Minerals Engineering 21 (2008) 873–876

Contents lists available at ScienceDirect

Minerals Engineering

journal homepage: www.elsevier.com/locate/mineng

Residual amine in iron ore flotation

Geraldo M.B. Batisteli [a], Antonio E.C. Peres [b,*]

[a] Samarco Mineração SA, Mina do Germano, Caixa Postal 22, 35420-000 Mariana, MG, Brazil
[b] EEUFMG, Rua Espírito Santo, 35/206, 30160-030 Belo Horizonte, MG, Brazil

ARTICLE INFO

Article history:
Received 27 November 2007
Accepted 11 April 2008
Available online 22 May 2008

Keywords:
Flotation collectors
Froth flotation

ABSTRACT

The possibility of recovering residual amine present in the water phase of tailings pulps of the reverse cationic flotation of iron ore was investigated. The samples collected in the tailings from mechanical and tank cells and also from flotation columns were kept under rest to promote the separation between the water and the solid particles. The water was used for determination of the residual amine concentration and for flotation tests. The residual amine concentration was determined through indirect quantitative analysis using a spectrophotometer. The flotation tests were performed using, separately, each tailings water and a composition of them. Four sets of tests were carried out. The first three sets indicated the strong possibility of reusing the amine present in the water. As a consequence, the dosage of amine was reduced and the residual amine concentration of three out of four collection points was below the detection limit in the sampling for the last set.

1. Introduction

The plant practice of the reverse cationic flotation of iron ores started in Brazil in 1977. Starch is used as depressant for iron minerals and amine acts as quartz collector and also as frother.

Amine is an expensive reagent and its consumption tends to be higher with the increasing contents of quartz in the ores being processed. Controlling the costs related to amines represents a major challenge for the process engineering team in charge of the iron ore concentrators.

Samarco uses approximately 1500 tonnes per year of amine, at a production rate of 16 million tons per year of concentrate, representing close to 50% of the overall costs with reagents. The concentrator is being refurbished to produce 24 million tones per year of pellet feed fines.

Most of the amine added to the flotation circuit is discarded to tailings ponds either adsorbed onto quartz particles or dissolved in the water fractions of the tailings. The reuse of part of this amine combines economical issues with environmental targets. Despite the lack of evidences of toxicity of the amines used in ores flotation (Peres and Ciminelli, 2002), reducing the presence of chemicals content in effluents is always desirable.

2. Literature review

Smith and Akhtar (1976) stated that, due to the fact that the adsorption of amine onto quartz is electrostatic and involves low heat transfer, it may be considered a reversible process. According to this reasoning, desorption of amine from quartz would be feasible at strongly alkaline or acidic conditions.

Nevertheless, experimental results show that extreme conditions are not necessarily required for desorption. Oliveira et al. (1996) investigated the desorption of dodecyl amine from pure quartz at pH levels of 4.0 and 6.4 adjusted with HCl. The suspension was filtered after 10 min of contact and the liquid phase was analysed for amine. It was possible to recover 60% of the amine added to the system, 2/3 coming from desorbed amine and 1/3 corresponding to non-adsorbed residual amine.

Baltar et al. (2002) reached 80% desorption of ether dodecyl amine adsorbed onto pure quartz in the size range <44 μm, working at pH 3. The desorbed amine was effective in pure quartz flotation at bench scale.

Barbato et al. (2004) investigated the adsorption of amine present in effluents from iron ores flotation onto stilbite, a mineral of the zeolites class. The target was purifying the water and there was no mention to the reuse of amine. Removal levels higher than 95% were achieved.

The topic of residual amine in flotation waters was addressed by Orphan and Bayraktar (2006), but the target of the investigation was to detect its effect on the quality of a feldspar flotation concentrate rather than the reuse of residual amine.

3. Methodology

The amine concentration in aqueous phases was determined as a function of the absorbance in a UV visible spectrophotometer. Two calibration curves were plotted based on an ether monoamine

---

* Corresponding author. Tel.: +55 3134091717; fax: +55 3134091815.
E-mail address: aecperes@demet.ufmg.br (A.E.C. Peres).

0892-6875/$ - see front matter © 2008 Elsevier Ltd. All rights reserved.
doi:10.1016/j.mineng.2008.04.002

and an ether diamine, manufactured by Clariant and used in the plant practice.

Tailings samples were collected in the four circuits of the concentrator: mechanical cells (76% of the total tailings), tank cells (10%), recleaner columns (8%), and slimes columns (6%). It is worthwhile recalling that in reverse flotation the tailings consist of the fraction collected at the top of the cell.

The tailings samples were left under rest for 12 h and then the solid liquid separation was performed via siphoning. The liquid phase was composed according to the proportions of each tailings fraction and used in the bench scale flotation experiments and in the residual amine concentration determinations.

The iron ore sample for the laboratory flotation experiments was composed from the feed of the first stage of concentration (mechanical cells) during a three months operation period. The overall sample was homogenised and quartered.

The experiments were performed in a Wemco laboratory machine, operated at 1300 rpm, in a 3 L tank. The depressant conditioning (5 min) was carried out with a pulp containing 1500 g of ore and 800 mL of tap water to prevent the contact of particles with water containing the collector prior to starch adsorption. After the conditioning with starch, 1600 mL of tailings water were added to the cell jointly with the amine dosage selected for the test. At the beginning of the test the weight percent solids of the pulp was 38.5%. Amine conditioning time was set at 1 min. After 1 min of froth collection, an extra volume of 300 mL of tailings water was added to the cell. The total time of froth collection was 3 min.

In a first set of experiments, amine from residual water was the only source of collector. Later, experiments were performed with the addition of amine dosages of 20 g/t, 35 g/t, and 50 g/t. For this sequence a *composition water* was prepared taking into account the proportion of tailings from each circuit. Another series of experiments simulated an operation with the use of only residual amine in the rougher flotation and amine solution addition in a cleaner stage at dosages of 20 g/t, 35 g/t, and 50 g/t.

The results of the tests with water containing residual amine were compared with those from the standard test, performed with distilled water and amine solution prepared in the laboratory.

The control parameter was silica content in the concentrate.

4. Results and discussion

The time sequence of the concentration of residual amine in the tailings of each individual circuit is illustrated in Fig. 1. The sharp decrease observed from 11/11/2005 to 13/09/2006 was due to several factors, such as: circuit changes, increase in the throughput due to the addition of new mechanical cells, increase of the proportion of diamine in the collector blend, start up of an advanced control system in the conventional flotation circuit, and decrease of the specific collector dosages in all addition points. This latter factor was a consequence of this study. Specific amine consumption at Samarco's concentrator was lower than that at other plants, nevertheless the detection of residual amine showed that reductions in the addition level should be carried out.

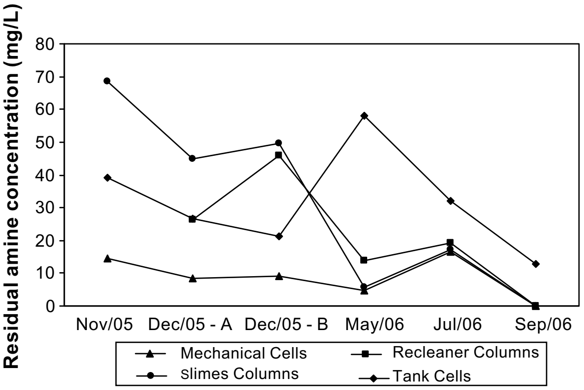

Fig. 1. Time sequence of the concentration of residual amine in the tailings of individual circuits.

Results of standard tests, illustrating the effect of amine dosage on silica content and iron recovery in the concentrate are presented in Table 1.

Results of silica content in the concentrate in tests performed with residual water from the mechanical cells circuit, in the absence of extra amine, are presented in Fig. 2.

Results from the test carried out in July 2006 (residual amine dosage 21.0 g/t; silica content in the concentrate 3.49%) are very similar to those from the standard test (residual amine dosage 20.0 g/t; silica content in the concentrate 3.48%).

Results of silica content in the concentrate in tests performed with residual water from the tank cells circuit, in the absence of extra amine, are presented in Fig. 3.

The results are erratic and it is difficult to explain the lack of correlation of the results of the tests carried out in May and July 2006. All experiments were performed in duplicate and the results

Table 1
Results of standard tests

Control parameter	Amine dosage (g/t)		
	20	35	50
%SiO_2 concentrate	3.48	1.97	1.67
%Fe recovery	92.19	88.47	84.85

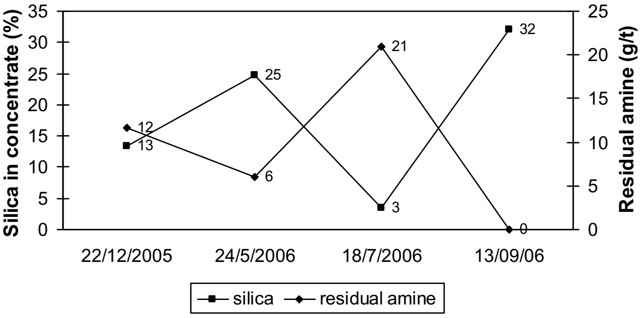

Fig. 2. Results of silica content in the concentrate in tests performed with residual water from the mechanical cells circuit, in the absence of extra amine.

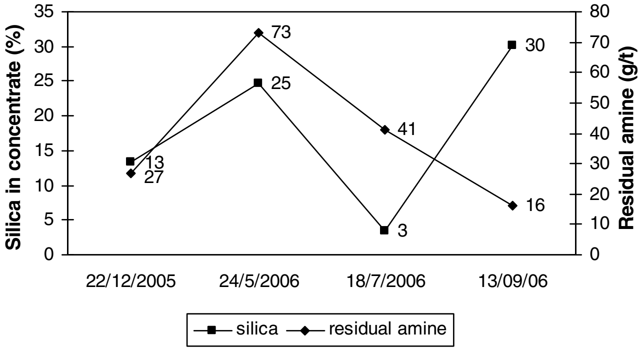

Fig. 3. Results of silica content in the concentrate in tests performed with residual water from the tank cells circuit, in the absence of extra amine.

are reliable. A possible explanation for the poor performance of the residual water containing 73 g/t residual amine is the presence of slow settling ultrafine particles. These particles are generated in the secondary grinding that precedes the tank cells circuit. The turbidity of this residual water is significantly higher than that of the other fractions. In the sampling of September 2006, this residual water was the only one presenting amine concentration above the detection limit, despite the fact that no collector is added to the tank cells circuit.

Results of silica content in the concentrate in tests performed with residual water from the recleaner flotation columns circuit, in the absence of extra amine, are presented in Fig. 4.

A thick froth layer was observed on the top of the tailings of the recleaner column flotation circuit even after a 12 h rest period. This froth layer was not observed in the other tailings. The results achieved with the residual water from the recleaner flotation columns were poor in comparison with those from the standard test. Once more, the presence of ultrafines from the regrinding stage might be causing the poor performance.

Results of silica content in the concentrate in tests performed with residual water from the slimes flotation columns circuit, in the absence of extra amine, are presented in Fig. 5.

The sharp drop in the concentration of residual amine was a consequence of the decrease of the addition level of collector from 200 g/t or 300 g/t to 50 g/t in September 2006. At this low level, no residual amine was detected in the residual water.

The results in Fig. 5 present the same trend observed in Fig. 2 (residual water from the mechanical cells circuit): higher levels of amine in the residual water led to lower figures of silica content in the concentrate.

The next series of results corresponds to tests with the use of the composition water plus extra amine solution. The values plotted in abscissa correspond to extra amine. The results of standard tests are also plotted for the sake of comparison. Fig. 6 presents the results of experiments performed with water collected in December 2005. The amine content of the composition water in December 2005 was 13 g/t.

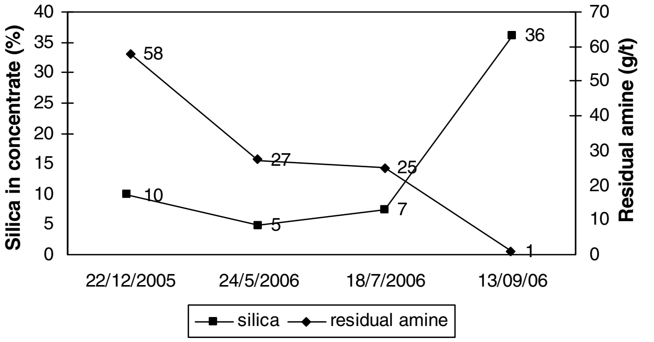

Fig. 4. Results of silica content in the concentrate in tests performed with residual water from the recleaner flotation columns circuit, in the absence of extra amine.

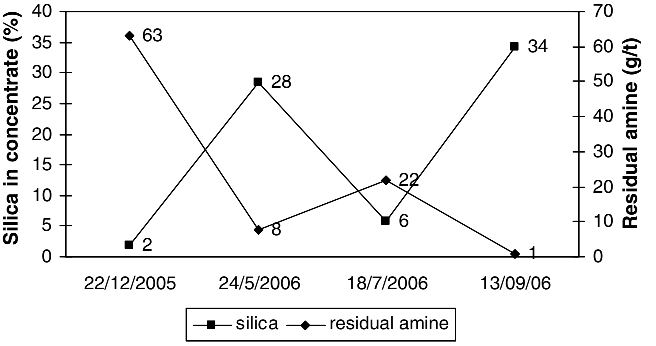

Fig. 5. Results of silica content in the concentrate in tests performed with residual water from the slimes flotation columns circuit, in the absence of extra amine.

The composition water enhances the performance of the amine solution suggesting the reuse of tailings water as a route to reduce the amine dosage.

Fig. 7 presents the results of experiments performed with water collected in May 2006. The amine content of the composition water in May 2006 was 7.9 g/t. Fig. 8 presents the results of experiments performed with water collected in July 2006. The amine content of the composition water in July 2006 was 13.4 g/t. In both cases there was no indication that the use of the composition water would reduce the collector addition level.

Fig. 9 presents the results of experiments performed with water collected in September 2006. The amine content of the composition water in September 2006 was 2.5 g/t.

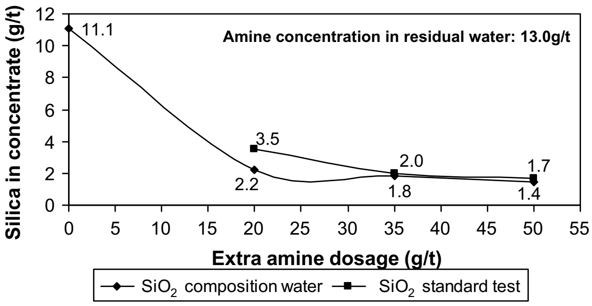

Fig. 6. Results of flotation tests performed with composition water in December 2005.

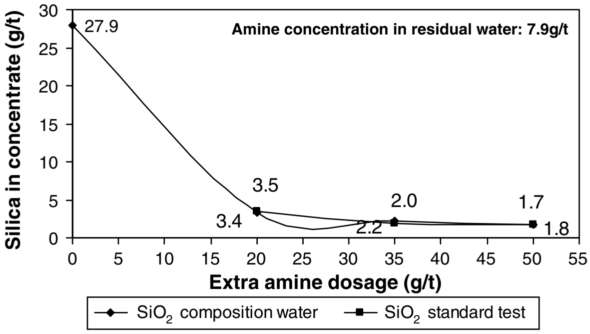

Fig. 7. Results of flotation tests performed with composition water in May 2006.

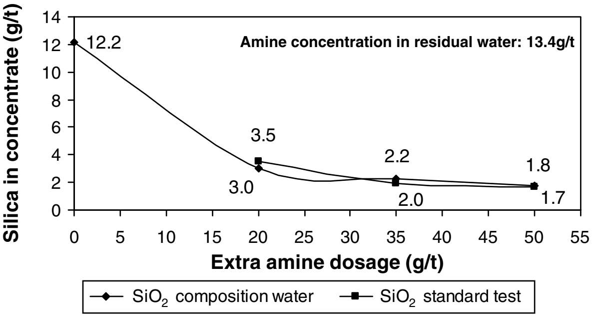

Fig. 8. Results of flotation tests performed with composition water in July 2006.

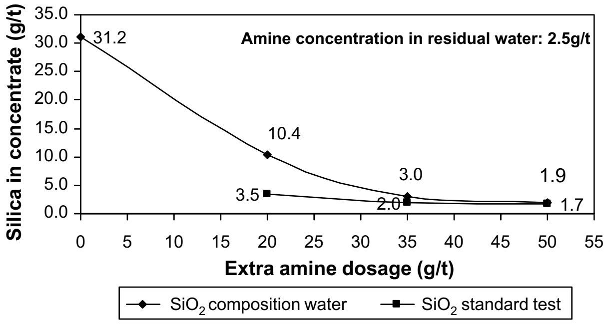

Fig. 9. Results of flotation tests performed with composition water in September 2006.

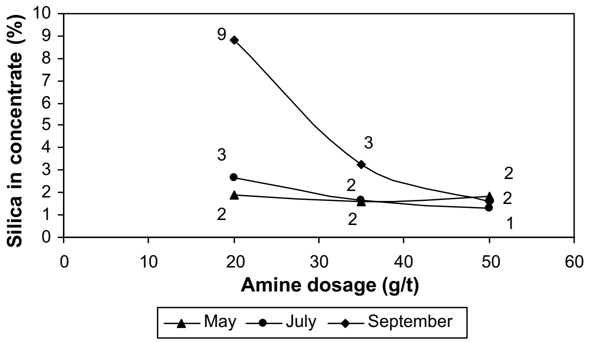

Fig. 10. Results of silica content in the cleaner concentrate in three sets of experiments.

As expected, the very low content of residual amine in the residual water did not suggest that its use would lead to a decrease in the collector addition level.

The difference in performance between the composition water collected in 2005 and those collected in 2006 may be due to circuit changes in the concentrator. The tank cells were moved from ahead of the regrinding mills to after this stage, generating ultrafines that were entrapped in the water phase, impairing its reuse.

Fig. 10 reports results of tests in which only residual amine was used in a rougher stage, followed by a cleaner stage with addition of amine solution. The results correspond to residual amine concentrations of 7.9 g/t (May), 13.4 (July), and 2.5 (September), respectively. Even a low dosage of extra amine (20 g/t) was effective to clean the rougher concentrates produced in May and July, despite an inversion in the results with respect to those predicted for the two dates. Higher dosages of extra amine performed well to clean all rougher concentrates.

There is a strong indication that the addition of residual water itself does not impair the flotation performance suggesting the possibility of reuse of water from the tailings fractions, since they do not contain fines from regrinding stage.

5. Conclusions

Residual amine, if present in the water phase from flotation tailings, may be reused for flotation. The effectiveness of the previously used amine depends on its concentration and is impaired by the presence of ultrafines.

The reuse of water from mechanical cells tailings, even at low amine concentration, does not impair the flotation process, representing a significant benefit, for this fraction contains 76% of the total water in tailings from different circuits.

References

Baltar, C.A.M., Araújo, J.M.M., Cunha, A.S.F., 2002. Study on the conditions of desorption and reuse of collector in quartz flotation. In: XIX National Meeting of Mineral Processing and Extractive Metallurgy (XIX Encontro Nacional de Tratamento de Minérios e Metalurgia Extrativa). Recife, vol. I, pp. 241–246 (in Portuguese).

Barbato, C.N., Andrade, M.C., França, S.C.A., Luz, A.B., 2004. Adsorption and quantifying of amines in mining effluents. In: Undergraduate Symposium – CETEM, XII. Rio de Janeiro, Brazil (in Portuguese).

Oliveira, J.F., Wilberg, K.Q., Baltar, C.A.M., Souza, C.C., Araujo, A.C. 1996. Recovery of amine adsorbed onto quartz and in the floated water aiming at its reuse in the reverse iron ore flotation. In: I Brazilian Symposium of Iron Ores: Characterisation, Processing, and Pelletising, Ouro Preto, pp. 375–381 (in Portuguese).

Orphan, E.C., Bayraktar, I., 2006. Amine–oleate interactions in feldspar flotation. Minerals Engineering 19 (1), 48–55.

Peres, A.E.C., Ciminelli, V.S.T., 2002. Environmental fate of an amine used in the concentration of Brazilian iron ores. In: TMS Annual Meeting, Poster Session. Seattle, USA.

Smith, R.W., Akhtar, S., 1976. Cationic flotation of oxides and silicates. In: Fuerstenau, M.C., Flotation, A.M. (Eds.), Gaudin Memorial Volume, vol. 1 (c 5). Society of Mining Engineers, pp. 87–133.