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Froth stabilisation using nanoparticles in mineral flotation

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Abstract: In this study, three kinds of nanoparticles (SiO_2, Fe_2O_3, and Al_2O_3) were used in the flotation of a sulphide ore to investigate the effects of nanoparticles on the froth stability and the flotation performance. The dynamic froth stability factor and the maximum froth depth were measured by using a non-overflowing flotation cell under various flotation conditions. The results were also related to the separation selectivity and efficiency of the flotation. The experimental results showed that the dynamic froth stability factor and the maximum froth depth can be increased 1.2-2 fold by using the Al_2O_3 nanoparticles. These increments led to significant improve in the froth recovery. In terms of the froth stability and the flotation performance, the Al_2O_3 nanomaterial was the best, followed by Fe_2O_3 and SiO_2. In addition, the flotation recovery increased from 83 to 91%, and the grade of the concentrate increased from 44 to 60% by using the Al_2O_3 nanoparticles.

Keywords: nanoparticles, flotation froth, sulphide ore, bubble size, froth stability

1. Introduction

Structure and stability of the flotation froths can be determined by many sub-processes occurring in froth phase of the mechanical flotation cells such as bubble coalescence in froth, drainage of liquid film between bubbles, bubble bursting at froth surface etc. Since some of hydrophobic mineral particles can detach from bubbles due to coalescence of bubbles in the froth or the bubble bursting at the froth surface, each of these sub-processes is important for flotation recovery. The deeper froths can be used to produce the higher quality concentrates in the flotation by obtaining a desired level of the froth stability. Therefore, it is commonly accepted that a froth of proper stability is essential to obtain high flotation performance (Ross, 1997; Tao et al., 2000; Schwarz and Grano, 2005; Gupta et al., 2007; Zanin et al., 2009; Farrokhpay, 2011).

The froth stability can be defined as the ability of bubbles in froth to resist coalescence and bursting (Farrokhpay, 2011). On the other hand, the froth stability is a result of the flotation conditions, and therefore, it can be considered as a dependent variable of the flotation process. Mostly, some of the flotation conditions are changed in order to obtain a desired (or a required) level of the froth stability. For instance, type or dosage of a frother used in a plant is changed to provide finer bubbles and/or more stable froth (Laskowski, 1998; Cho and Laskowski, 2002; Grau et al., 2005, Harvey, et al., 2005; Nguyen et al., 2006; Gupta et al., 2007; Ata, 2012). On the other hand, changing physical and/or chemical conditions used in the flotation affects not only the froth stability but also the flotation performance. In many cases, the flotation performance can be adversely affected. For these reasons, it is clear that a new approach is necessary to control the froth stability without changing the flotation conditions.

Literature reviews show that many experimental studies have been performed to investigate not only the usability of the nanoparticles as foam/froth stabilisers but also to define mechanism of the foam/froth stabilisation with the nanoparticles. The results of the these experimental studies reported in literature reveal that the nanoparticles can be used to obtain more stable froths in the flotation (Binks, 2002; Paunov et al., 2002; Ata et al., 2002, 2003; Du et al, 2003; Dickinson et al., 2004; Binks and Horozov,

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2005; Horozov, 2008; Liu et al., 2010; Zech et al., 2012; Bournival et al., 2014; Cilek and Karaca, 2015). The nanoparticles have been used as foam/emulsion stabiliser in majority of these studies. Only limited studies have been performed in single mineral flotation (Zech et al., 2012; Cilek and Karaca, 2015). Therefore, using of the nanoparticles as a froth stabiliser in the flotation of a real ore is very important. Stability of the flotation froths becomes controllable factor, and it can be adjusted easily to the desired level during the flotation. Thus, the separation efficiency and selectivity of the flotation process could be enhanced.

In this study, the flotation tests were performed with a sulphide ore sample to determine the effects of three kinds of the nanoparticles on the froth stability. It is known that the detachment and attachment processes in the froth occur continuously, and therefore, the selective re-attachment of the detached hydrophobic minerals from the coalesced bubbles can be caused to improve the flotation performance (Ata et al., 2002; 2003). It is expected that the re-attachment process can be more effective in the nanoparticle stabilised froths. Therefore, efforts were also made to determine and interpret potential improvement in selectivity and efficiency of the flotation by using the nanoparticles.

2. Experimental

2.1 Materials

A sulphide ore sample was a copper-iron sulphide ore containing 1.32-1.63% Cu and 4.18-5.41% Fe. Main minerals of the ore were chalcopyrite associated with pyrite in a non-sulphide gangue. Potassium amyl xhantate (Aero 350) was supplied from Cytec Industries, USA. Sodium hydroxide and sodium silicate (analytical grades) were purchased from Sigma-Aldrich. Three nanomaterials of high purity (>99%) (SiO_2; 15-20 nm, α-Fe_2O_3; 20-40 nm and γ-Al_2O_3; 20 nm) were purchased from SkySpring Nanomaterials Inc., USA. The ion concentrations of the tap water used for all experiments contained Ca^{2+}: 89.43 mg/dm^3; Mg^{2+}: 28.23 mg/dm^3; Na^+ : 6.86 mg/dm^3; K^+ : 4.01 mg/dm^3; Cl^-: 5.77 mg/dm^3; and HCO^{3-}: 445.3 mg/dm^3.

2.2 Methods

Prior to the systematic flotation tests, the preliminary experiments were conducted without the nanoparticles to determine the flotation conditions of the ore. These conditions were kept constant in the systematic tests. In each test, timed increments of the flotation concentrates were collected at periods ending at 0.25, 0.5, 1, and 2 min to determine the overall flotation rate constant (k) together with the rate constant of the pulp zone (k_p). The froth recovery (R_f) was calculated as the total rate of transfer from the pulp to the concentrate divided by the rate of transfer from the pulp to the froth phase (Vera et al., 1999; Mathe et al., 2000; Vera et al., 2002).

$$R_f = k/k_p \,. \tag{1}$$

In order to determine the flotation rate constant, the time recovery data obtained from the kinetic tests were fitted to the modified first-order rate equation (Vera et al., 1999; Wills and Napier-Munn, 2006).

$$R = RI\,[1\text{-}\exp(\text{-}kt)] \tag{2}$$

where, R is the overall flotation recovery at time t (min), RI and k are the ultimate recovery and the overall rate constant (min^{-1}), respectively.

It is known that the froth stability can be defined quantitatively by the dynamic froth stability factor (Σ, s). Several methods were proposed to determine the dynamic froth stability factor or the froth characteristics in the literature (Ata et al., 2002, 2003; Barian et al., 2003, 2005; Du et al., 2003; Neetling et al., 2003; Dickinson et al., 2004; Bailey, et al., 2005; Binks and Horozov, 2005; Schwarz and Grano, 2005; Ata et al., 2006; Gupta et al., 2007; Stevenson, 2007; Giribabu et al., 2008; Farrokhpay, 2011). Barbian et al. (2003) used a specially constructed non-overflowing cell to measure the dynamic froth stability in their experiments. Similarly, a non-overflowing cell made of glass was used to determine the maximum froth depth using the Denver flotation machine in this study (Fig. 1). The cross-sectional dimensions of the cell were 11.5×15 cm. These tests were performed under same flotation conditions to those used in the kinetic flotation tests by using standard cells. The air at a fixed flow rate was introduced into the modified cell, and at the end of a period of time a constant depth of the froth was reached, which means the bursting and growth rates of the bubbles are equal. During each test, two or three photos were taken from the cell wall when the constant froth depth was reached. These photos were scaled and used to measure the maximum froth depth (F_{Dmax}, cm). The dynamic froth stability factor was calculated using the equation by Bikerman (1973):

$$\Sigma = (A \cdot F_{D_{\max}})/Q_{\mathrm{a}} \tag{3}$$

where Q_a is the air flow rate (cm^3/s), and A is the cross-sectional area of the modified flotation cell (cm^2).

The bulk flotation method was used in the flotation of the ore. The percentage of solids by weight in the pulp was 20% for all flotation experiments. In order to perform the bulk flotation of the sulphide minerals in the ore (chalcopyrite and pyrite), natural pH of the pulp (7.26-7.53) was used, and therefore, the pulp pH did not adjusted to a specific value. The prepared pulp in the cell was agitated for a few minutes, then Aero 350 as the collector at a dosage of 75 g/Mg was added, and the pulp was conditioned for a further 10 min. As a frother, a polyglycol ether (Dow Froth 250) was used in all tests.

Three different glass cells were used to obtain a constant pulp level with different froth depths (20, 30, and 40 mm) in the kinetic flotation tests. The air flow rates (Q_{a1} and Q_{a2}) were controlled with rotameters. The froth depth was kept constant by adding of a frother solution (of same pH value and concentration to that of the pulp in the cell) to the pulp zone by means of an on-off valve activating by a pulp level sensor.

Fig. 1. Non-overflowing glass cell used to determine the maximum froth depth

A 0.25 g/dm^3 of a nanoparticle suspension was prepared for the flotation tests. A required amount of each nanoparticle type was dispersed in sodium silicate solution of 25 mg/dm^3 (of the predetermined pH value and frother concentration) by sonicating the suspension for 30 min by using an ultrasonic probe (Bandelin Sonoplus HD 2200, 20 kHz, 0.2 kW). In order to transfer the nanoparticles into the froth, the following method was found suitable. During each flotation test, a secondary air (Q_{a2}) was injected trough an air sparger placed in a vessel filled with the nanoparticle suspension to occur supplementary froth (or foam), which overflowed into the cell froth zone. This vessel was mounted to the little above of the cell overflow level. Thus, the nanoparticles were transferred to the froth by means of the liquid film between the bubbles of the supplementary froth. It must be noted that the air flow rate (Q_a) of each test was sum of standard air flow rate (Q_{a1}) fed into the cell with the secondary air flow rate (Q_{a2}). In addition, the bubble size distribution was represented by the Sauter-mean bubble diameter (d_{32}), which was determined by measuring at least 300 bubbles for each test. Further details are given in Cilek and Karaca (2015).

3. Results and discussion

3.1 Effect of the nanoparticles on the froth stability

Fig. 2 shows effects of the nanoparticles on the maximum froth depth and the dynamic froth stability factor determined from the flotation tests using the non-overflowing cell. As can be seen from Fig. 2a, a monotonic increase or decrease of the maximum froth depth with the increasing the air flow rates was not observed in the flotation with various nanoparticles. However, the effect of the nanoparticles on the maximum froth depth was relatively pronounced (up to 5 cm or 62.5%). Binks and Horozov (2005) reported that the nanoparticles are effective foaming agents of air and water, foams that are completely stable to collapse, coalescence, and disproportionation can be prepared. Their results showed that in the case of the particle-stabilized foam drainage takes place relatively slowly and is over after several hours (the froth stability factor). Even if these results were obtained for two-phase system, they showed that similar improvements in the foam stability can be obtained in the froth stability by using the nanoparticles.

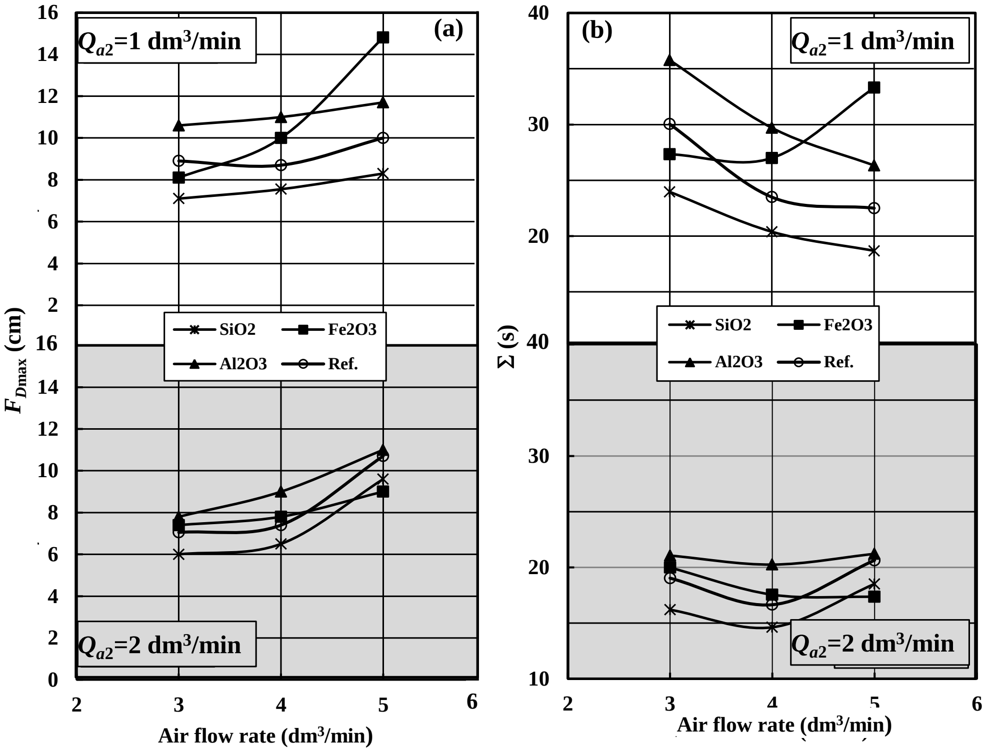

Fig. 2. Maximum froth depth (a) and dynamic froth stability factor (b) as function of air flow rate and nanoparticles. Ref.: Results of the flotation test performed without the nanoparticles

The dynamic froth stability factor consistently decreased with the increasing the air flow rate as expected (Fig. 2b). Similar trends in the dynamic stability shown in Fig. 2b were reported by Barbian et al. (2003; 2005) for the Platinum Group Metal ore. Even if the nanoparticles did not change this trend, it was observed that they led to increase the dynamic froth stability factor.

Effects of the nanoparticles on the Sauter-mean bubble diameter, d_{32}, determined from the flotation tests as a function of air flow rate and the froth depth are given in Fig. 3. The reference d_{32} determined from the tests performed without nanoparticles are also given in Fig. 3. As expected, the bubble size increased with the increasing the froth depth and the air flow rate in the tests performed with and without the nanoparticles. An interesting result that can be seen in Fig. 3 is that using of the nanoparticles showed an insignificant effect on the d_{32} at the low air flow rate and the shallow froth. As other investigators have also highlighted, it is expected that the deep froth increases the froth residence time (Fig. 2b), and the rate of drainage and, thus the rates of the coalescence and bursting increase in the froth phase (Tao et al., 2000; Ata et al., 2002, 2003; Harvey et al., 2005; Gupta et al., 2007). Contrarily to this, the experimental results presented in Fig. 3 suggest that the d_{32} or the rate of the bubble coalescence decreased significantly with the nanoparticles at the deep froths and the high air flow rates. In other words, even though the large bubbles appeared due to coalescence at these conditions, a significant number of small bubbles presented in the froth phase by adding of the supplementary froth carrying the nanoparticles. It must be noted that the differences between of the Sauter-mean bubble sizes obtained from the tests conducted with and without the supplementary froth for all cases were found to change from 0.42 to 0.89 mm, respectively. Therefore, the results obtained at the shallow froths are attributed to the use of the supplementary froth which contains the small bubbles with and without the nanoparticles.

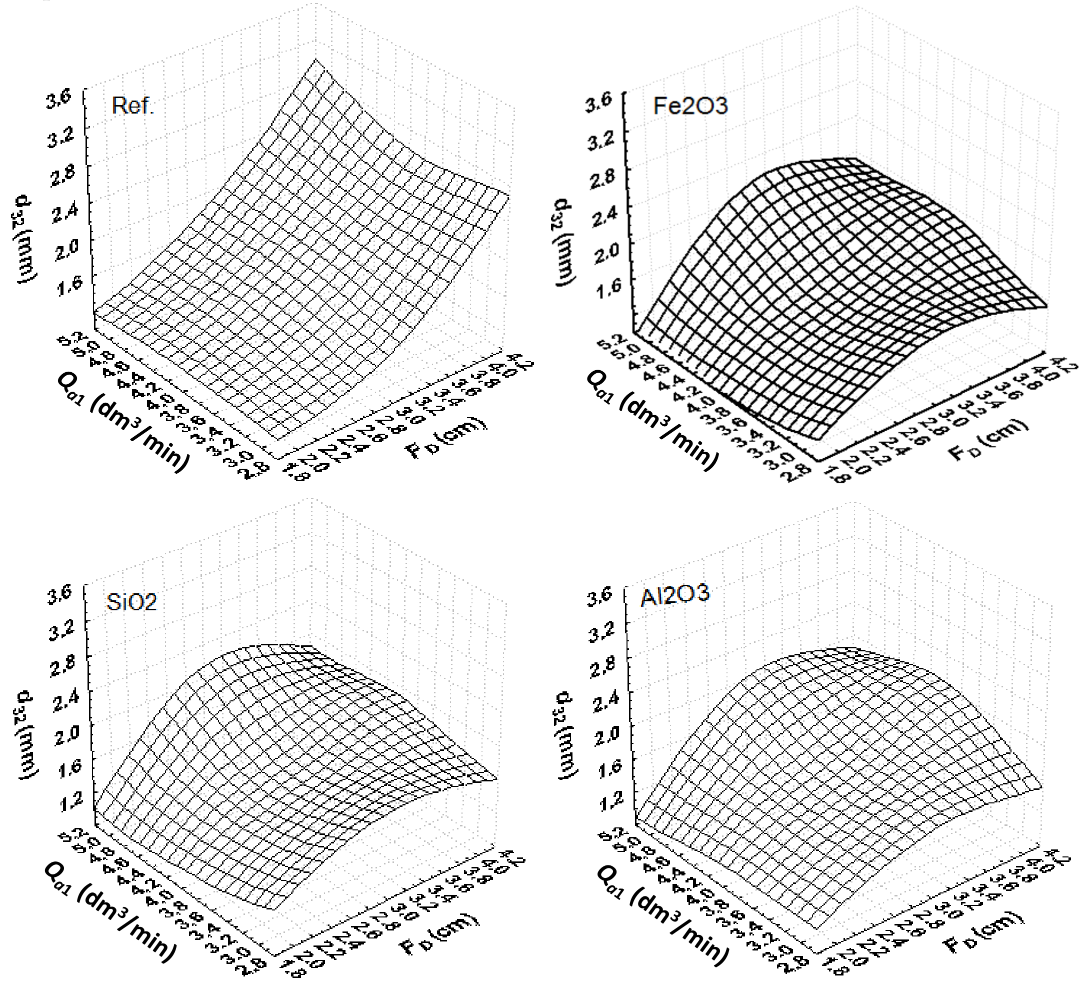

Fig. 3. Effect of the nanoparticles on the Sauter-mean bubble size

3.2 Effect of the nanoparticles on the flotation performance

Fig. 4 shows the experimental results of the bulk flotation recovery with and without the nanoparticles. In general, the flotation recovery decreases at the deep froths. This is due to the fact that the froth depth has two counteracting effects on the flotation performance. The deep froths cause more drainage of the inter-bubble liquid non-selectively carrying the fine particles (hydrophobic or hydrophilic), increasing the quality of the flotation concentrates. On the other hand, the deep froths cause to increase the froth residence time (the froth stability factor), increasing the coalescence rate of the mineralized bubbles. Since some of the hydrophobic mineral particles detach from the coalesced bubbles and some of these particles can drain back to the pulp phase, the flotation recovery decreases (Ross, 1997; Tao et al., 2000, Vera et al., 2002).

The results presented in Fig. 4 show that the bulk flotation recovery seemed to have the tendency to decrease slightly with the deep froths even though by using the SiO_2 and Fe_2O_3 nanoparticles. Contrary to these, using of the Al_2O_3 nanoparticles provides an increase up to 15% in the recovery of the bulk concentrate. Regarding the effect of the nanoparticles on the dynamic froth stability and the maximum froth depth, it may be expected that the following mechanisms enhance the flotation recovery. Since the Al_2O_3 nanoparticle stabilised froth (the supplementary froth) is added into the froth phase during each test, the sufficient bubble surface area for attachment in the froth phase is provided, and therefore, the detached sulphide minerals re-attach to the surfaces of the nanoparticle stabilised bubbles. In addition to this, it would be expected that the entrained sulphide minerals (small sized) in the froth phase also attach selectively to the surfaces of these bubbles, instead of the drainage with together the entrained gangue minerals into the pulp phase.

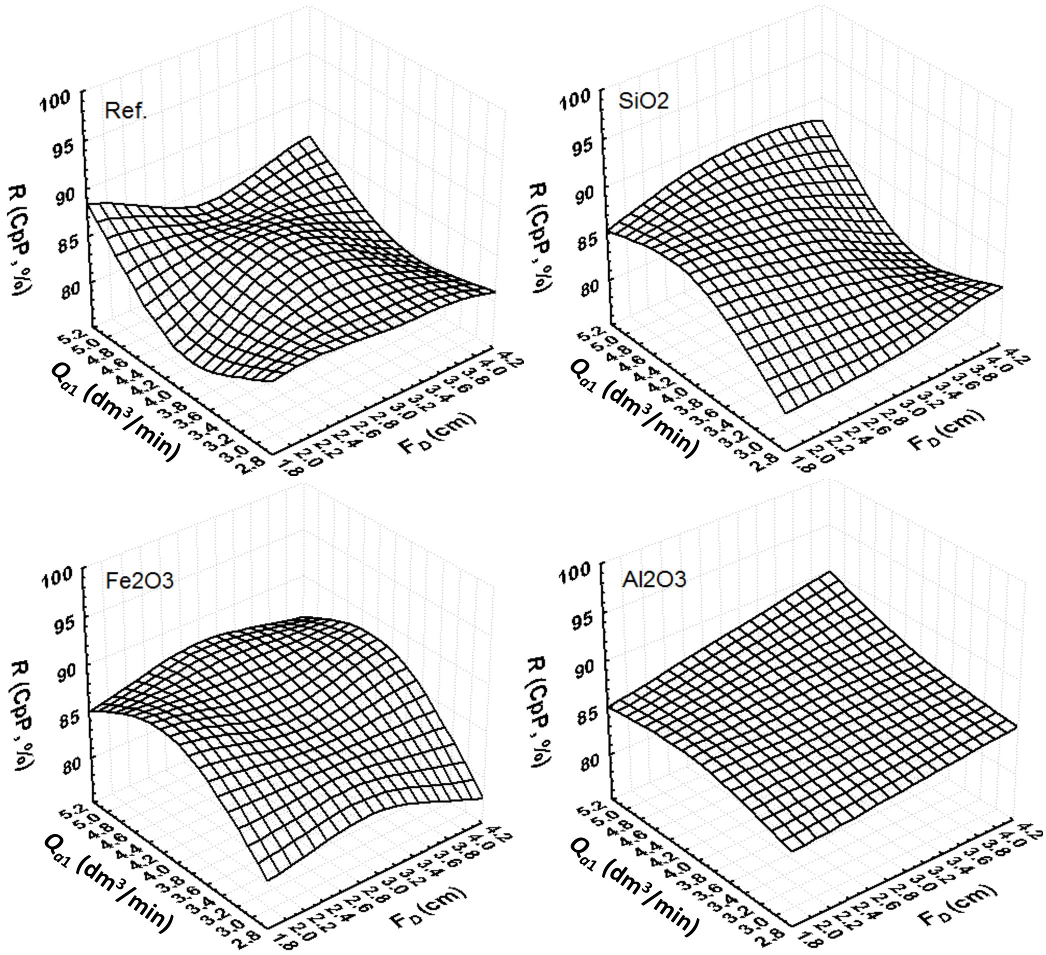

Fig. 4. Effect of the nanoparticles on the bulk flotation recovery [CpP=Chalcopyrite (%) + Pyrite (%)]

Influences of abovementioned mechanisms on the quality of concentrate are also observed as can be seen in Fig. 5. The increase in the concentrate grade is expected at the deep froths. The concentrate grade increased up to 15%, especially at lower air flow rates (Q_{a1}) with using the nanoparticles (Fig. 5). A remarkable increase obtained in the concentrate grade by using the and Fe_2O_3 and Al_2O_3 nanoparticles suggests that type of the nanoparticles is also important to improve the flotation performance.

The examination and discussions of the results by using different nanoparticles used in this study showed that significant differences between the results was obtained from in which one of the nanoparticle types used and the results obtained from using the other types. On the other hand, it is evident that the relationship between types of the nanoparticles and the experimental results is complex. The particle size is an important factor in the froth stabilization, but there are no significant differences between the sizes of the nanomaterials used as indicated above. However, the zeta potentials of the nanoparticles can be differentiated during the flotation tests. In a recent study, the zeta potential of each of the nanoparticles used in this study has been reported Cilek and Karaca (2015). The measurements done using the distilled water showed that the values of the point of zero charge (pzc) of the SiO_2, Fe_2O_3, and Al_2O_3, nanoparticles were obtained as 1.7, 6.2, and 7.6, respectively. The chemical analysis of the tap water used in the experiments showed that it can be classified as hard water. Ca^{2+} and Mg^{2+} ions in tap water was very high, and they may cause to change of the surface charge of the nanoparticles. As a results of the effects of these ions, the froth stability can also be improved (Farrokhpay, 2011; Bicak et al., 2012; Garbin et al., 2012; Liu et al., 2013). In addition to these, the morphological properties and the crystal structures of the nanoparticles can also be caused to complexity of the relationship between their types and the experimental results (Horozov, 2008; Garbin et al., 2012; Guven et al., 2015, 2016; Karakas et al., 2016).

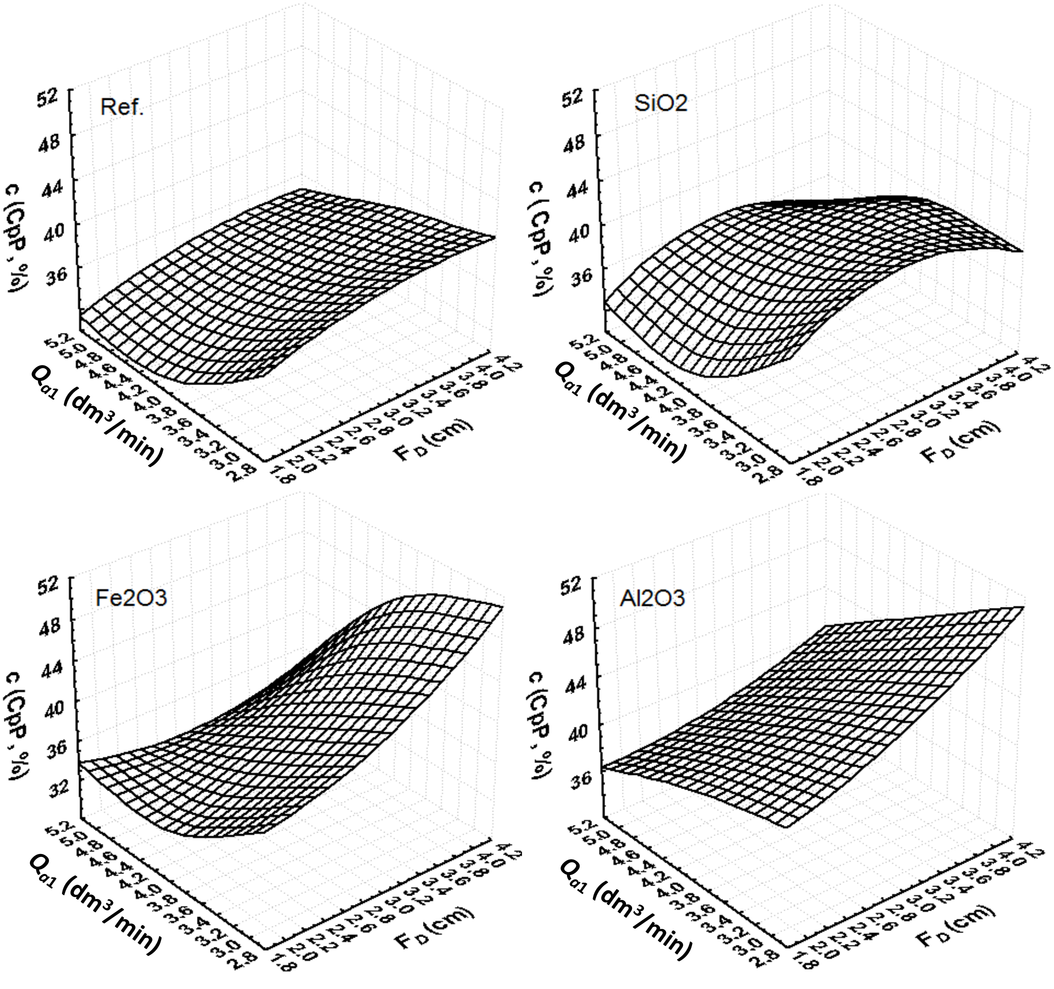

Fig. 5. Effect of the nanoparticles on grade of the bulk concentrate

Although it is expected that the deep froths causes to the decrease in the froth recovery, more than 10% increase in the froth recovery was obtained by using the Al_2O_3 nanoparticles (Fig. 6). As a result of this increase, the flotation recovery also increased as seen from Fig. 5. Contrarily, it has been observed that lower froth recovery obtained by using the SiO_2 and Fe_2O_3 nanoparticles. These findings are consistent with the experimental results presented in Fig. 2. As a result of the efficiency of the Al_2O_3 nanoparticles on the maximum froth depth and the froth stability factor, it provides highest increase in the froth recovery. Additionally, these findings suggest that the type of the nanoparticles is important not only in terms of the froth stability but also in the separation selectivity and efficiency of the flotation process.

In the light of the above discussions, it is evident that the nanoparticles and the supplementary froth fed into the froth phase improve the froth recovery and/or the froth stability. However, the pulp recovery for each case was calculated, and is not reported here because it was found not to be affected by using the nanoparticles and/or the supplementary froth. This is due to fact that events occurring in the froth phase do not directly influence the pulp phase, as also stated by Vera et al. (2002).

As shown by several investigators (Xu, 1998; Sripriya, 2003), the selectivity index can be used to measure the flotation selectivity. Therefore, the selectivity index, $SI_{S/G}=(RI_S k_S)/(RI_G k_G)$, where RI_S and RI_G are the ultimate flotation recovery of the sulphide minerals (S) and the gangue minerals (G), k_S and k_G are the flotation rate constants, was used to evaluate the flotation selectivity.

The selectivity index was calculated from the rate parameters for each case and, the results are presented in Fig. 7. It is clear that the presence of the SiO_2 and Fe_2O_3 nanoparticles did not significantly alter the flotation selectivity, as also observed in the concentrate grade (Fig. 5). However, the effect of the Al_2O_3 nanoparticles is most pronounced in all cases. As can be seen from Fig. 7, the flotation selectivity ($SI_{S/G}$) is reduced to a minimum by the use of Al_2O_3 nanoparticles. However, this minimum value is even higher than the maximum value of the selectivity index obtained from the experimental results in which the nanoparticles did not used.

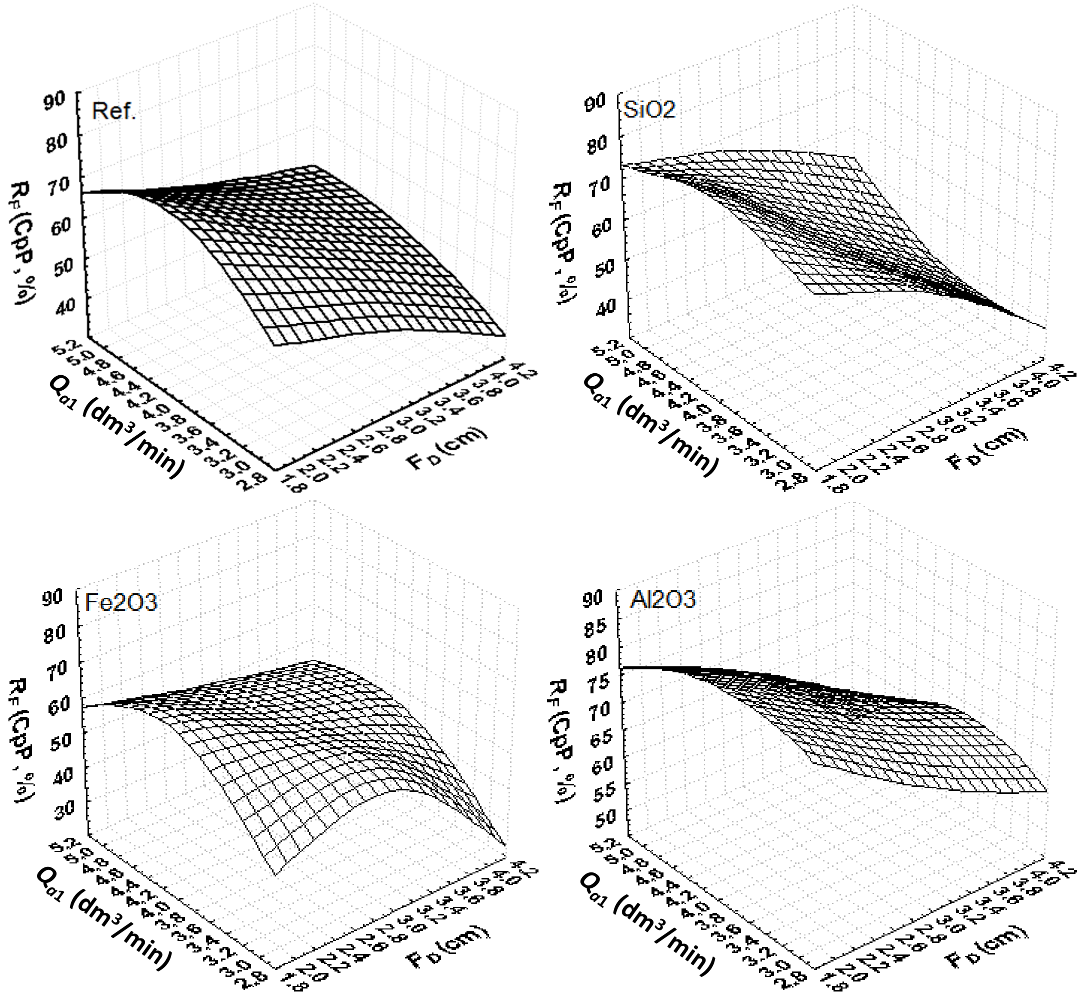

Fig. 6. Effect of the nanoparticles on the froth recovery

The results obtained from the tests without and with the nanoparticles have indicated once again that the froth phase has a significant role in the flotation performance in laboratory scale tests. It is known that a locked cycle flotation test (*LCT*) is conducted to experimentally simulate a continuous flotation process. In order to reveal the efficiency of the Al_2O_3 nanoparticles in the continuous flotation circuit, the first cycle of the *LCT* was conducted. The Al_2O_3 nanoparticles were used in each stage. The circuit consists of rougher-scavenger-cleaner (*RSC*) stages. A method developed by Agar and Kipkie (1978) was used to predict the flotation performance which can be obtained from the flotation circuit by using the first cycle data of the *LCT* (the primary split factors of each stage in the circuit). Details of the method can be found in the literature (Agar and Kipkie, 1978; Agar and Stratton-Crawley, 1982; Nishimura and Shobu, 2000). The simulation method can be described briefly as follows: The split factors are the fraction (mass or a mineral) of the feed (*F*) reporting to the tailings (*T*) streams from each stage, i.e., the mass split factor of ith stage is;

$$SF_i^M = TF^{-1}. \quad (4)$$

The mineral (or the element) split factor of i*th* stage is

$$SF_i^E = Tt(Ff)^{-1} \quad (5)$$

where *t* and *f* are grades of the tailings and feed streams, respectively. Each stream and separators (i.e., stages) in the flotation circuit are numbered in sequence and a set of the mass balance equations is generated. By using the split factors which are obtained from the first cycle of the locked cycle flotation test, the calculations of the mass and the mineral content of each stream are sequentially repeated until the steady state is reached. Mostly, six or eight iterations are required to arrive at the steady-state balance.

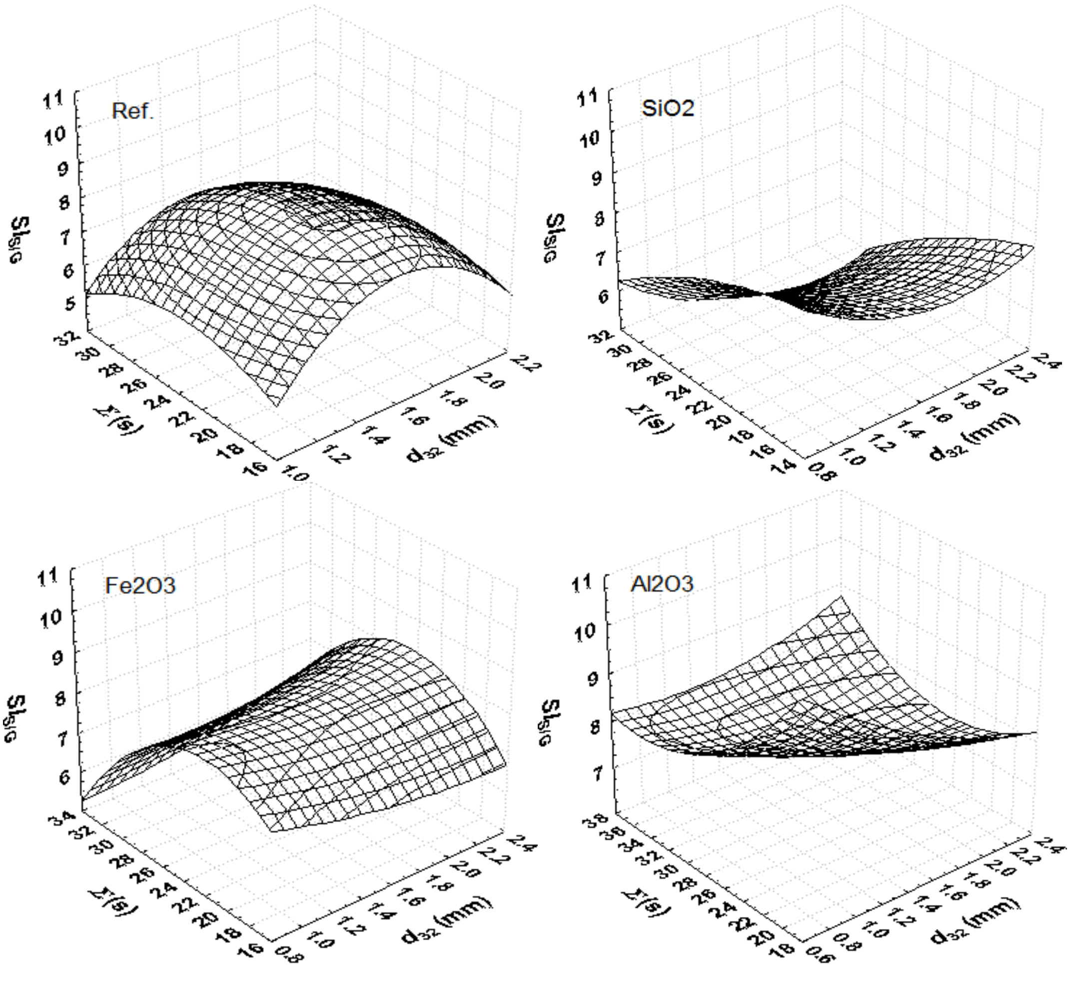

Fig. 7. Effect of the nanoparticles on the selectivity index as a function of bubble size and froth stability

The results of the first cycle test and the predicted data of the *LCT* for the circuit are presented in Table 1. It is clear from the data presented in Table 1 that there is a pronounced effect of the Al_2O_3 nanoparticles on the flotation performance for each stage. Most importantly, the predicted results of the *LCT* suggest that an increase in the flotation recovery (~10%) and the concentrate grade (~15%) can be accomplished by using the Al_2O_3 nanoparticles in the continuous flotation. The evaluations of the experimental and predicted results show that a nanoparticle of properly selected can be used to improve the separation selectivity and efficiency of the flotation process.

Table 1. Comparison of the results of the first cycle test of the *LCT* with and without the Al_2O_3 nanoparticles for the sulphide ore and the predicted results of the RSC circuit

Tests	Stages	Products	Results of the first cycle				Predicted results of the LCT			
			Grade (%)			Rec.(%)	Grade (%)			Rec.(%)
			Cu	Fe	CpP	CpP	Cu	Fe	CpP	CpP
With the nano-sized Al_2O_3	Rougher	Concentrate	5.07	13.75	34.61	84.62	4.96	13.33	33.59	104
		Tailings	0.26	0.93	2.26	15.38	0.25	0.90	2.19	18.90
	Scavenger	Concentrate	1.84	4.62	11.77	8.20	1.80	4.47	11.41	10.07
		Tailings	0.08	0.51	1.18	7.19	0.08	0.49	1.14	8.82
	Cleaner	Concentrate	9.18	24.70	62.25	74.16	8.98	23.93	60.39	91.13
		Tailings	1.16	3.35	8.36	10.48	1.14	3.25	8.11	12.87
	Feed		1.53	4.32	10.81	100	1.53	4.32	10.81	100
Without nanoparticles	Rougher	Concentrate	4.50	10.84	28.04	79.30	4.45	10.82	27.69	101.62
		Tailings	0.28	1.39	3.27	20.70	0.28	1.37	3.23	26.52
	Scavenger	Concentrate	1.67	4.08	10.44	7.69	1.65	4.01	10.27	9.82
		Tailings	0.10	1.04	2.33	13.07	0.10	1.02	2.30	16.70
	Cleaner	Concentrate	7.26	17.30	44.43	65.00	7.22	17.07	43.88	83.30
		Tailings	1.49	4.18	10.47	14.29	1.48	4.12	10.34	18.32
	Feed		1.57	4.35	10.91	100	1.57	4.35	10.91	100

4. Conclusions

The effect of the increase in the froth depth on the Sauter-mean bubble size or the bubble coalescence becomes negligible by using the nanoparticles in the flotation. Thus, the sufficient bubble surface area for attachment in the froth phase is provided, and therefore, the detached and entrained (fine-sized) valuable minerals re-attach to the surfaces of the nanoparticle stabilised bubbles. As a result of these events occurring in the froth phase, the selectivity and efficiency of the flotation increased.

The experimental results suggest that type of the nanoparticles is also important to improve the flotation performance.

The predicted results of the *LCT* show that the nanoparticles can play an important role not only in batch flotation but also in the continuous flotation.

The predicted results of the *LCT* also indicate that the bulk flotation recovery of the sulphide minerals (chalcopyrite and pyrite) in the ore increased from 83% to 91%, and the concentrate grade increased from 44% to 60% with the Al_2O_3 nanoparticles.

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