CLOSED WATER CIRCUIT IN A PHOSPHATE ORE CONCENTRATOR

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ABSTRACT

A concentrator producing phosphate rock for the Brazilian fertilizers industry was fully refurbished, mechanical cells being replaced by flotation columns. The investigations necessary for this major change led to the development of a batch column. The effect of interfering ions in the stages of barite and apatite flotation was assessed. Tolerance limits were defined for the ions that might interfere with the flotation performance. Plant operation with the concentration of interfering ions kept below these limits was possible with a closed water system. The contamination levels of the species considered to be deleterious to the environment in the receiving body of Serrana's effluents were brought down to values inferior to those established by the government environmental agency.

INTRODUCTION

Fertilizantes Serrana operates a mine and concentrator, producing phosphate rock for the fertilizers industry, located in the neighborhood of the town of Araxá and also of a very famous resort hotel. The Grande Hotel Araxá was built in 1945 to host a casino. It is a magnificent construction, decorated with crystals from Bohemia and Persian rugs. A few months later, gambling was declared illegal in Brazil. The hotel struggled for years and was finally closed in the early 90's. Gambling is on the verge of being legalized again and the hotel is being fully refurbished. Medicinal and mineral waters are the major features of the park around the hotel.

The operation of the concentrator, since the start up in 1978, always represented an environmental hazard. A closed water system was an expectation of government agencies and also of the population of Araxá. A technological solution for the problem was developed, involving the neutralization of the effluent with lime. Nevertheless, it was economically unfeasible to treat 2,800 m^3/h using this method. Despite the constant pressure from government agencies, ONG's and the press the activities went on and effluents containing some elements beyond the legal limit were disposed. The impossible dream of a closed water circuit started to become a reality seven years ago. As it will be described later the overwhelming problem was solved with the help of straight mineral processing technology.

In the early 90's the Brazilian market for phosphate rock was in trouble. Imported rock was reaching the country at a low price and there was no definite government policy for agriculture. The company had two options: production costs reduction x shutting down. The decision for the first option forced the company to technological innovations.

The concentrator was fully refurbished. Plant practice experience, knowledge of flotation fundamentals and mathematical modelling were the tools for the changes: six flotation columns substituting for 64 mechanical cells (Guimarães, 1995) and increasing the apatite regrinding capacity with a new ball mill (Fernandes, 1997).

A methodology of batch column flotation testing was developed for circuit design and was a key feature for the success of the changes (Guimarães, 1997).

The environmental benefits were a consequence of the full knowledge and control of the mineral processing plant practice (and its fundamentals) in the company. The batch column was then used to investigate the effect of interfering ions in the barite and apatite flotation circuits.

Apatite and the major gangue minerals, barite, calcite, dolomite, micas and altered silicates are classified as sparingly soluble minerals. Ions introduced in the pulp by these species and also the collectors of barite and apatite build up in the circuit when process water is recirculated. The challenge was: is a closed water circuit feasible? The answer based on the previous experience was: there is no selectivity in the presence of interfering ions.

By means of the experiments reported in this paper, acceptable limits for interfering ions in the flotation circuits of barite and apatite were determined in a batch column and checked in the industrial plant.

Plant operation with the concentration of interfering ions kept below these limits was possible with a closed water system. The contamination levels of the species considered to be deleterious to the environment in the receiving body of Serrana's effluents were brought down to values inferior to those established by the government environmental agency.

PLANT PRACTICE

Serrana is the largest producer of phosphate fertilizers in Brazil. The company operates two concentrators in Cajati, São Paulo state, and Araxá, Minas Gerais state. Araxá's concentrator processes approximately 3.5 Mt of ore per year with grades in the range of 13.0% to 14.0% P_2O_5 and a production capacity of 800,000 t per year of concentrate at a grade of 36% P_2O_5.

The Barreiro carbonatite complex is located in Araxá, state of Minas Gerais, center-south of Brazil. The shape of the complex is roughly circular, with a diameter of 4.5 km, and an area of approximately 16 km^2. The major components of the rock formation are carbonatite and glimmerite rocks. The following remarks refer specifically to the Serrana's concession, representing 20% of the complex total area.

The weathering effects on glimmerite (with an essentially micaceous composition) caused partial alteration to vermiculite; other silicates were transformed into chlorite group minerals; iron minerals were altered and some of the apatite was changed to hydrated amorphous phosphates. The carbonatite suffered, beyond the above mentioned phenomena, leaching of carbonates and reprecipitation of iron, barium, phosphate and silicon ions.

The total measured reserve represents 142.5 Mt. The oxidised ore (reserves of 74.6 Mt) is the only one currently mined. The annual production is 3.5 Mt of ore and 3.5 to 3.8 Mt of overburden material.

The ore is processed in four physically distinct facilities:

- crushing plant,
- concentrator,
- high field demagnetizing, and filtration plant, and
- drying plant.

The ore is crushed (jaw and impact crushers) and homogenized in the crushing plant and then it is reclaimed to the concentrator. The unit operations in the concentrator are: grinding (rod and ball mills), classification in hydrocyclones, low field magnetic separation, desliming in hydrocyclones and barite and apatite flotations.

Two kinds of apatite concentrates are produced:

- GCA: apatite concentrate from the coarse ore fraction,
- FCA: apatite concentrate from the fine ore fraction.

The GCA production circuit consists of a pre-treatment step of barite flotation, followed by apatite flotation. The tailings from the apatite flotation circuit are reground and then retreated in a separate flotation bank. Concentrates from the two apatite flotation stages are combined and feed a high field magnetic separation circuit, which is responsible for controlling the Fe_2O_3 content. The concentrate is filtered using horizontal disk filters.

The natural fines and the fines generated by grinding are deslimed in small diameter cyclones and feed two lines of apatite flotation. The concentrates are combined forming the so-called FCA fraction, which is filtered using drum filters.

The total concentrate production capacity is 800,000 t per year (560,000 t GCA and 240,000 t FCA). These concentrates form the raw material for the production of phosphate fertilizers by means of acidulation with sulphuric acid. Part of the consumption of these concentrates is internal to the group with the remaining portion being sold to other Brazilian companies that solubilize the phosphate rock.

Flotation operations at Serrana were carried out using mechanical cells (300 ft^3) since the beginning of industrial operation in 1978. The original flowsheet is presented in Figure 1. The flotation circuits presented excessive recirculating load and consequent difficulties in operational control.

The plant refurbishing (mechanical cells being replaced by flotation columns) was performed in two stages: 1st stage - apatite flotation from natural fines and fines from grinding (started up in February 1993); 2nd stage - barite and apatite flotation from the coarse ore fraction and apatite flotation from the reground fraction (started up in February 1994). The new flowsheet is presented in Figure 2.

Serrana´s flowsheet was significantly simplified with the introduction of the columns. The number of stages in the flotation circuit was outstandingly reduced. Overall, 66 mechanical cells were replaced by only six flotation columns.

The laboratory procedure for checking the plant behavior was based on tests in conventional mechanical cells. The need for a fast and reliable method for monitoring column flotation led to the assemblage of a batch flotation column.

EXPERIMENTAL

The batch flotation column is built in acrylic, presents internal diameter of 2" (51 mm) and total height of 3,748 mm and consists of four sections:

- first section: height 1,030 mm, contains the weir for collecting the floated fraction,
- second section: height 1,013 mm, contains the pulp feed opening,
- third section: height 1,013 mm,
- fourth section: height 680 mm, contains the air sparger and the discharge end for the non floated fraction.

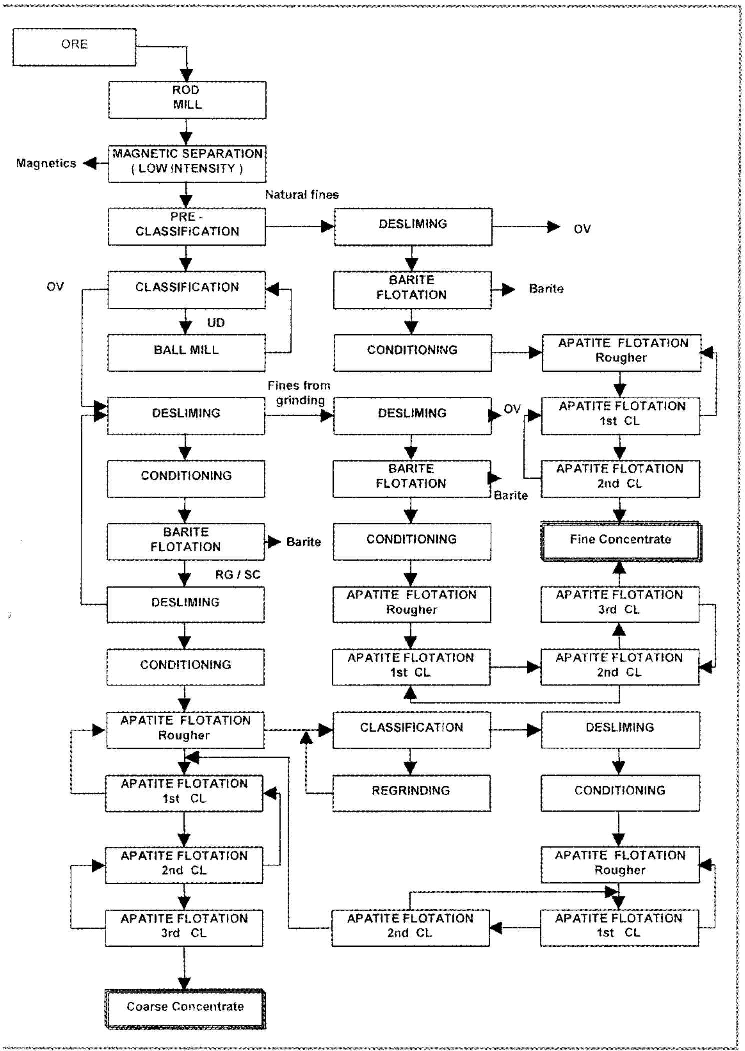

Figure 1. Original flowsheet of the concentration plant.

The air sparger utilized is a polyethylene tube with 30% porosity and the following dimensions: external diameter = 40 mm; internal diameter = 24 mm and height = 75 mm.

Air and wash water flow rates are measured by means of flowmeters. A pump peristaltic to the feed, positioned in the second section recirculates the pulp.

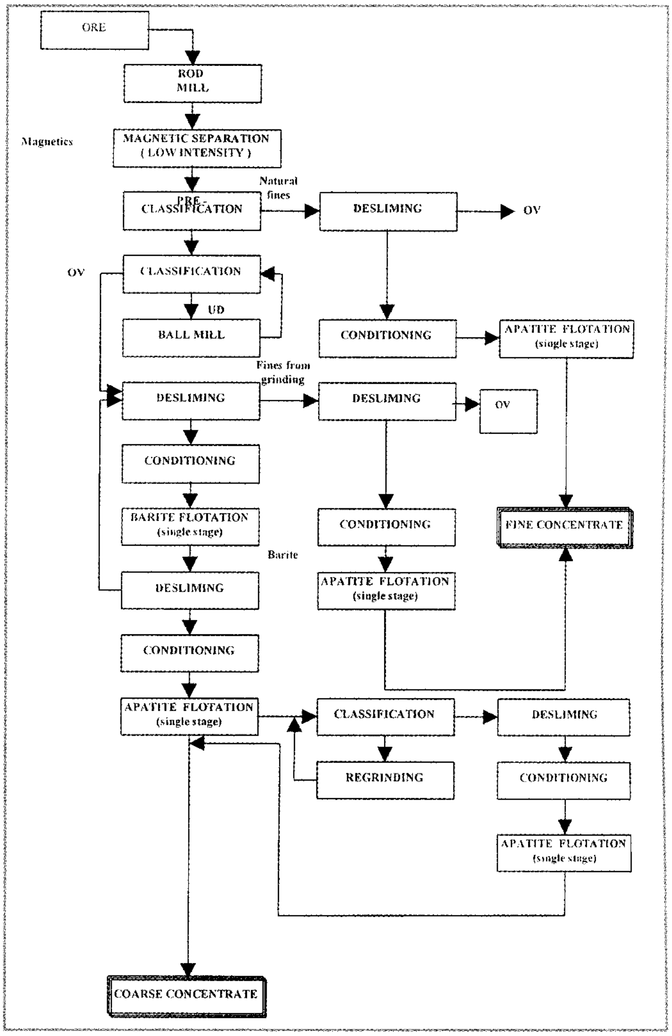

Figure 2. Concentration plant flowsheet after being fully refurbished.

The pulp is previously conditioned and then diluted to feed the batch flotation column from the top, after adjusting the air flow rate and connecting the pulp recirculation device. The wash water is turned on and the floated material is collected until the froth is barren. The fraction that did not float is then drained from the equipment.

The wash water spray consists of a plastic tube with external diameter 5/16" (7.9 mm) and 12 holes with diameter of 1/16" (1.6 mm) distributed along three rows.

The following flotation studies were performed:

- definition of the standard test procedure,
- influence of ions on the flotation of barite and apatite.

The effect of the following variables was evaluated: air flow rate, wash water flow rate, non floated recirculated pulp flow rate, mass of solids and feed pulp volume.

The reagents dosages utilized in the industrial plant were reproduced in the batch column.

The influence of ions was checked individually for the flotation of barite and apatite:

- barite flotation: the effect of cations (sodium, calcium and magnesium) and anions (chloride, phosphate, fluoride and fatty) dosed one at a time and together was assessed,
- flotation of apatite: the effect of cations (sodium, calcium and magnesium) and anions (chloride, phosphate, fluoride) dosed one at a time and together was assessed.

The samples for the batch column flotation experiments were collected in the industrial plant, from a pile that may be considered as typical of the oxidized ore, representing the feed to the circuits of barite and apatite flotation, respectively:

- ore that was ground, deslimed and subjected to low field magnetic separation (barite flotation feed),
- ore that was ground, deslimed and subjected to low field magnetic separation and barite flotation (apatite flotation feed).

The chemical analyses of the two samples is:

	$BaSO_4$%	P_2O_5%	CaO%	Fe_2O_3%	MgO%	SiO_2%
Barite flotation feed	9.03	19.23	25.64	17.64	0.74	9.45
Apatite flotation feed	1.71	22.40	26.96	19.68	0.60	8.94

The major difference between the samples is the barite grade that decreases from 9.03% to 1.71% after the pre-flotation stage. As a consequence the P_2O_5 content is increased by 3%.

The standard test for barite flotation is described next.

The sample was conditioned with the barite collector and the frother, at 45% solids, pH = 9.5, for 1 min. The pulp was then diluted with water to 7 L and was ready to feed the column. The following variables were evaluated: dosage of the barite collector (Flotinor S72 - Clariant), dosage of the frother (Flotanol D14 - Clariant), air superficial velocity, wash water superficial velocity, pulp recirculation flow rate, mass of solids in the flotation feed and initial froth layer height.

The procedure of the standard test for apatite flotation follows.

The sample was conditioned with gelatinized corn starch and rice bran oil soap, at 60% solids, pH = 11.5, for five min for each reagent. The pulp was then diluted with water to 7 L and was ready to feed the column. The following variables were evaluated: dosage of gelatinized corn starch, dosage of rice bran oil soap, air superficial velocity, wash water superficial velocity, pulp recirculation flowrate, mass of solids in the flotation feed and froth layer initial height.

RESULTS

With the same reagents dosage utilized in the industrial plant, the selectivity did not change and the apatite recovery was 2% lower.

The minor deviations between the results from the batch column tests (mean of 5 experiments) and industrial results (24 hours operation) are within the accuracy range of flotation tests.

The ions were dosed in the conditioning stage. Due to the fact that the cations were added as chlorides and the anions as sodium salts, the effect of the addition of sodium chloride was also evaluated.

Barite recovery decreases with an increase in the dosage of calcium, magnesium, phosphate and fluoride ions. The recovery was expressed on a relative basis. Barite recovery in the presence of interfering ions was divided by the recovery in the standard test, in the absence of these ions. This ratio was designated as R/Rba.

The interference of ions on barite recovery is illustrated in Figure 3. Apatite loss was lower than 2% in all tests. The addition of sodium chloride did not affect barite flotation, even at high dosages.

Barite flotation was performed at pH = 9.5. Equilibrium diagrams of species in the liquid phase predict the following sequence, in decreasing concentrations:

- barium: Ba^{+2}, $Ba(OH)^{+}$, calcium: Ca^{+2}, $Ca(OH)^{+}$, magnesium: Mg^{+2}, $Mg(OH)^{+}$, fluoride: F^{-}, phosphorus: HPO_4^{-2} and fatty: $RCOO^{-}$, $(RCOO)_2^{-2}$, $(RCOO)_2H^{-}$ and $RCOOH_{(aq)}$.

Apatite recovery decreases significantly with an increase in the concentration of interfering ions. The recovery was expressed on a relative basis. Apatite recovery in the presence of interfering ions was divided by the recovery in the standard test, in the absence of these ions. This ratio was designated as R/R_{ap}.

The interference of ions on apatite recovery is illustrated in Figure 4. The ions calcium and magnesium react with the apatite collector, forming insoluble rice bran oil soaps, reducing, then, the collector level in the system. Phosphate and fluoride ions are common to the apatite lattice, being depressants conventionally mentioned in the literature. The addition of sodium chloride did not affect barite flotation, even at high dosages.

Apatite flotation was performed at pH = 11.5. Equilibrium diagrams of species in the liquid phase predict the following sequence, in decreasing concentrations:

- barium: Ba^{+2}, $Ba(OH)^{+}$, calcium: Ca^{+2}, $Ca(OH)^{+}$, magnesium: $Mg(OH)_{2(S)}$, Mg^{+2}, $Mg(OH)^{+}$, fluoride: F^{-}, phosphorus: HPO_4^{-2}, PO_4^{-3} and fatty: $RCOO^{-}$, $(RCOO)_2^{-2}$, $(RCOO)_2H^{-}$ and $RCOOH_{(aq)}$.

The concentration of hydroxy-complexes in the liquid phase of apatite flotation is higher than that in barite flotation due to the higher pH level in the former system.

An evaluation of the influence of ions on the flotation of barite and apatite provided the suggestion of tolerance limits for these ions expressed in Table I.

The next step was evaluating of the combined effect of interfering ions. No significant changes were detected in the tolerance limits for barite and apatite flotations (Tables 2 and 3, respectively).

The quality of water for flotation was established based on the tolerance limits for apatite, lower than those for barite. A water recirculation project for Serrana's concentrator was developed, resulting in a closed water system.

A system for monitoring and controlling the quality of recirculated water in terms of ions content (magnesium, calcium, fluoride and phosphorus), illustrated in Figure 5, was developed.

Water recirculation reduced the phosphorus concentration in the receiving body of Serrana's wastewater (Capivara river) to values lower than the background legal limit 0.22 mg/l (Figure 6).

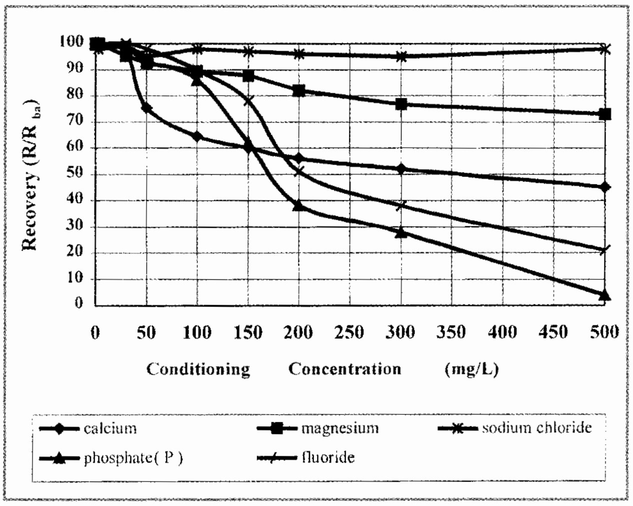

Figure 3. Influence of ions on barite flotation.

CONCLUSIONS

Tolerance limits of ions in barite flotation: magnesium 30 mg/l; calcium 30 mg/l; phosphate 40 mg/l (expressed as P); fluoride 50 mg/l.

Tolerance limits of ions in apatite flotation: magnesium 30 mg/l; calcium 20 mg/l; phosphate 10 mg/l (expressed as P); fluoride 10 mg/l.

The utilization of water containing ions at their limit tolerance concentration did not impair the flotation processes of barite and apatite.

The quality of process water to be recirculated was defined and a monitoring system for ions was developed.

A water recirculation project was developed resulting in a closed water system.

The concentration of phosphorus ions in the receiving body of the plant wastewater was brought down to a value below the legal limit.

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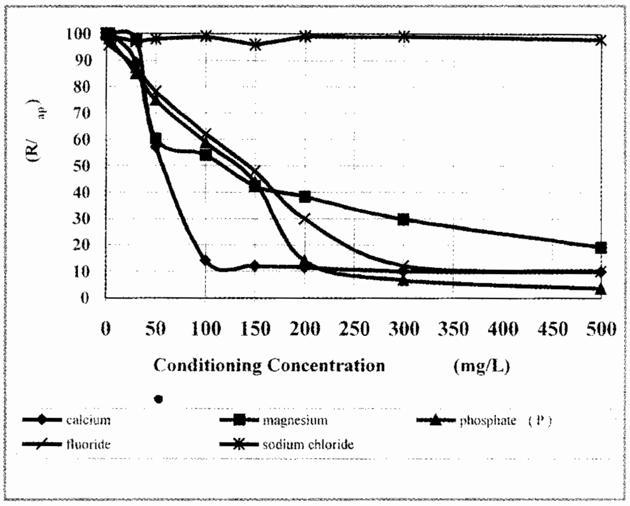

Figure 4. Influence of ions on apatite flotation.

Interfering ion	Limit concentration Barite	(mg/l) Apatite
Fluoride	50	10
Phosphate(*)	40	10
Calcium	30	20
Magnesium	30	30
Rice bran oil soap	5	-
Remark: * - Phosphorus concentration is expressed as P		

Table 1. Limit concentrations of interfering ions in batch column flotation of barite and apatite.

	Item	Unit	Condition	
			New water	Limit
1	Concentration			
	. fluoride	mg/l	0.02	50
	. phosphate(*)	mg/l	0.10	40
	. calcium	mg/l	3.4	30
	. magnesium	mg/l	2.9	30
	. rice bran oil soap	mg/l	0	5
2	Metallurgical balance			
	. floated			
	. barite recovery	%	83.2 (0.6)	83.5 (0.7)
	. apatite loss	%	1.7 (0.1)	1.9 (0.2)
	. grades: $BaSO_4$	%	82.4 (0.5)	82.1 (0.4)
	P_2O_5	%	3.5 (0.03)	3.8 (0.04)
	. non floated			
	. grade: $BaSO_4$	%	1.5 (0.02)	1.2 (0.03)

Table 2. Combined effect of interfering ions on batch column flotation of barite.

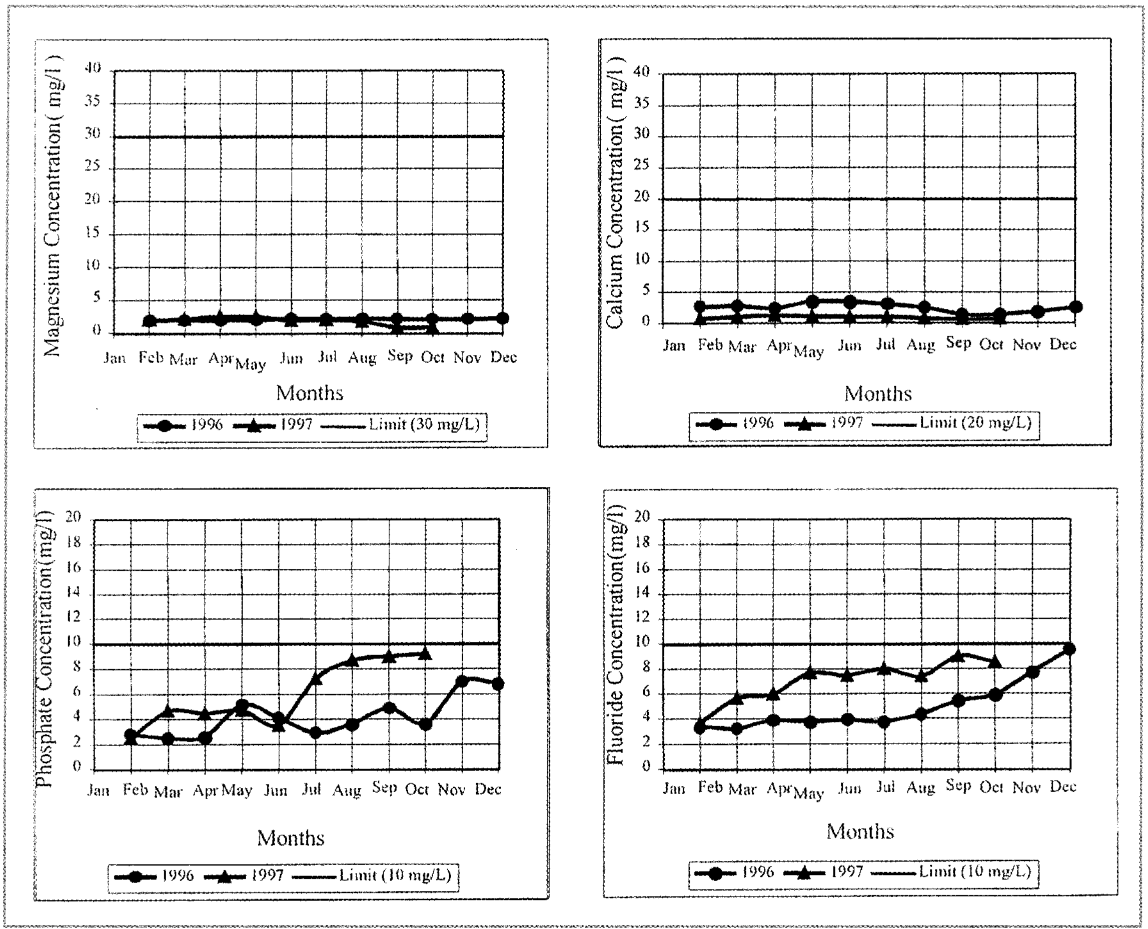

Figure 5. Quality of the recirculated water.

Item	Unit	Condition	
		New water	Limit
1 Concentration			
. fluoride	mg/l	0.02	10
. phosphate(*)	mg/l	0.10	10
. calcium	mg/l	3.4	20
. magnesium	mg/l	2.9	30
2 Metallurgical balance			
. floated			
. apatite recovery	%	69.8 (0.61)	70.0 (0.61)
. barite recovery	%	64.1 (0.71)	63.9 (0.81)
. grades: P_2O_5	%	35.3 (0.05)	35.6 (0.3)
Fe_2O_3	%	4.9 (0.05)	4.7 (0.03)
$BaSO_4$	%	1.7 (0.03)	2.0 (0.04)

Remarks: - the results correspond to the mean of 5 tests - the value in brackets corresponds to the standard deviation
* - phosphorus concentration expressed as P

Table 3. Combined effect of interfering ions on batch column flotation of apatite.

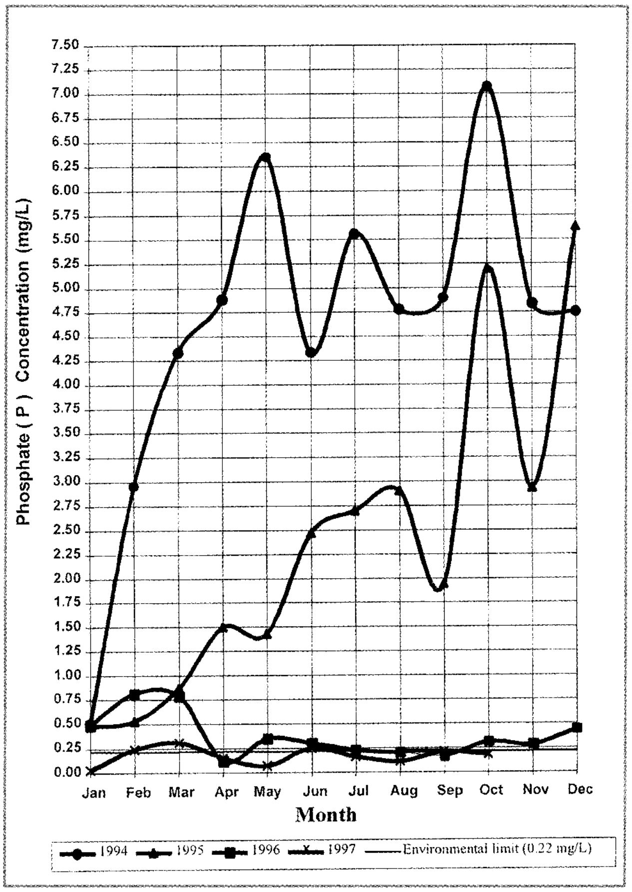

Figure 6. Phosphorus monitoring in Capivara river.