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OPTIMIZATION OF FINE ILMENITE FLOTATION PERFORMED IN A CYCLONIC-STATIC MICRO-BUBBLE FLOTATION COLUMN

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Abstract: A cyclonic-static micro-bubble flotation column was applied to upgrade fine ilmenite. The optimum parameters of flotation in the column were determined basing on the grade-recovery upgrading curve. A continuous pilot plant test was conducted using the optimum parameters during rougher-cleaner process. When compared with the optimized parameters of the industrial flotation machines, the cyclonic-static micro-bubble flotation column provides higher concentrate grade and recovery: 48.11% with a growth of 1.08 percentage points and 82.36% with an increment of 13.64 percentage points, respectively. Moreover, the flowsheet is simplified to two steps (rougher-cleaner) in the cyclonic-static micro-bubble flotation column from six steps (one rougher-two scavengers-three cleaners) in the flotation machines. Therefore, the cyclonic-static micro-bubble flotation column is an effective tool for fine ilmenite beneficiation.

Keywords: *cyclonic-static micro-bubble flotation column, fine ilmenite, optimum parameters, continuous flotation*

Introduction

Titanium is an important raw material for space industry, nautical engineering and medicine (Omidvar et al., 2012; Parker and Stec, 2012). China, with the largest titanium reserves, accounts for about 48% of the world's reserves (Li et al., 2007a). The Panzhihua and Xichang region in Chinese Sichuan Province is a cornucopia of titanium, where there are more than 90% of titanium reserves of China (Li et al., 2006; Wu et al., 2011). About 98% of titanium is present as ilmenite ($FeTiO_3$). The theoretical grade of TiO_2 in ilmenite is 52.63%. This ilmenite ore is refractory to flotation processing (Fan and Rowson, 2000; Zhou et al., 2006). Flotation machines

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are conventionally used for industrial flotation of ilmenite. However, the recovery is low and complicated flowsheets are used for the fine ilmenite fractions (Banisi and Finch, 2001; Rule and Anyimadu, 2007).

There is an interest in column flotation in mineral processing and industrial application (Honaker and Mohanty, 1996; Hulya and Ugur, 2012). In 1980, the first column was applied in Noranda's Mines Gaspe, which was shut down in 1983 (Dobby and Finch, 1991). The major disadvantages of flotation column were the mixing in the axis of the column, the blockage of spargers and problems posed by column height in installations. In recent decades, flotation column had directly driven some developments (e.g. in sparger systems), spawned derivatives (e.g. the packed column) and new concepts (e.g. the Jameson cell), for instance froth washing (Hasan and Hale, 2007). However, not all developments are accepted by the technology and market. One of the new approaches is the cyclonic-static micro-bubble flotation column (CSMC), commercially available as FCSMC, that is with additional F letter in the front of the acronym. It was invented by the China University of Mining and Technology. The characteristic innovation of the CSMC is multiple mineralization steps, including countercurrent mineralization, cyclone mineralization and pipe flow mineralization (Zhang et al., 2013; Gui et al., 2013). The CSMC has many advantages, such as higher concentration ratio and optimized flowsheet (Yianatos et al., 2005). Recently, the CSMC has been successfully used in flotation of metallic and non-metallic minerals. Cao et al. (2009) used the CSMC to separate copper-nickel sulfide. They obtained nickel concentrate with a grade of 8.21% and a recovery of 55.24%, compared to only 5.02% and 44.71%, respectively, when using flotation machines for the same ore.

In this work, a pilot plant test was carried out using the CSMC, to probe the optimum parameters of the fine ilmenite flotation in rougher and cleaner process. The 72 h continuous test was then performed under the optimum parameters. The feed, concentrate and tailings were sized to several fractions and each of them was assayed to calculate the by-size grade and recovery of concentrate in continuous pilot plant test.

Experimental

Materials

The ilmenite ore was supplied by the Pan Steel Mining Company of Sichuan in China. The valuable minerals in the ore were ilmenite and titanomagnetite while the gangue minerals were titanaugite, plagioclase, olivine, and kaersutite. The chemical analysis of the ore performed by the X-ray fluorescence (XRF) method is presented in Table 1. The ore liberation degree was determined by the mineral liberation analyzer (MLA). As shown in Fig. 1, most of the ilmenite was liberated.

The ilmenite ore was screened using the 154, 100 and 74 μm sieves and then the fine size fraction (–74 μm) was analyzed by a cyclosizer (cyclonic size analysis instrument) having 38, 30 and 20 μm sieves. The obtained results are shown in Table 2. The grade of +74 μm size fraction is lower (12.37% TiO_2) than the ilmenite ore grade (20.26% TiO_2), which indicates that this fraction is hard to recovery. For the size from 30 to 74 μm, the yield is 51.45% and the TiO_2 distribution is 63.39%. The yield of –20 μm size fraction is only 3.70% due to pre-desliming. Therefore, the ilmenite ore is difficult to process because of non-uniform distribution in size. The TiO_2 grade and distribution at the size of –74+20 μm indicates that ilmenite is expected to be liberated from its associated gangues.

Table 1. Chemical composition of ilmenite ore by XRF

	Fe_T	FeO	TiO_2	Fe_2O_3	SiO_2	Al_2O_3	MgO	CaO	Others
Content (%)	7.61	22.95	20.52	6.40	22.68	5.98	7.34	5.28	1.24

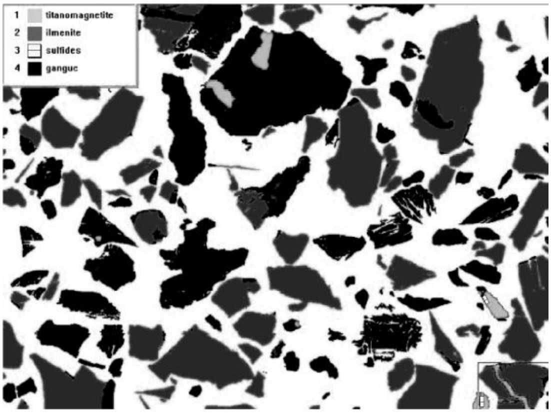

Fig. 1. Mineralogical and liberation analyses of ilmenite ore by MLA technique

Table 2. Particle size distributions of the ilmenite ore

Particle size /μm	Yield (%)		Grade (TiO_2 %)		TiO_2 Distribution (%)	
	Individual	Accumulation	Individual	Accumulation	Individual	Accumulation
+154	9.53	9.53	4.73	4.73	2.22	2.22
–154+100	7.71	17.24	12.51	8.21	4.76	6.98
–100+74	18.86	36.10	16.17	12.37	15.05	22.03
–74+38	42.39	78.49	24.01	18.66	50.24	72.27
–38+30	9.06	87.55	29.41	19.77	13.15	85.42
–30+20	8.75	96.30	26.60	20.39	11.49	96.91
–20	3.70	100.00	16.90	20.26	3.09	100.00
Total	100.00		20.26		100.00	

Flotation reagents

H_2SO_4, MOH and diesel oil (D_O) were used as pH regulator, collector and auxiliary collector, respectively. H_2SO_4 (diluted to 25 wt%) was used for altering pH value of the pulp. MOH was dissolved in water to 3 wt% at 70 ^oC. MOH collector was invented by the Chinese Central South University (Zhu et al., 2007). It is a commercial collector, widely used in fine ilmenite upgrading. The detail composition of the MOH was neither provided by the company nor reported in literature. In order to get more information about MOH, a Fourier transform infrared spectrometer (FTIR) and atmospheric solids analysis probe (Model ASAP^{TM}, IonSense, USA)/time of flight-mass spectrometer (ASAP/TOF-MS) were applied.

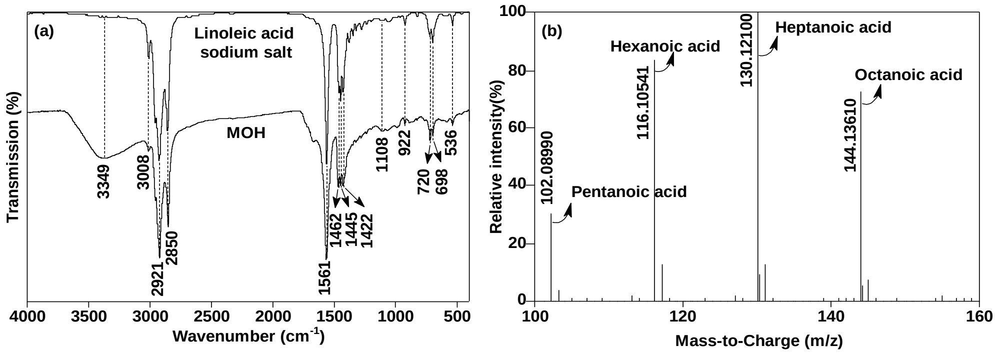

Fig. 2. FTIR (a) and ASAP/TOF-MS (b) analyses of the MOH

As Figure 2 shows, the spectrum of MOH is matched excellently with that of sodium salt of linoleic acid (peak at 3349 cm^{-1} was assigned to water). With ASAP/TOF-MS analysis, four compounds were identified in MOH, which probably were pentanoic acid, hexanoic acid, heptanoic acid and octanoic acid. Therefore, MOH seems to be a synthetic mixture of fatty acids or their salts. The auxiliary collector was industrial diesel oil (D_O), which has a synergistic effect with MOH. It can reduce the dosage of MOH and improves the flotation rate.

Flotation devices and tests

CSMC and its separation principles

As Figure 3 displays, the typical CSMC is composed of the column separation zone, cyclone separation zone and pipe flow mineralized zone. The sieve plates are packed in the column separation zone to improve the beneficiation efficiency, because the sieve plates provide the necessary low-turbulence environment, decrease the axial mixing, and increase the collision and adhesion effectively between bubbles and particles (Zhang et al., 2009; Kawatra and Eisele, 1995; Xia et al., 2006; Eisele and Kawatra, 2007). The CSMC has three input streams (feed, air and wash water) and two output streams (concentrate and tailings).

The separation principles of the CSMC was reported in literature (Zhang et al., 2013; Liu et al., 2013; Li et al., 2009; Yan et al., 2012; Deng et al., 2013). The circulation pressure (C_P) of circulating pump was measured by the pressure gaugue and adjusted by converter. The aeration quantity (A_Q) in pipe flow mineralized zone was modified by a flow meter.

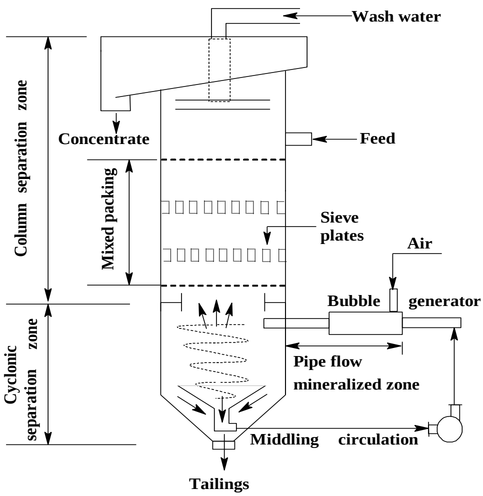

Fig. 3. Schematic representation of CSMC

Previous laboratory tests using the CSMC

The previous laboratory tests provide data for the pilot plant design of the best-suited circuit. According to previous laboratory tests (Table 3), the rougher-cleaner flotation flowsheet was chosen for the pilot plant test. The grade of the final cleaner concentrate is dependent on the grade of the rougher concentrate. In order to reach the optimum cleaner grade, it is necessary to keep the rougher grade at a predetermined value. As shown in Table 3, TiO_2 grade of rougher concentrate in pilot plant tests should reach 36–38% and rougher concentrate recovery should be as high as possible. Therefore, the quality of cleaner concentrate (the grade of TiO_2 is over 47.50%) was guaranteed.

Table 3. Previous laboratory closed-circuit flotation tests. Rougher process was carried out in CSMC of diameter 100 mm and height 2000 mm while cleaner process in CSMC of diameter 50 mm and height 2000 mm

	Products	Yield (%)	Grade (TiO_2, %)	Recovery (TiO_2, %)
Rougher-cleaner process	Cleaner concentrate	35.40	47.98	83.91
	Rougher tailings	64.60	5.04	16.09
	Rougher concentrate		36.89	
	Cleaner tailings		19.56	
	Ilmenite ore	100.00	20.24	100.00

Pilot plant tests using the CSMC

As described in Fig. 4, the pilot plant tests system consists of mixture section, separation and automatic control section. The flotation tests were optimum parameters exploration in rougher and cleaner process, and continuous pilot plant test.

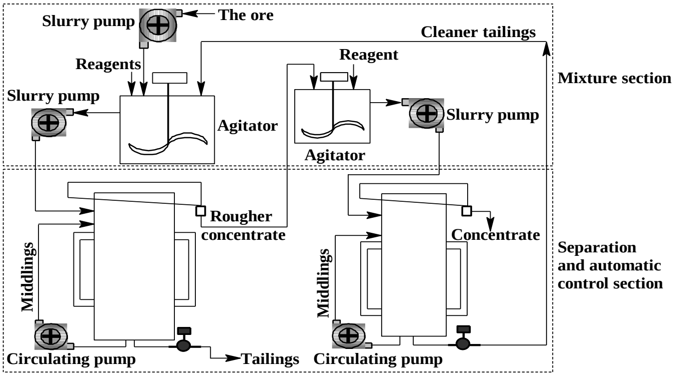

Fig. 4. Schematic diagram of pilot plant test. Rougher flotation was performed in a CSMC of diameter 350 mm and height 4000 mm while cleaner flotation in a CSMC of diameter 200 mm and height 4000 mm

In rougher process, the cleaner CSMC system was not operated. The optimum pH value, MOH dosage, ratio of solids to liquid phase (S/L ratio), D_O dosage, C_P and A_Q value were considered using one CSMC. The feed, concentrate and tailings were acquired after the system running steadily for 3 h. On each process variable, the experiment was repeated at least three times. The concentrate grade and recovery was analyzed with the grade-recovery curve (Neethling and Cilliers, 2008; Santana et al., 2008; Drzymala, 2007) and 95% confidence interval (*CI*) in both concentrate grade and recovery expressed as error bars in the y-direction and x-direction respectively. The *CI* can be calculated separately for each timed mean grade and recovery. The following formula is used for calculating *CI* (Napier-Munn, 2012):

$$CI = \pm \frac{t_\alpha}{\sqrt{n}} s \tag{1}$$

where t_α is the *t*-value for a 2-sided confidence level of 100(1–α) % with n–1 degrees of freedom, the *t*-value can be obtained from a table of the *t*-distribution or from the Excel function = TINV (α, n–1); s is the standard deviation of the sample, and n is the number of replicates in the sample. In this paper, $1 - \alpha = 95\%$, $n = 3$, $t_\alpha = 4.30$, s is a variable.

The cleaner process was operated on the optimal rougher parameters. It's worth noting that the cleaner feed was the rougher concentrate, and the cleaner tailings was recycled (Fig. 4). The optimal pH, C_P and A_Q value were explored.

The 72 h continuous test was operated with two CSMCs, due to the optimized conditions in rougher-cleaner flowsheet. Flotation tests in industrial flotation machines proceeded with one rougher-two scavengers-three cleaners. The type of the flotation machines is GF-8/JJF-8, which consisted of GF-8 flotation machine (8 m^3 of effective volume, for adsorbing slurry) and JJF-8 flotation machine (8 m^3 of effective volume, for agitation). The GF-8/JJF-8 flotation machines are self-aerating and mechanical agitation flotation devices.

In the continuous test, the feed, concentrate and tailings were collected from the CSMC and GF-8/JJF-8 flotation machines at the same time to evaluate the beneficiation index. The particle size distributions of the feed, concentrate and tailings were analyzed to calculate the by-size grade and recovery of concentrate in CSMC and flotation machines by the following equation:

$$F(i) = \frac{\varepsilon\,\varepsilon(i)}{\gamma\,\gamma(i)} \quad (2)$$

where $F(i)$ is the concentrate recovery of size i fraction (%), ε is the recovery of concentrate, $\varepsilon(i)$ is the metal distribution rate of size i fraction in concentrate, γ is the recovery of the ilmenite ore, $\gamma(i)$ is the metal distribution rate of size i fraction in the ilmenite ore.

Results and discussion

Optimum parameters in rougher process

Figure 5 plots the effect of the parameters on the mean grade-recovery in rougher process using one CSMC and 95% *CI* on the mean concentrate grade-recovery. The H_2SO_4 was chosen as the pH regulator to remove the hydration shell, deslime on the mineral surface and enhance the concentrate grade. Figure 5a shows pH value effect on concentrate grade and recovery when the other operating parameters were as follows: *S/L* ratio = 0.67, MOH = 2000 g/Mg, D_O = 500 g/Mg, C_P = 0.21 MPa, A_Q = 0.8 $m^3/(m^2{\cdot}min)$. With the pH decreases, the concentrate recovery decreases while the concentrate grade increases. The optimized pH value is 5.0, while the concentrate grade and recovery is 37.81% and 80.98%, respectively. MOH dosage is a vital flotation parameter in the flotation experiments. Suitable MOH dosage can enhance the stability of mineral particles and bubbles. In Fig. 5b (*S/L* ratio = 0.67, pH value = 5.0, D_O = 500 g/Mg, C_P = 0.21 MPa, A_Q = 0.8 $m^3/(m^2{\cdot}min)$), as the MOH dosage increases, the concentrate grade declines and the recovery increases. Comprehensive consideration, 2800 g/Mg is selected as the optimal MOH dosage. The D_O was used to reinforce the flotation effect and strengthen the flotation rate. As exhibited in Fig. 5d (*S/L* ratio 0.67, pH value 5.0, MOH 2800 g/Mg, C_P 0.21 MPa and A_Q 0.8 ($m^3/m^2{\cdot}min$)), 700 g/Mg is determined as the optimum dosage, and the concentrate grade is 36.73%, with a recovery of 84.34%. The effect of the *S/L* ratio on the flotation is presented in Fig. 5c (pH value 5.0, MOH 2800 g/Mg, D_O 700 g/Mg, C_P 0.21 MPa and A_Q 0.8 ($m^3/m^2 \cdot min$)). Assay evidence indicated that the *S/L* ratio of 0.82 is established as the optimum condition.

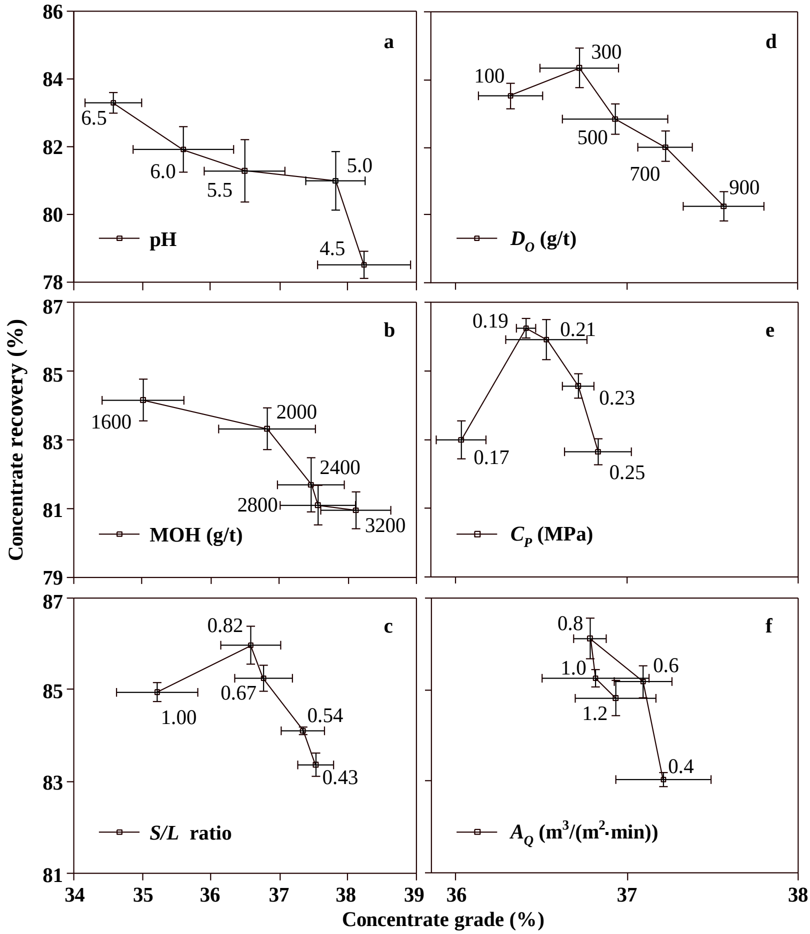

Fig. 5. Effect of the parameters on grade-recovery curves in rougher process using one CSMC, including 95% *CI* on the mean concentrate grade-recovery

The C_P has a critical influence on separation effect. In this section, other operating parameters were constant: *S/L* ratio 0.82, pH value 5.0, MOH 2800 g/Mg, D_O 700 g/Mg and A_Q 0.8 $m^3/(m^2 \cdot min)$. As shown in Fig. 5e, when the C_P is 0.19 MPa, the concentrate recovery (86.24%) is higher than the others, and the concentrate grade is similar with the others. Herein, the optimum C_P is 0.19 MPa. The A_Q has an obvious influence on the particles-bubbles contact and bubbles quantities (Asplin et al., 1998). As exhibited in Fig. 5f (*S/L* ratio 0.82, pH value 5.0, MOH 2800 g/Mg, D_O 700 g/Mg and C_P 0.19 MPa), when the A_Q is 0.8 $m^3/(m^2 \cdot min)$, the concentrate grade is 36.79%

and the recovery is 86.10%. Under this condition, the concentrate recovery is optimal even the concentrate grade has a slight decrease.

The 95% *CI* on the mean rougher concentrate grade-recovery (Fig. 5) indicates that there is a smaller uncertainty at each point, in both concentrate grade and recovery. This is a result of high stability of system and carefully control precision. The standard deviations for both concentrate grade (in the range < 1%) and recovery (in the range < 1%) are normal, so smaller *CI* can be achieved and good data repeatability obtained.

Optimum parameters in cleaner process

The principal role of cleaner process is to enhance the concentrate grade. This work investigates the influence of pH, C_P and A_Q value on the concentrate grade and recovery in cleaner. Results show that the optimal parameters of cleaner are follows: pH value 4.0, C_P 0.16 MPa and A_Q 0.5 $m^3/(m^2 \cdot min)$. Under these conditions, the cleaner concentrate grade is 48.18% with recovery of 82.25%.

Continuous pilot plant test using two CSMCs

The closed-circuit continuous pilot plant flotation test is to provide continuous operating data and compare equipment performance. Under the optimized parameters, the total reagent dosages in flotation machines are similar to that in CSMC. The detailed reagent dosages in flotation machines are: in one rougher, pH 5.0, MOH dosage 2300 g/Mg, D_O dosage 500 g/Mg; in one cleaner, pH 4.0; in one scavenger, MOH dosage 500 g/Mg, D_O dosage 180–200 g/Mg. No reagents were added in other steps.

Table 4. Comparison of separation system between FCSMC and FM for the ilmenite ore

Separation system	Grade (TiO_2, %)			Concentrate yield (%)	TiO_2 recovery in concentrate (%)
	Feed	Concentrate	Tailings		
FCSMC	20.16	48.11	5.43	34.51	82.36
FM	20.13	47.03	8.92	29.41	68.72

As Table 4 displays, the CSMC concentrate grade and recovery is 48.11% and 82.36%, respectively. Compared with the flotation machines, the CSMC concentrate grade and recovery increases 1.08 and 13.64 percentage points, respectively. Moreover, the flowsheet is simplified from six steps in flotation machines to two steps in the CSMC.

By-size grade and recovery of concentrate in continuous pilot plant test

Figure 6 shows the by-size grade and recovery of concentrate in the CSMC and flotation machines. As the particle size decreases, the by-size concentrate grade increases (+38 μm) then decreases (–38 μm), and the by-size concentrate recovery increases in both CSMC and flotation machines. However, the CSMC achieves significant concentrate grade and recovery for all size fractions expect for +74 μm size fraction. In the –74+38 μm fraction, the CSMC concentrate recovery is 84.87%, while the CSMC concentrate grade is 49.33%. Nevertheless, the flotation machines concentrate grade and recovery is only 48.51% and 69.67%, respectively. In the size range –38+30 μm fraction, the CSMC concentrate grade increases by 2.32% and concentrate recovery increases by 14.33%, compared with flotation machines. In the size range –30+20 μm fraction, the CSMC obtained concentrate with a grade of 48.72% and a recovery of 92.71%, compared to only 44.40% and 80.09%, respectively, when using flotation machines for the same size fraction. For the fraction –20 μm, the CSMC concentrate recovery is the highest, however, the grade of this size fraction is lower relative to other size fractions, which may result from gangue entrainment.

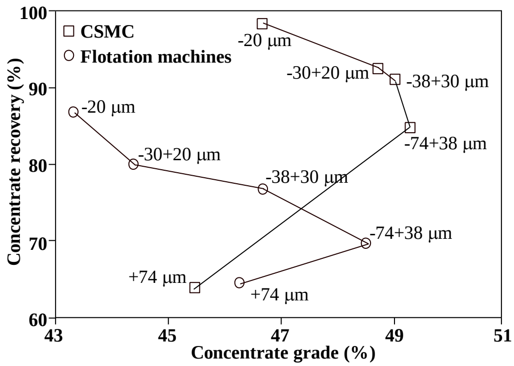

Fig. 6. By-size grade and recovery of concentrate curve with CSMC and flotation machines

Therefore, the CSMC is a more effective device for fine ilmenite beneficiation, compared with flotation machines. The kinetic analysis of bubbles and particles in cyclone zone on the radial direction also gives the same conclusion (Li et al., 2012; Zhou and Liu, 2007).

Conclusion

This work exploits the CSMC to process fine ilmenite ore. The optimum parameters of the ilmenite flotation in rougher and cleaner process are obtained basing on the mean grade-recovery curve. A continuous pilot plant test is conducted in rougher-cleaner process using two CSMC's. The by-size grade and recovery of concentrate is analyzed. The conclusions drawn from this study are as follows.

- The optimal conditions in rougher are follows: S/L ratio 0.82, pH 5.0, MOH dosage 2800 g/Mg, D_o dosage 700 g/Mg, C_P 0.19 MPa, A_Q 0.8 $m^3/(m^2 \cdot min)$. The optimal conditions in cleaner are follows: pH 4.0, C_P 0.16 MPa, and A_Q 0.5 $m^3/(m^2 \cdot min)$.

- Compared with flotation machines, the cleaner concentrate grade of the CSMC is 48.11% with an improvement of 1.08 percentage points, while the concentrate recovery is 82.36% with 13.64 percentage points increment in continuous pilot plant test. The flowsheet is simplified from six steps (one rougher-two scavengers-three cleaners) in flotation machines to two steps (rougher-cleaner) in the CSMC. Results show the CSMC is an effective beneficiation device to recovery –74 μm size fraction.

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