

*Received June 29, 2016; review; accepted January 27, 2016*

## Flotation behavior of nickel sulfide ore in a cyclonic flotation column

Lijun Deng<sup>\*</sup>, Guosheng Li<sup>\*</sup>, Yijun Cao<sup>\*\*</sup>, Zilong Ma<sup>\*\*</sup>

<sup>\*</sup> School of Chemical Engineering and Technology, China University of Mining and Technology, Xuzhou 221116, China. Corresponding author: lgscumt@163.com (Guosheng Li)

<sup>\*\*</sup> National Engineering Research Center of Coal Preparation & Purification, Xuzhou 221116, China

**Abstract:** This study aimed to show the flotation behavior of a nickel sulfide ore in a cyclonic flotation column. The flotation experiments were carried out using a sample of nickel sulfide ore obtained from a mineral processing plant of China. Representative samples collected from the feed, concentrate, tailing, and circulation middling were sieved using a cyclonic particle analyzer to collect different size fractions for analysis. The function of the column cyclonic zone was evaluated by comparing the quality of tailing and circulation middling. The flotation results showed that the concentrate with Ni grade of 1.78% and recovery of 65.56% was obtained under given test conditions. The content of main sulfide minerals and coarse particles in the circulation middling was higher than that in the tailing. The results indicated that, unlike conventional cyclone classification, separation achieved in the cyclonic zone of the column was not dependent on the particle size and density. Sulfide minerals with good floatability were easily captured by bubbles and moved toward the center of the column, even if these particles were coarser and heavier.

**Key words:** *cyclonic flotation column, floatability, flotation, nickel sulfide ore*

### Introduction

A conventional flotation column is a reactor mainly composed of collection and froth zones. The collection zone has the objective of attaching hydrophobic particles to bubbles, while the froth zone is responsible for the carrying capacity and froth enrichment (Jameson, 2010). Some typical flotation columns such as the Microcel (Yoon et al., 1992), packed (Yang et al., 2003) and Leeds flotation column (Degner and Person, 1991) were studied systematically in terms of both fundamental principles and industrial application. As an important method of mineral processing technology, the column flotation has reached its maturity and offered some advantages over the conventional flotation cell. Advantages of a column flotation include the requirement of less physical space, less power and greater ability to recover valuable fines at a

better grade of target mineral due to either minimization or prevention of hydraulic entrainment of undesirable fines (Gu and Yalcin, 2012; Dey et al., 2013; Gui et al., 2014).

Introduction of the cyclonic circulation method is one of the most important development in the column flotation technology in recent years. The air-sparged hydrocyclone (ASH), which was distinguished by the high-capacity flotation of fine particles in a centrifugal field, appeared at the beginning of the 1980s (Miller, 1981). Since then, numerous studies have been conducted on ASH including both theoretical investigations (Van Deventer et al., 1988; Das and Miller, 1996) and application developments (Yalamanchili and Miller, 1995; Niewiadomski et al., 1999; Liu et al., 2013; Li et al., 2015).

Yalcin (1995) indicated that the centrifugal force field in the flotation device could provide a high rate of bubble–particle contact and enable the process to take place in a very short column. The cyclonic flotation column used in this study is a novel method developed in the recent years at the China University of Mining and Technology. Related studies indicated that the column had some advantages over the conventional flotation machines (Cao et al., 2012; Gui et al., 2013; Zhang et al., 2013).

In this work, the flotation behavior of nickel sulfide ore in the cyclonic flotation column was evaluated in the laboratory scale. The function of the column cyclonic zone was evaluated by comparing quality of the tailing and circulation middling. The force analysis of particles and bubbles in a centrifugal force field was used to demonstrate the separation mechanism of the column used.

## **Experimental**

### **Materials and reagents**

The flotation experiments were carried out using a sample of nickel sulfide ore obtained from a mineral processing plant of Jin-Chuan Mining Company, Jin-Chang, China. The sample was received in a slurry form with a solid concentration of 20% by weight. Nearly 85% of the particles were finer than 74  $\mu\text{m}$ . As valuable minerals, the grade of Ni and Cu was 0.55 and 0.34%, respectively. The mineralogical analysis of the feed indicted that pentlandite, chalcopyrite and pyrite were the main sulfide minerals, while ophiolite and olivine were the main gangue minerals.

Sodium carbonate, cupric sulfate, and Calgon were used as the pH regulator, activator and inhibitor, respectively. Ethyl xanthogenate and ammonium dibutyl dithiophosphate (ABD) were applied together as the collector. Terpeneol oil was used as a frother. Both collectors and frother were provided by Jin-Chuan Mining Company.

## Experimental device

The schematic diagram of experimental set-up of the cyclonic flotation column is graphically presented in Fig. 1. The experimental apparatus consists of the mixing tank, peristaltic pump for feeding, circulation pump, tailing peristaltic pump, and column body. The column was made of Plexiglas, 100 mm in diameter and 2000 mm in height.

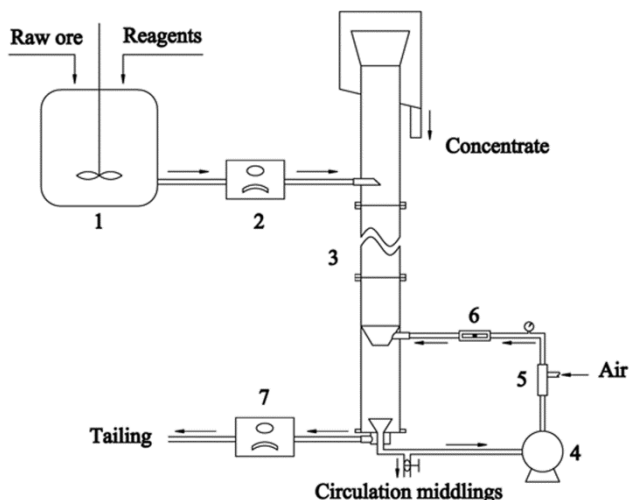


Fig. 1. Schematic illustration of the lab-scale cyclonic flotation column system.  
 1 mixing tank, 2 feed peristaltic pump, 3 column body, 4 circulation pump,  
 5 bubble generator, 6 gas flowmeter, 7 tailing peristaltic pump

## Methods

### Sample analysis

Representative samples collected from the feed, concentrate, tailing and circulation middling were sieved using a cyclonic particle analyzer to collect different size fractions for analysis. All the sieved samples were dried in an oven at about 100 °C and cooled to room temperature. The mineralogical compositions of the materials were determined using scanning electron microscope equipped with an energy-dispersive X-ray spectrometer (SEM-EDS). Size distribution of main sulfide minerals was analyzed using the Minerals Liberation Analyser (MLA) from Julius Kruttschnitt Mineral Research Centre in Australia.

### Flotation procedure and conditions

For the flotation process, a slurry with a certain pulp density (solid by weight) was conditioned in a mixing tank for 5 min. Then, the reagents were added subsequently. Conditioning between the additions of reagents was allowed. Conditioning times were 4 min for the pH regulator  $\text{NaCO}_3$ , 6 min for the activator  $\text{CuSO}_4$ , 4 min for the

depressant calgon, 5 min for the collector and 1 min for the frother. In the flotation tests, the column was run first with water and air until a steady state was reached. The feed was introduced from the upper section of the collection zone by pumping the slurry from the mixing tank. The circulation pressure of the flotation column was metered using a pressure gauge and controlled by the circulation pump. Air was self-induced, and micro-bubbles were generated in the self-aerating bubble generator. The superficial gas velocity was metered using a flow meter. The induced bubbles with floating particles attached to them rose to the column top to be discharged as a concentrate, and the non-floating material discharged via the column base as a tailing at a controlled rate. The sampling of circulation middling was achieved by switching the ball valve, as shown in Fig. 1. The test conditions are presented in Table 1. The conditions listed were the practical operational parameters of the mineral processing plant as well. It should be noted that the feeding rate was determined according to the practical flotation time of the nickel sulfide ore in the plant.

Table 1. Operating parameters used in the column flotation tests

Conditions	Value
Sodium carbonate (g/Mg)	1200
Cupric sulfate (g/Mg)	80
Calgon (g/Mg)	250
EX + ABD (g/Mg)	90 + 10
Terpineol oil (g/Mg)	30
Pulp density (%)	20
Pulp pH	7.8
Feeding rate (g/min)	230~250
Circulation pressure (Mpa)	0.32
Superficial gas velocity (cm/s)	1.8~2.0
Foam layer thickness (mm)	300~350

### Flotation behavior of particles in the centrifugal force field

The separation mechanism of the cyclonic flotation column is shown in Fig. 2. In the cyclonic separation zone, the circulating materials are split into three parts due to the centrifugal force. The particles with high density, which are not adhered to bubbles, move toward the column wall, and then downward along the column wall, whereas the mineralized bubbles, which carry a substantial number of valuable minerals, move toward the column center, and then upward to the column separation zone. The remaining particles are pumped as the circulating middling stream (Zhou and Liu, 2007; Li et al., 2012).

Assuming that the shape of the particles and mineralized bubbles is spherical, forces exerted on the particles and mineralized bubbles in the radial direction of the

cyclonic separation zone of the column include centrifugal  $F_c$ , centripetal buoyancy  $F_b$ , and drag  $F_\mu$  forces. For the mineralized bubbles, these forces can be defined as:

$$F_c = \frac{\pi}{6} d_m^3 \rho_m r \omega^2 \quad (1)$$

$$F_b = \frac{\pi}{6} d_m^3 \rho r \omega^2 \quad (2)$$

$$F_\mu = 3\pi\mu d_m u_b \quad (3)$$

where  $\pi = 3.14$ ,  $d_m$  is the equivalent diameter of the mineralized bubble,  $\rho_m$  is the density of the mineralized bubble,  $r$  is the radius of gyration,  $\omega$  is the angular velocity of the pulp,  $\rho$  is the density of the fluid,  $\mu$  is the viscosity of the pulp, and  $u_b$  is the relative velocity between the pulp and mineralized bubble. Dynamic equations of the mineralized bubble in the radial direction can be expressed by:

$$F_b - F_\mu - F_c = m \frac{du_b}{dt} \quad (4)$$

When the radial velocity of the mineralized bubble remains constant, the forces acting on it are in equilibrium. This condition can be defined as:

$$\frac{du_b}{dt} = 0 \quad (5)$$

Substituting Eqs. (1), (2), (3), and (5) into Eq. (4) leads to:

$$u_b = \frac{d_m^2 r \omega^2 (\rho - \rho_m)}{18\mu} \quad (6)$$

where  $\rho_m$  can be defined as:

$$\rho_m = \left( 1 - \frac{d_b^3}{d_b^3 + n d_p^3} \right) \rho_p \quad (7)$$

where  $\rho_p$  is the density of the particles,  $d_b$  is the diameter of the bubble,  $d_p$  is the diameter of the particle,  $n$  is the number of particles attached to the bubble.

Similarly, the radial velocity of the particle ( $u_p$ ) can be defined as:

$$u_p = \frac{d_p^2 r \omega^2 (\rho - \rho_p)}{18\mu} \quad (8)$$

In the flotation system, the density of mineralized bubbles ( $\rho_m$ ) is much smaller than that of the fluid ( $\rho$ ), and generally speaking, the density of particles ( $\rho_p$ ) that cannot adhere to bubbles is much higher than that of the fluid ( $\rho$ ). Therefore, the velocity of the mineralized bubbles and the hydrophilic particles that cannot adhere to bubbles will satisfy the following condition to Eqs. (6) and (8):

$$u_b > 0, u_p < 0. \tag{9}$$

Under the above condition, the mineralized bubbles move toward the center of the column. On the contrary, the hydrophilic particles that cannot adhere to bubbles travel in an opposite direction.

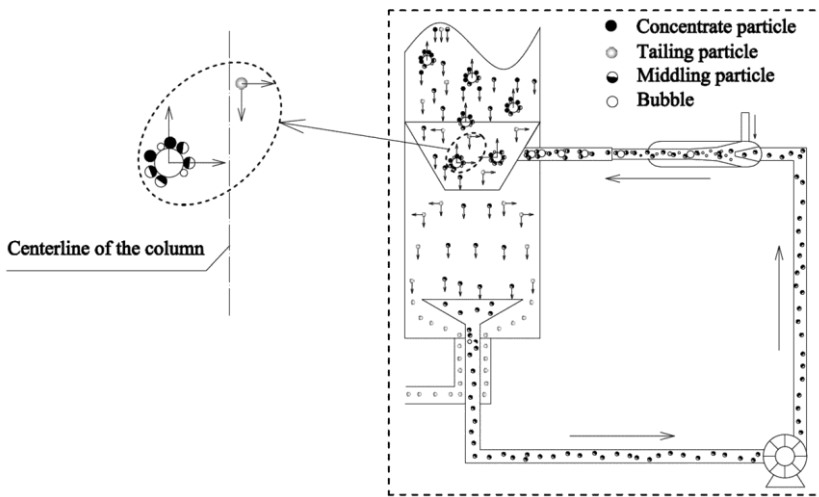


Fig. 2. Mechanism of cyclonic separation in the cyclonic flotation column

### Results and discussion

The objective of the present study was to ascertain the cyclonic flotation behavior of the nickel sulfide ore in the flotation column. Therefore, a parametric evaluation was not the focus of this study. Using the practical operating conditions listed in Table 1, flotation experiments were performed resulting in the separation performance described by the data in Table 2. When separating a sample with the Ni grade of 0.54%, a concentrate with Ni grade of 1.78% and recovery of 65.56% was obtained, and the Ni grade of the tailing was about 0.23%. Particle size analysis results of the concentrate and tailings are shown in Table 3. As shown in Table 3,  $Y_{CC}$ ,  $\beta_C$ ,  $Y_{TT}$  and  $\beta_T$  were obtained directly from the flotation results.  $Y_{CF}$ ,  $Y_{TF}$ ,  $Y_F$ ,  $\beta_F$  and  $\epsilon_C$  at individual size fraction were calculated according to the following equations:

$$Y_{CF} = Y_{CC}Y_C/100 \tag{10}$$

$$Y_{TF} = Y_{TT}Y_T/100 \quad (11)$$

$$Y_F = Y_{CF} + Y_{TF} \quad (12)$$

$$\beta_F = (Y_{CF}\beta_C + Y_{TF}\beta_T)/Y_F \quad (13)$$

$$\varepsilon_C = Y_{CF}\beta_C/Y_F\beta_T 100 \quad (14)$$

where the values of  $Y_C$  and  $Y_T$  are 19.87 and 80.13% (shown in Table 2), respectively.

Table 2. Results of the flotation test

Products	Yield (%)	Ni grade (%)	Ni recovery (%)
Concentrate	19.87	1.78	65.56
Tailing	80.13	0.23	34.44
Total	100.00	0.54	100.00

Table 3. Size analysis results of the flotation products

Size fraction/ $\mu\text{m}$	Concentrate			Tailings			Feed		Ni recovery in concentrate/%
	Yield (proportion in concentrate)/%	Yield (proportion in feed)/%	Ni grade/%	Yield (proportion in tailings)/%	Yield (proportion in feed)/%	Ni grade/%	Yield/%	Ni grade/%	
	$Y_{CC}$	$Y_{CF}$	$\beta_C$	$Y_{TT}$	$Y_{TF}$	$\beta_T$	$Y_F$	$\beta_F$	
+74	20.19	4.01	1.34	26.88	21.54	0.26	25.55	0.43	49.03
-74+45	8.98	1.79	2.40	8.23	6.59	0.33	8.38	0.77	66.27
-45+29	24.30	4.83	2.42	15.39	12.33	0.22	17.16	0.84	81.18
-29+21	6.37	1.27	1.38	8.84	7.08	0.19	8.35	0.37	56.44
-21+15	12.35	2.45	1.17	7.77	6.23	0.18	8.68	0.46	71.93
-15+11	5.63	1.12	0.98	5.18	4.15	0.18	5.27	0.35	59.49
-11	22.17	4.41	1.79	27.71	22.20	0.22	26.61	0.48	61.76
Total	100.00	19.87	1.76	100	80.13	0.23	100.00	0.54	65.29

As can be seen from Table 3, the Ni grades in the size fractions of 74–45  $\mu\text{m}$ , 45–29  $\mu\text{m}$ , and 29–11  $\mu\text{m}$  were improved to a greater degree compared with the other four size fractions. The size-by-size Ni recoveries of the concentrate are also presented in Table 3. As seen, the column allow to achieve the excellent recovery for all size fractions except for materials coarser than 74  $\mu\text{m}$ . For the size fraction of 45–29  $\mu\text{m}$ , the flotation recovery reached 81.18%.

In the flotation tests, sampling of the circulating middling and tailing streams was conducted to obtain the evidence of the cyclonic flotation behavior of the nickel sulfide ore in the centrifugal force field of the column.

The percentage of sulfide minerals (pentlandite, chalcopyrite, vallerite, and pyrite) in the circulating middling reached 6.55%, and was higher than that in the tailing by

0.97 percentage points, as shown in Table 4. Especially it should be noted that the pentlandite content in the circulating middling was higher than that in the tailing by 0.04 percentage points, and it was recovered as a valuable mineral. The aforementioned results indicated that the easy-to-float minerals (sulfide minerals) can adhere to bubbles and move toward the center of the column along the radial direction.

Table 4. Mineral composition of the circulation middling and tailing (wt.%)

Samples	Nickel pyrite	Chalcopyrite	Vallerite	Pyrite	Iron oxide	Ophiolite	Olivine	Pyroxene	Biotite	Other minerals	Total
Tailing	0.35	0.12	1.04	4.07	7.72	62.87	5.84	10.50	2.38	5.11	100.00
Circulating middling	0.39	0.20	1.02	4.94	7.89	61.98	6.05	9.93	2.41	5.19	100.00

The size weight percentage and Ni grade of different size fractions of the circulating middling and tailing were tested and the results are shown in Fig. 3. It was found that, for four size fractions coarser than 21  $\mu\text{m}$ , the size weight percentage of the middling was higher than that of the tailing. Inversely in the case for the fractions smaller than 21  $\mu\text{m}$ . For the size fraction smaller than 11  $\mu\text{m}$ , the size percentage of the middling was higher than that of the tailing by 7.3 percentage points. Based on these results, it can be concluded that the particles are not classified according to their size in the three-phase systems of gas–solid–liquid in the cyclonic separation zone of the column.

The Ni grade was also analyzed on a particle size-by-size basis and the results are plotted as a histogram in Fig. 3. It is well-known that pentlandite has a significantly greater density than ophiolite which is one of the main gangue minerals. Therefore, pentlandite as well as other sulfide minerals with a higher density moves toward the wall of the column along the radial direction in the centrifugal force field generated in the cyclonic separation zone and is discharged as a tailing. However, as shown in Fig. 3, it is observed that the Ni grade of the circulating middling stream is higher than that of the tailing stream in nearly all of the size fractions. Therefore, it can be concluded that the particles are not separated according to their density in the cyclonic separation zone of the column.

Figures 4 and 5 show the size distribution of the main valuable metal sulfide minerals (pentlandite and chalcopyrite) in the circulating middling and tailing, respectively. The size of the pentlandite particles contained in the circulating middling stream is coarser than that in the tailing stream. The percentage of the particles smaller than 75  $\mu\text{m}$  is 94.69% for the tailing stream and 84.14% for the circulating middling stream. Especially for the four size fractions larger than 32  $\mu\text{m}$  ( $-53 +32 \mu\text{m}$ ,  $-75 +53 \mu\text{m}$ ,  $-90 +75 \mu\text{m}$ , and  $+90 \mu\text{m}$ ), the percentage of pentlandite particles in the circulating middling is higher than that in the tailing by 2.49, 1.75, 2.88, and 7.67 percentage points, respectively. The conditions in Fig. 5, describing the size distribution of chalcopyrite which has good floatability as pentlandite, are the same as



those in Fig. 4. In addition, it is noteworthy that nearly all of the chalcopyrite particles coarser than 53  $\mu\text{m}$  are concentrated in the circulating middling. These findings can be attributed to adhesion of bubbles to coarser particles with preferable floatability.

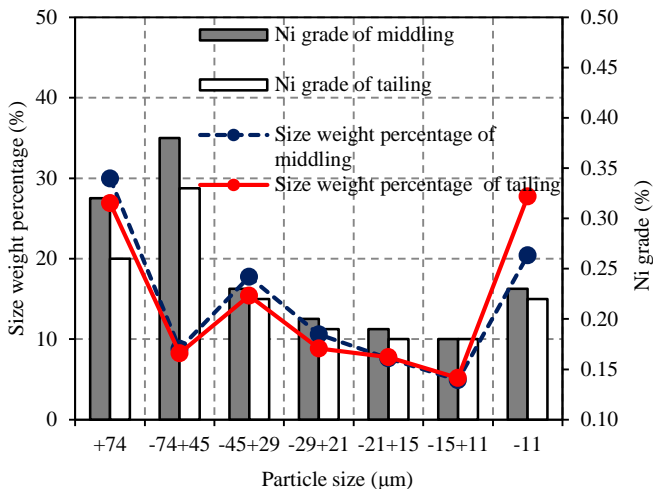


Fig. 3. Size analysis of circulating middling and tailing

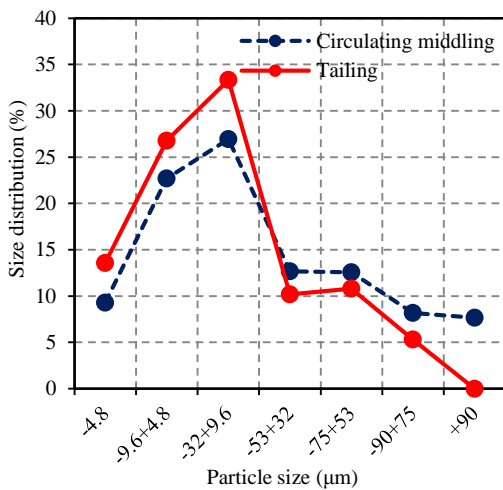


Fig. 4. Size distribution of pentlandite in circulating middling and tailing

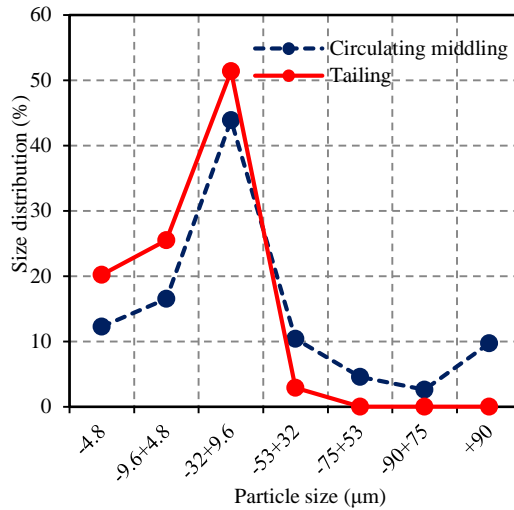


Fig. 5. Size distribution of chalcopyrite in circulating middling and tailing

## Conclusions

Under given conditions, a concentrate with Ni grade of 1.78% and recovery of 65.56% was obtained in the lab-scale cyclonic flotation column, while the Ni grade of the tailing was only 0.23%.

Unlike conventional cyclone device, separation achieved in the cyclonic zone of the column was not dependent on the particle size and density. If the particles have a better hydrophobic surface, even for those coarse and heavy particles, they will be captured by bubbles and move toward the center of the column. This mechanism might be responsible for the conclusions obtained from the comparative analysis of the circulating middling and tailing.

## Acknowledgment

This research was supported by the Fundamental Research Funds for the Central Universities of China (No. 2015XKMS039) and the National Nature Science Foundation of China (No. 51504254) for which the authors express their appreciation.

## References

- CAO Y. J., LI G. S., LIU J. T., ZHANG H. J., ZHAI X., 2012. *Removal of unburned carbon from fly ash using a cyclonic-static microbubble flotation*. The Journal of The Southern African Institute of Mining and Metallurgy, 112, 891-896.
- DAS A., MILLER J. D., 1996. *Swirl flow characteristics and froth phase features in air-sparged hydrocyclone flotation as revealed by X-ray CT analysis*. International Journal of Mineral Processing, 47(3),251-274.

- DEGNER V. R., PERSON P. L., 1991. *Leeds column performance evaluation*. Minerals Engineering, 4(7), 935-950.
- DEY S., PAUL G. M., PANI S., 2013. *Flotation behaviour of weathered coal in mechanical and column flotation cell*. Powder Technology, 246,689-694.
- GU L. P., YALCIN T., 2012. *Semi-batch flotation column*. Minerals Engineering, 26: 105-107.
- GUI X. H., LIU J. T., CAOY. J., CHENG G., ZHANG H. J., WANG Y. T., 2013. *Process intensification of fine coal separation using two-stage flotation column*. Journal of Central South University, 20(12), 3648-3659.
- GUI X., WANG Y., ZHANG H., LI S., 2014. *Effect of two-stage stirred pulp-mixing on coal flotation*. Physicochemical Problems of Mineral Processing, 50(1), 299-310.
- JAMESON G. J., 2010. *New directions in flotation machine design*. Minerals Engineering, 23(11-13), 835-841.
- LI G. S., CAO Y. J., LIU J. T., WANG D. P., 2012. *Cyclonic flotation column of siliceous phosphate ore*. International Journal of Mineral Processing, 110, 6-11.
- LI G. S., DENG L. J., LIU J. T., CAO Y. J., ZHANG H. J., RAN J.C., 2015. *A new technique for removing unburned carbon from coal fly ash at an industrial scale*. International Journal of Coal Preparation and Utilization, 35(5), 273-279.
- LIU A. R., GAO J. C., FAN M. Q., 2013. *Performance of an air-injected water-only cyclone for the separation of fine coal*. International Journal of Coal Preparation and Utilization, 33(5), 218-224.
- MILLER J. D., 1981. *An air-sparged hydrocyclone apparatus and method*. US, 4279743[P].
- NIWIADOMSKI M., HUPKA J., BOKOTKO R., MILLER J. D., 1999. *Recovery of coke fines from fly ash by air sparged hydrocyclone flotation*. Fuel, 78 (2), 161-168.
- VAN DEVENTER J. S. J., BURGER A. J., CLOETE F. L. D., 1988. *Intensification of flotation with an air-sparged hydrocyclone*. Journal of the South African Institute of Mining and Metallurgy, 88(10),325-332.
- YALAMANCHILI M. R., MILLER J. D., 1995. *Removal of insoluble slimes from potash ore by air-sparged hydrocyclone flotation*. Minerals engineering, 8(1),169-177.
- YALCIN T., 1995. *The effect of some design and operating parameters in the cyclo-column cell*. Minerals Engineering,8(3), 311-319.
- YANG D. C., BOZZATO P., FERRARA G.,2003. *Iron ore beneficiation with packed column jig*. Journal of Minerals and Materials Characterization and Engineering, 2(1), 43-51.
- YOON R. H., LUTTRELL G. H., ADEL G. T., Mankosa M.J.,1992. *The application of Microcel column flotation to fine coal cleaning*. Coal Preparation, 10(1-4),177-188.
- ZHANG H. J., LIU J. T., WANG Y. T., CAO Y. J., MA Z. L., LI X. B., 2013. *Cyclonic-static micro-bubble flotation column*. Minerals Engineering, 45, 1-3.
- ZHOU X. H., LIU J. T., 2007. *Particle residence time in column flotation based on cyclonic separation*. Journal of China University of Mining and Technology, 17(3), 349-353.