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## Comparison of mechanical flotation cell and cyclonic microbubble flotation column in terms of separation performance for fine graphite

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**Abstract:** Comparison of flotation performance between the flotation column and mechanical flotation was carried out to promote the grade and economic value of the graphite ore (15.40% ash content). The ash content of the concentrate of the mechanical flotation was 10.77% at the yield of 79.34%. In contrast, the yield of the concentrate of the column flotation was increased to 88.93% with 10.55% ash content. Comparative study of the Fuerstenau upgrading curves indicated that the column flotation was more efficient for cleaning the graphite ore in the presence of the centrifugal force field, nanobubbles (generated by hydrodynamic cavitation), and the thicker froth layer in comparison with the mechanical flotation.

**Keywords:** mechanical flotation, column flotation, Fuerstenau upgrading curve, graphite

### 1. Introduction

Natural graphite may occur in metamorphosed siliceous rocks (typically quartzite) as well as metamorphosed carbonate rocks. Associated minerals with graphite occur as quartz, rock-forming silicates such as amphiboles, kaolinite, mica, and feldspars, also calcite, sulphides, or magnetite (Wakamatsu and Numata, 1991; Chelgani et al., 2015). Flotation is a main beneficiation method for graphite ores (Wakamatsu and Numata, 1991; Lu and Forssberg, 2001; Shi et al., 2015; Jin et al., 2016). Mechanical and column flotation technologies have been used to upgrade graphite ores by combining a simple rougher flotation step with several stages of cleaning. Column flotation can give superior performance compared to mechanical cell by either reducing the number of stages of operation or by improving the grade and recovery (Acharya et al., 1996; Misra, 2003; Abd El-Rahiem, 2004; Vasumathi et al., 2015).

Centrifugal force field flotation has been used to improve the separation efficiency of fine particles since the 1980s (Miller and Camp, 1982). Air-sparged hydrocyclone (ASH), pioneered by Miller and Camp (1982), diminishes the entrainment of gangue materials via an increase in the fine particle flotation rate and froth drainage in the presence of a centrifugal force (Tils and Tels, 1992; Yalamanchili and Miller, 1995; Miller and Halbe, 2005). Basically, the flotation column consists of two distinct zones, the collection zone (the bubbling zone and slurry or pulp zone) below the interface, and cleaning zone (the froth zone) above the interface (Dobby and Finch, 1991). Cyclonic microbubble flotation column, developed by China University of Mining and Technology, contains an additional zone named cyclonic zone compared to the other flotation column (Bu et al., 2016b). The bubble generator used in the flotation column, taking advantage of the Venturi principle, is the most widely used hydrodynamic cavitation device (Fan, 2010). The occurrence of nanobubbles by hydrodynamic cavitation increases the probability of collision and attachment and decreases the probability of detachment compared with conventional-

size bubbles (Tao et al., 2006). In other words, cyclonic microbubble flotation column can extend the lower particle size limits for effective flotation due to the combination of nanobubbles and centrifugal force separation (Miller and Camp, 1982; Rahman et al., 2014). In contrast to mechanical flotation cells or dissolved air flotation column, the cyclonic microbubble flotation column was more efficient for the beneficiation of coal fines (Li et al., 2003; Bu et al., 2017b), siliceous phosphate ore (Li et al., 2012), magnetite ore (Zhang et al., 2013), and oil-in-water emulsion (Li et al., 2016).

It is observed that the literature regarding the cyclonic microbubble flotation column lacks emphasis on the graphite flotation performance. So, in this article, the cyclonic microbubble flotation column was employed to upgrade the graphite ore, and a comparison of the mechanical and column flotation was also carried out in terms of flotation separation efficiency.

## 2. Materials and methods

### 2.1 Materials

Graphite sample from south China had 15.40% ash content. The size distribution was determined by a Microtrac S3500 device using a laser diffraction method (Microtrac Inc., USA). The calculated  $d_{95}$  of the sample was 68.92  $\mu\text{m}$ . The mineralogical phase was investigated by XRD analysis (Bruker D8 Advance, Germany). The detailed operating process of the XRD measurements has been described in the literature (Bu et al., 2017a). The major impurity mineral of this sample was quartz according to the X-ray diffraction (XRD) measurement (Fig. 1).

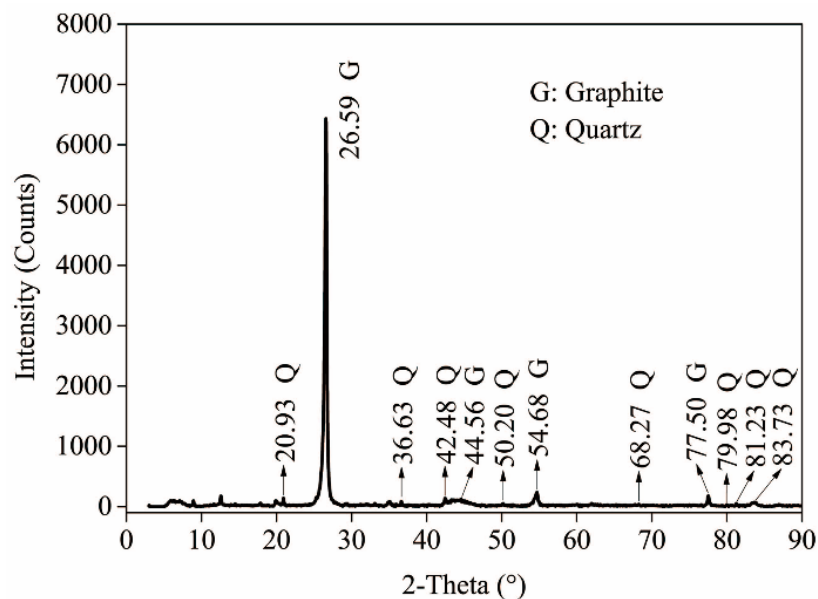


Fig. 1. XRD patterns of the graphite ore

### 2.2 Mechanical flotation

Kerosene and sec-octyl alcohol (analytical reagent, Sinopharm Group) were used as collector and frother, respectively. A standard laboratory RK/FD-II sub-aeration flotation cell (volume = 1.5  $\text{dm}^3$ ) (Fig. 2.). The sample was conditioned at 5-30% solids by weight with tap water. The required amount of kerosene was added and conditioned for three more minutes. After that, the required amount of sec-octyl alcohol was added, and the slurry was conditioned for 1 min. Then the air valve was opened at 4.17  $\text{dm}^3/\text{min}$  air flow rate and 1900 rpm agitation speed, and the froth was collected for 3 min. In each test, tap water was added to maintain a constant pulp level and a froth layer of 1 cm. The experiments were carried out by varying different operating parameters such as collector concentration, frother concentration and solids concentration. A detailed description of the working process of the mechanical flotation cell is reported in the literature (Bu et al., 2016a).



Fig. 2. Actual diagram of experimental system for mechanical flotation tests

### 2.3 Column flotation test

Column flotation tests were carried out using a laboratory-scale flotation column (diameter = 100 mm, height = 1800 mm) in China University of Mining and Technology, Xuzhou, China. The effects of the collector dosage, froth dosage, froth depth and circulation on the flotation performance of the column flotation were investigated. The sample was prepared at 5.81% solids by weight with tap water for each column flotation test. The procedure of the flotation column test is described in previous literature (Bu et al., 2017b).

## 3. Results and discussion

### 3.1 Mechanical flotation

#### 3.1.1 Effect of collector dosage

The effect of collector dosage in the mechanical flotation performance was investigated with 0.75 g/kg frother dosage (mass of frother per unit mass of solids) and 5.81 wt. % solids concentration. As shown in Fig. 3., the yield of the concentrate increased significantly from 50.01% to 91.25% with the increasing collector dosage, while the ash content also witnessed an increase from 10.86% to 11.66%. High collector dosage can enhance the flotation yield, which also leads to the low selectivity between graphite and quartz (Shi et al., 2015).

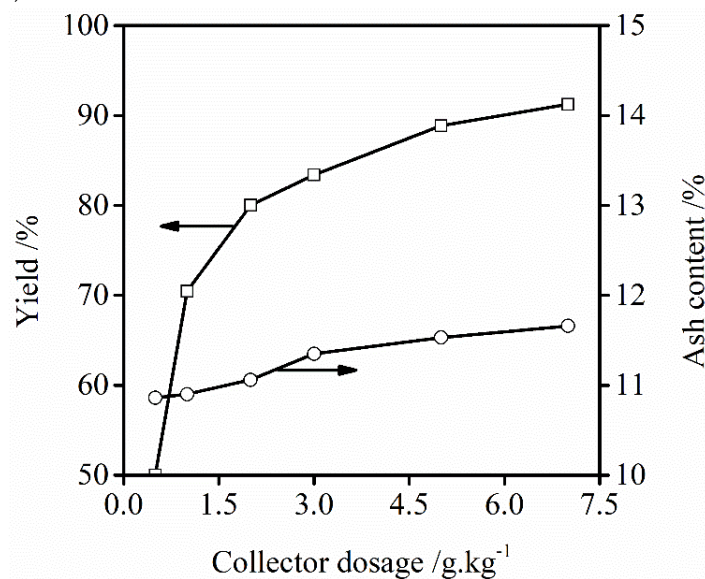


Fig. 3. Effect of collector dosage on the mechanical flotation

### 3.1.2 Effect of frother dosage

The variations of the yield and ash content of the concentrate at various frother dosages with the collector dosage of 3 g/kg and solids concentration of 5.81 wt. % are shown in Fig. 4. With the increase in frother dosage from 0.75 g/kg to 1 g/kg, the yield varied from 83.41% to 84.37%, while the ash content revealed a decrease from 11.35% to 11.18%. The adsorption of frothers on the gas/liquid interface can prevent bubble coalescence, which can lead to the high-grade product by stable froth (Harris, 2000). With the further increase in the frother dosage, the serious entrainment of gangue materials results from the exorbitantly stable froth, which leads to the increase of the ash content of the concentrate, which is in agreement with previous observations by Nguyen and Schulze (2004).

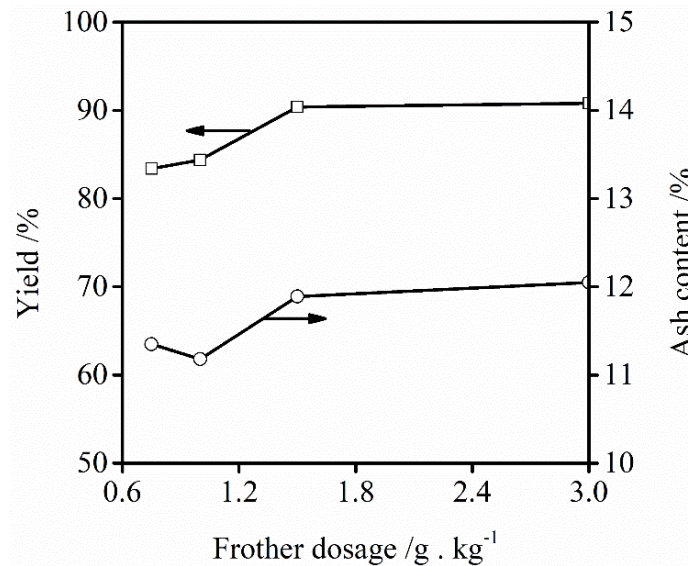


Fig. 4. Effect of frother dosage on the mechanical flotation

### 3.1.3 Effect of solids concentration

The influence of solids concentration on mechanical flotation at 3 g/kg collector dosage and 750 frother dosage is presented in Fig. 5. The yield witnessed an increase from 79.34% to 94.97% with increasing solids concentration. Meanwhile, the ash content of the concentrate increased apparently from 10.77% to 14.21% with the decrease in solids concentration. This is attributed to the more serious entrainment caused by the large amount of water transferred to the froth product due to the stable froth at a high solids concentration and frother dosage (Akdemir, 2003; Li et al., 2015).

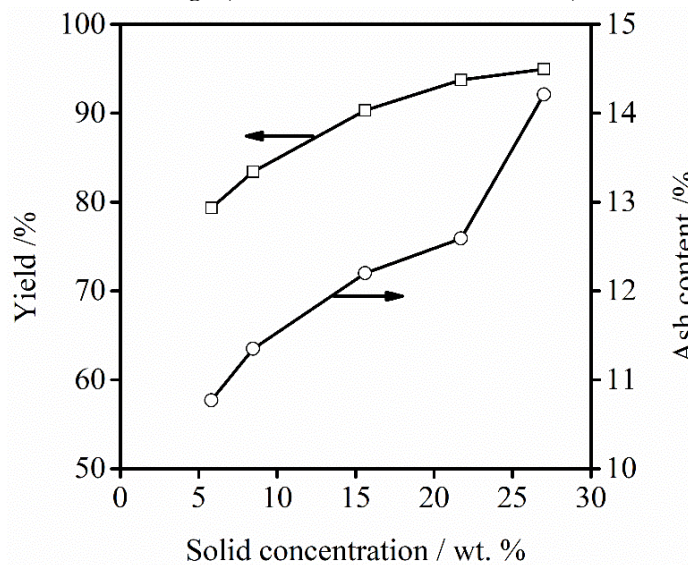


Fig. 5. Effect of solids concentration on the mechanical flotation

### 3.2 Column Flotation

#### 3.2.1 Effect of collector dosage and frother dosage

Fig. 6. presents the effect of collector dosage and frother dosage on the flotation performance of the column flotation at 0.15 Mpa circulation pressures and 20 cm froth depth. The ash content of the concentrate (10.86% - 12%) using the flotation column exhibited the same trend to that of the mechanical flotation, under different collector dosages (or frother dosages). At a low collector dosage (<3000 g/Mg), the yield of the concentrate the yields of the flotation column was much higher than that of the mechanical flotation cell. This is due to the increase in the collision probability between fine particles and bubbles in the presence of the centrifugal force field and nanobubbles (Miller and Camp, 1982; Rahman et al., 2014).

With the increase in the frother dosage, the ash content of the flotation column varies from 11.46% to 11.98%. This high ash content of the concentrate can be explained by the severe entrainment resulting from the stable froth. The froth is stabilized by fine hydrophobic graphite particles and becomes very tenacious (Ata et al., 2004; Li et al., 2014). A more stable froth has fewer coalescence and bursting events, which leads to the increase in the amount of material recovered by entrainment.

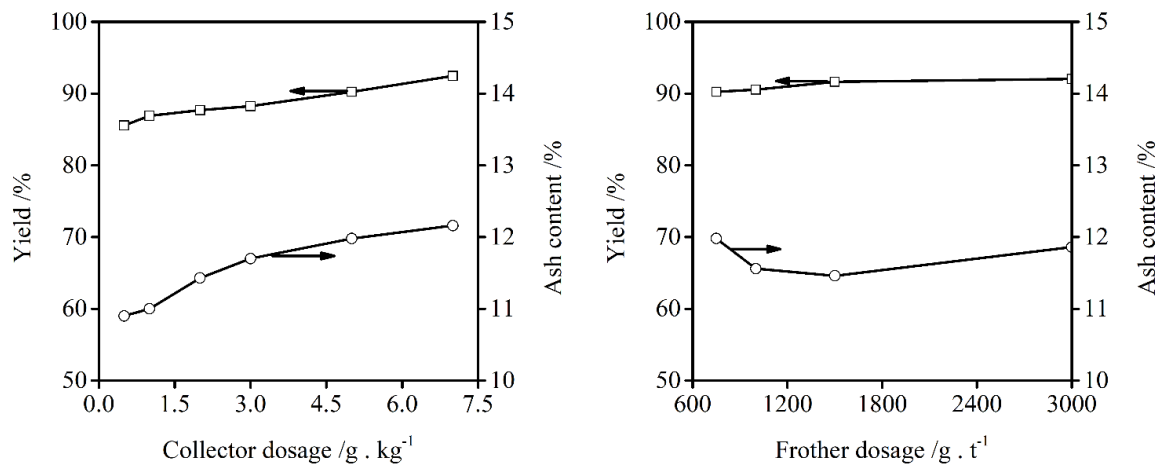


Fig. 6. Effect of collector dosage and frother dosage on the column flotation (left: 0.75 g/kg frother dosage; right: 5 g/kg collector dosage)

#### 3.2.2 Effect of circulation pressure

Column flotation tests carried out at various circulation pressures with 20 cm froth depth, 5 g/kg collector dosage and 1.67 g/kg frother dosage are shown in Fig. 7. The yield and ash content of the concentrate witnessed a stable increase in a relatively low circulation pressure (0.15 MPa - 0.20 MPa). The lack of the enough strength of the negative pressure in the bubble generator causes the low superficial air velocity and big bubbles under a relatively low circulation pressure (Li et al., 2003). With the circulation pressure increasing from 0.15 MPa to 0.20 MPa, the increase in the gas dispersion promotes the recovery of fine particles, while the entrainment of gangue materials also gets serious. With the further increase in the circulation pressure, the ash content and the yield decreased significantly, which relates to the increase in the centrifugal force strength of the flotation column. The strong centrifugal force can improve the particle-bubble collision frequency, which leads to the improvement in the concentrate grade (Zhou and Liu, 2007). Furthermore, the application of the Venturi principle in the bubble generator can enhanced hydrophobic particle aggregation and bubble-particle collision probability in the occurrence of nanobubbles generated by hydrodynamic cavitation (Calgaroto et al., 2015; Zhou et al., 2016). With the increase in the separation efficiency, a large amount of gangue materials reports to the tailings, resulting in the decrease of the yield.

#### 3.2.3. Effect of froth depth

Fig. 8. presents the influence of froth depth on the column flotation performance at 0.15 MPa circulation pressure, 5 g/kg collector dosage and 1.67 g/kg frother dosage. With the increasing froth depth, the

yield of the concentrate decreased from 95.67% to 89.41%, while the ash content of the concentrate decreased from 12.32% to 10.79%. At higher froth depth, the froth residence time is longer, which results in that more gangue materials detach from the froth zone. The mechanical entrainment and entrapment of gangue materials decrease in column flotation in the presence of a thick froth layer (Yianatos et al., 1988).

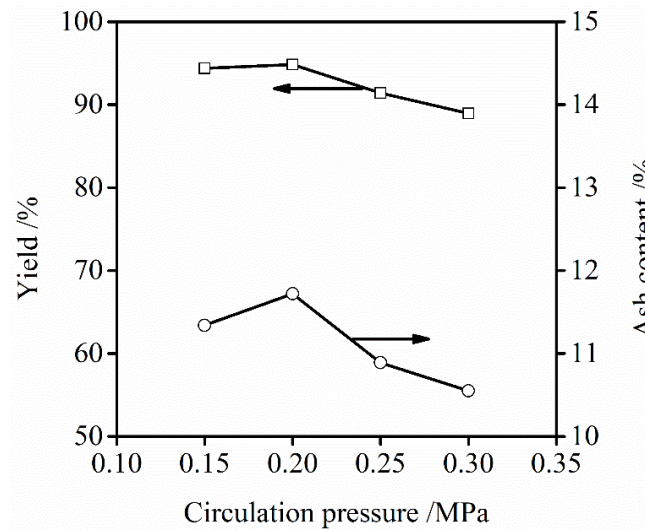


Fig. 7. Effect of circulation pressure on the column flotation

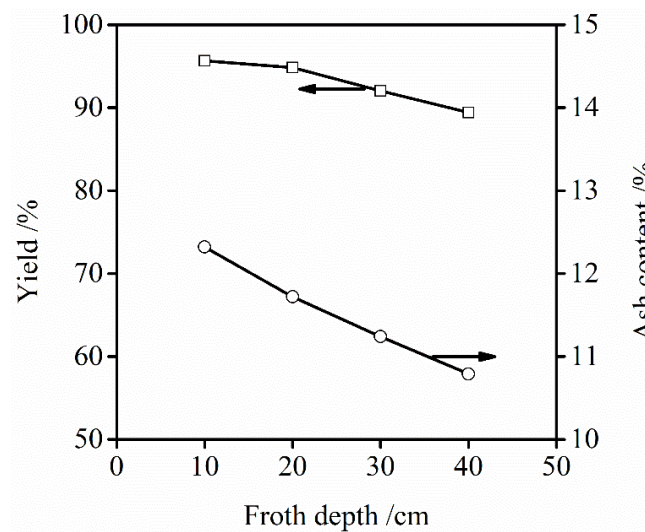


Fig. 8. Effect of froth depth on the column flotation

### 3.3 Comparison of separation efficiency

The Fuerstenau upgrading curves have been used in the characterization, comparison and analysis of the beneficiation for coal fines (Drzymala, 2005; Fuerstenau, 1978; Jia et al., 2002; Li et al., 2013), Polish copper ore (Drzymala et al., 2003; Drzymala et al., 2013; Drzymala et al., 2010; Zarudzka, 2009). Those curves can be approximated by various mathematical equations with simple multi-parameter polynomials and ending with more sophisticated formulas (Drzymala and Ahmed, 2005). An equation containing only one adjustable parameter, which is used as a measure of selectivity of separation is used to compare the flotation separation efficiency between the mechanical and column flotation. This equation can be represented as follows:

$$R_{1,C} = \frac{100^\alpha - R_{2,T}^\alpha}{100^{\alpha-1}} \tag{1}$$

where  $R_{1,C}$  stands for the recovery of valuable matter (also known as non-ash matter) in the concentrate and  $R_{2,T}$  denotes the recovery of ash matter in the tailings, respectively.  $\alpha$  is the separation selectivity factor. If  $\alpha = 1$ , there is no upgrading between graphite and gangue minerals; when  $\alpha > 1$ , the upgrading process exists in the concentrate. The bigger the value of  $\alpha$  is, the higher the separation efficiency is; the upgrading process occurs in the tailings with the value of  $\alpha$  between 0 and 1; idea separation presents when  $\alpha$  is equal to 0 or  $\infty$ .

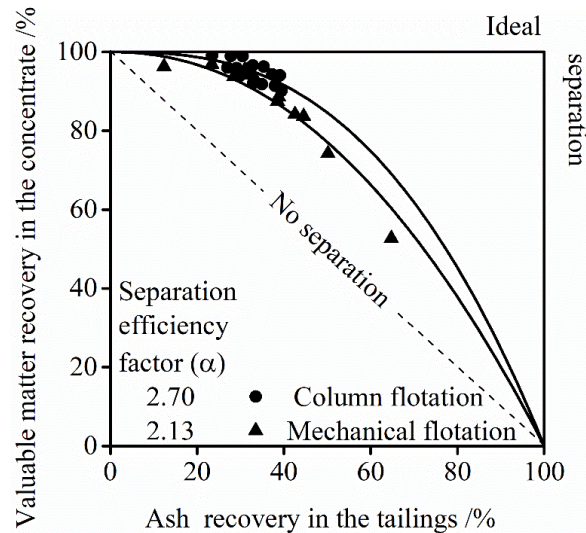


Fig. 9. The Fuerstenau upgrading curve of the mechanical and column flotation

The upgrading curves of the mechanical and column flotation are presented in Fig. 9. The lines represent the Fuerstenau upgrading curves for the mechanical and column flotation obtained by fitting their experimental results, respectively. The calculated separation efficiency factor ( $\alpha$ ) results by MatLab software are shown in Table 1. The lowest ash content was obtained at 5.81 wt. % solids concentration, 20 cm froth depth and 0.30 MPa circulation pressure. The amount of the collector and frother were 5 g/kg and 1.67 g/kg, respectively. The optimum flotation performance of the mechanical flotation cell was obtained at 5.81 wt. % solids concentration, 3 g/kg collector dosage and 0.75 g/kg frother dosage.

Table 1. Comparison of the efficiency factor and optimum flotation performance between the mechanical and column cells

Flotation device	Concentrate		Model Parameter	
	Yield, %	Ash content, %	$\alpha$	$R^2$
Mechanical	79.34	10.77	2.13	0.98
Column	88.93	10.55	2.70	0.99

Joglekar and May (1987) suggest that for a good fit of a model, the coefficient of determination ( $R^2$ ) should be at least 0.80. The  $R^2$  values were above 0.80, which indicated that Equation (1) could be used for the approximation of separation results. The separation efficiency factor of the column flotation was higher than that of the mechanical flotation, which indicated that the separation between graphite and gangue particles using the flotation column was closer to the ideal separation. However, there is still a big difference of the separation performance between the flotation column and the ideal separation. In the column flotation, the thick froth layer and long residence time can diminish the entrainment of ash materials compared to the mechanical flotation. Furthermore, the yield of the concentrate of the column flotation was increased from 79.34% to 88.93% at the similar level of the ash content in comparison with the mechanical flotation. This can be attributed to the scavenging process in the use of the centrifugal force and nanobubbles.

#### 4. Conclusions

The ash content of the graphite was reduced from 15.40% to about 10%-11%. The cyclonic microbubble flotation column technology can significantly increase the yield from 79.34% to 88.93% in the use of

centrifugal force and nanobubbles in comparison with the mechanical flotation. Comparison of the Fuerstenau upgrading curves indicated that the upgrading process of the column flotation was closer to the ideal separation than that of the mechanical flotation.

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