float and sink products acquired were dried, and sieved through a 150 µm to separate the quartz that could entrained into the

concentrate then measured in weight. The recovery was determined as a percentage by comparing the quantity of floated material to the quantity of feed. All the experiment was conducted in duplicate to ensure that any identified changes were statistically significant.

Figure 5. a) The Batch Flotation Cell b) The Microflotation Cell Used in The Tests

2 g of pyrite mineral was mixed with 50 ml of distilled water in the beaker with a magnet at 500 rpm. The slurry was transferred into the microflotation cell (Figure 5b) with a capacity of 70 ml. Microflotation tests were carried out at an airflow rate of 0,4 dm³ min-1. The mixing process of the pulp was carried out using a IKA stirrer and a magnet in the cell at 750 rpm.

The most commonly used xanthate type collectors Potassium Ethyl Xanthate (PEX) and Potassium Amyl Xanthate (PAX) from ECS Chemistry Inc. were selected and used in the experiments. Xanthate solutions were prepared daily. Aerofroth 70 (AF-70) supplied by Solvay Group was used as a frother and NaOH and HCl as the pH adjusting agents. The experiments were conducted using 15, 25 and 30 g/t collectors with the frother at 48

g/t. The pH was controlled during the experiments using a Hach Lange pH meter.

4. Results

Numerous research efforts have demonstrated the impact of pH on mineral flotation, the pH level of the pulp being a critical indicator during the flotation process (Lin, Wang, Liu, Sun, and Jing, 2022). In the literature, the floatability of pyrite was investigated at varying pH dosages. While there are two humps in which pyrite can float in alkaline and acidic environments, it has been shown that it is depressed at points close to neutral pH values (Fuerstenau, Kuhn and Elgillani ,1968; Fuerstenau, Misra and Palmer, 1980).

In order to determine the pyrite floatability in the use of different collectors at varying dosages and pH values, a series of experiments were carried out. Pyrite was floated between pH 7.5 and 9.5 in the use of PAX and PEX at varying dosages (15, 25, and 30 g/t).

While Figure 6a shows the results of the experiments using PAX, Figure 6b shows the results of the experiments using PEX. In Figure 6 , it is observed that as pH values increase, pyrite recovery values decrease at all reagent dosages. The reduced stability of dixanthogen as pH increases is also a contributing factor to the poor flotation of pyrite, given that dixanthogen is the primary xanthate species on the pyrite surface. The lowest recovery values are obtained in experiments using 15 g/t PAX and PEX. When using 15 g/t PAX, a recovery value of 78.09% was obtained at pH 7.5, whereas recovery value of 70.61% was obtained at pH 9.5. In addition, the recovery values were acquired in the use of 25 g/t and 30 g/t collectors are close to each other. At pH 7.5, 86.64% recovery value was obtained in the use of 30 g/t PAX. In the use of 15 g/t PEX, 74.66% recovery was obtained at pH 7.5 and 68% recovery at pH 9.5.

Figure 6. a) Pyrite Recovery vs pH at Different PAX Dosages in the Batch Flotation Cell b) Pyrite Recovery vs pH at Different PEX Dosages in the Batch Flotation Cell

In Figure 7a, it is seen that the pyrite recovery decreases with increased pH values in the use of PAX. On the other hand, the increased use of PAX dosage has resulted in increased pyrite recovery. Comparable recovery values have been achieved for both types of collectors using 25 g/t and 30 g/t. Figure 7 b shows that parallel to the results obtained with PAX, the recovery values of pyrite decrease along with the rise in pH values. When the results of microflotation experiments are evaluated, the pyrite recoveries obtained with PAX are higher than with PEX. The data obtained from the microflotation experiments are parallel to the results of the batch flotation experiments.

The findings indicated that PAX consistently yielded the highest relative recoveries across all scenarios. The length of the carbon chain of the collector is recognized to affect the hydrophobic nature of the mineral surface. Enhancing the length of the hydrocarbon chain in the collector leads to a rise in the contact angle (Wark and Wark, 1932). The trend of increasing hydrophobicity of the mineral surface aligns with the pioneering research by Wark and Wark (1932), demonstrating that longer collector chain lengths corresponded to higher contact angles on pyrite.

Figure 7. a) Pyrite Recovery vs pH at Different PAX Dosages in the Microflotation Cell b) Pyrite Recovery vs pH at Different PEX Dosages in the Microflotation Cell

Figure 6 and 7 contain data from experiments conducted in batch flotation cell and microflotation cell. When the data are examined, the recoveries in microflotation appear to be higher than in batch flotation. However, the trends between the two flotation techniques are similar. In order to determine the relationship between two different flotation techniques, the relationship graph of all the findings is given in the following figures. As seen in Figures 8 a and b, it is seen that the two techniques are in high agreement with each other, the results of the experiments with PAX have a 94% correlation, and the results of the experiments with PEX have a correlation of 98%. The data indicate that the two flotation techniques may be used alternatively to determine the flotation behavior of the investigated pure pyrite mineral by using the microflotation technique, which is a simpler and more practical procedure.

Figure 8. The Correlation Between Batch Flotation and Microflotation with Different Collector Usage (a: PAX b: PEX)

5. Conclusions

In this study, the flotation behaviour of pyrite, an important sulphur mineral gangue, was examined at various pH levels and various concentrations of collectors with different chain lengths by batch flotation and microflotation experiments, and the correlations between the two flotation techniques were determined.

In the pH range studied, a decrease was observed in the pyrite recovery as the pH value increased which is consistent with the literature. The highest recovery value, 90%, was reached in the use of 30 g/t PAX at pH 7.5. Due to the longer chain length of PAX, higher recovery values were obtained in experiments using PAX compared to experiments performed with PEX.

Higher recovery values were acquired in microflotation tests compared to batch flotation tests. This can be explained by the production of a finer and higher amount of bubbles in microflotation compared to the batch flotation experiments. Despite this, a high correlation was obtained between these two techniques. In the experiments performed with PAX, the R' between the two flotation techniques was obtained as 0.9478, whereas in the experiments conducted with PEX, R was obtained as 0.98.

The findings show that the two flotation techniques can be used interchangeably in determining the flotation behaviour of pure pyrite minerals studied. In this specific case, results can lead to scale-up can be carried out with the microflotation technique, which is a simpler and more practical method. This shows that the microflotation method, which is smaller and relatively easier to perform, can be used to estimate cell flotation efficiencies. Moreover, by developing such models, predictions can be made for plant design through the microflotation method. On the other hand, it should be remembered that the relationship between these two flotation techniques is limited to the parameters and conditions used in this study. In future studies, the relationship between these two flotation techniques will be revealed more clearly by working with different minerals and mineral groups.

Author Contributions

Işıl TOKCAN and Hasan Serkan GÖKÇEN contributed to the publication with the design and implementation of the research, analysis of the results, and writing of the manuscript and discussion of the results and review of the manuscript.

Conflict of Interest

No conflict of interest was declared by the authors.

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