FROTHER ASSISTED AMINE FLOTATION OF IRON ORES[1]

Ronney Rogério Rodrigues Silva[2]
Armando Correa de Araujo[3]
José Farias de Oliveira [4]

Abstract

The main purpose of this work is to describe the experience obtained from laboratorial, pilot plant and industrial test runs of cationic reverse flotation of iron ores in the presence of both amine and frothers. Enhanced iron recovery is the main gain obtained from the synergic interaction of amines and frothers when jointly applied for the flotation of itabiritic iron ores. Laboratory scale tests were performed employing several frother/amine ratios as well as different reagents. Pilot scale test runs were performed to establish and optimize frother/amine ratios for best overall performance. Industrial long-term test runs confirmed the results obtained at bench and pilot scales. Increased iron recovery and separation efficiency were obtained at industrial scale test runs when amine collector addition was partially replaced by a alcoholic frother. These industrial results confirmed previous tests performed at bench scale and at pilot scale on different iron ore samples and using several collectors and frothers. Overall, metallurgical balances demonstrate the efficacy of replacing up to 10% in weight of the collector by a commonly used straight chain alcoholic frother.
Key words: Flotation, Iron Ore, Frother.

FLOTAÇÃO DE MINÉRIO DE FERRO COM AMINAS NA PRESENÇA DE ESPUMANTE

Resumo

A finalidade principal deste trabalho é descrever a experiência obtida através dos testes realizados em laboratório, planta piloto e dos testes realizados em escala industrial para a flotação catiônica reversa de minério de ferro na presença de amina e dos espumantes. O aumento da recuperação de ferro é o ganho principal obtido da interação sinérgica das aminas e dos espumantes quando aplicada conjuntamente para a flotação de minérios de ferro itabiríticos. Os testes em escala de laboratório foram executados utilizando diversas relações entre espumante/amina e diversos reagentes. Os testes em escala piloto foram executados para estabelecer e aperfeiçoar as relações do espumante/amina. Os testes industriais confirmaram os resultados obtidos no laboratório e em escala piloto. O aumento da recuperação de ferro e da eficiência de separação foi obtido em todas as etapas realizadas, ou seja, testes laboratoriais, planta piloto e industriais. Os resultados dos testes industriais confirmaram os testes precedentes executados em de laboratório e piloto mostrando que pode haver uma substituição de até 10% de amina por espumantes.
Palavras-chave: Flotação; Minério de Ferro; Espumante.

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[2] *B.Sc. Mining, M.Sc., Dr., Sandwell Engineering, Belo Horizonte, MG.*
[3] *B.Sc. Mining, M.Sc., Ph.D., Associate Professor, Dept. Mining Eng., UFMG, Belo Horizonte, MG.*
[4] *B.Sc. Mining, M.Sc., Dr. Professor, COPPE, UFRJ, Rio de Janeiro, RJ.*

1 INTRODUCTION

Recent studies demonstrated that a synergic interaction exists between a collector (etheramine) and frothers (non-ionic surfactants). The synergic interaction takes place when certain frothers are used. Deleterious effects of adding certain frothers to the system can occur as well.
Industrial application shows the possibility of expressive gains once the interaction between a frother and the collector would enhance flotation performance and at the same time would reduce the total costs of reagents. Frothers are usually less expensive than etheramines.
The studies reported herein have the main objective the demonstration of technical and economical feasibility of applying frothers in combination with etheramines for the Vargem Grande beneficiation plant of Vale in Nova Lima, Minas Gerais. Most applications of cationic reverse flotation of iron ores currently employ collector only for performing a dual role of making quartz surfaces hydrophobic and stabilizing the froth.(1)

2 MATERIALS AND METHODS

Testing took place at all scales, starting on bench scale flotation tests, passing through pilot plant scale and eventually reaching the industrial scale during two long-run demonstration campaigns.

2.1 Laboratory (Bench) Scale Tests

A sample from Vargem Grande homogenization stockpile was collected. This sample was separated in size at 0.15mm and the undersize fraction was deslimed. The deslimed portion of the sample passing through the 0.15mm sieve was employed on the bench scale test program.
Bench scale tests were performed on a Denver D12 laboratory flotation machine. An initial percent solid was fixed at 40% by weight. Throughout the tests, the cell was rotated at 1500 rpm and pH was kept constant at 10.5. Cornstarch (iron oxides depressant) was used at a dosage of 0.72 kg/t for all tests. A primary etheramine was used as collector and the following surfactants were employed as frothers: pine oil (YSEROL), alcohol blends containing heavy aldehydes and esters (MONTANOL), and ethers (BENEWET). Tests were performed replacing etheramines by the surfactants in the following weight proportions: 0%, 10%, 15%, 20% and 30% at a fixed collector dosage of 40g/t (0.04kg/t). Table 1 details the bench scale program.

2.2 Pilot Scale Tests

Another sample, weighing 8000 kg, was collected at Vargem Grande plant for pilot plant test runs (flotation feed, after desliming and before reagent addition).
Pilot test were performed on a row of four DENVER/DARMA cell of 40L capacity each. Other details of the pilot tests are described in Table 2.

Table 1 – Bench scale test program

Test	Etheramine	Frother	Replacement	pH	Dosagem (g/t) _ Total / amine / frother	Preparation of solution
1	EDA 3B	--	--	10.5	28 / 28 / --	Jointly
2	EDA 3B	--	--	10.5	32 / 32 / --	Jointly
3	EDA 3B	--	--	10.5	34 / 34 / --	Jointly
4	EDA 3B	--	--	10.5	36 / 36 / --	Jointly
5	EDA 3B	--	--	10.5	40 / 40 / --	Jointly
6	EDA 3B	Montanol 800	10%	10.5	40 / 36 / 4	Jointly
7	EDA 3B	Montanol 800	15%	10.5	40 / 34 / 6	Jointly
8	EDA 3B	Montanol 800	20%	10.5	40 / 32 / 8	Jointly
9	EDA 3B	Montanol 800	30%	10.5	40 / 28 / 12	Jointly
10	EDA 3B	Yserol 60	10%	10.5	40 / 36 / 4	Jointly
11	EDA 3B	Yserol 60	15%	10.5	40 / 34 / 6	Jointly
12	EDA 3B	Yserol 60	20%	10.5	40 / 32 / 8	Jointly
13	EDA 3B	Yserol 60	30%	10.5	40 / 28 / 12	Jointly
14	EDA 3B	Benewet 685P	10%	10.5	40 / 36 / 4	Jointly
15	EDA 3B	Benewet 685P	15%	10.5	40 / 34 / 6	Jointly
16	EDA 3B	Benewet 685P	20%	10.5	40 / 32 / 8	Jointly
17	EDA 3B	Benewet 685P	30%	10.5	40 / 28 / 12	Jointly

Table 2 – Pilot plant test program

Test	Conditions								
	Reagentes							Feed	
	Collector	Frother	Depressant	Starch/NaOH	Dosage (g/t) Depressant	Replacement level (%)	Dosage (g/t)	%SOL	kg/h
01	EDA3B	Montanol 800	Amisol	04:01	720	0	45	40	300
02	EDA3B	Montanol 800	Amisol	04:01	720	15	45	40	300
03	EDA3B	Montanol 800	Amisol	04:01	720	20	45	40	300
04	EDA3B	Montanol 800	Amisol	04:01	720	30	45	40	300

2.3 Industrial Trials

Two long-term industrial trials were carried out at Vargem Grande plant. The first trial lasted for 8 days spanning from the end of April to the beginning of May of 2006. The second trial was longer with a total of 19 days, during the months of August and September of 2006. Collector was replaced by a frother at weight ratios of 5%, 10% and 20%.
The chemicals used during the industrial trials were:

- Frother: blend of alcohols, ester and aldehyde.
- Collector: etheramine of 20% neutralization for the first trial and 30% for the second trial.

Chemical assays were obtained at 2 hours intervals for tailing and concentrate streams and every shift (6 hours) for the feed stream, following routine procedure at this plant. Vargem Grande flotation circuit operates two independent parallel lines with rougher, cleaner and scavenger flotation columns of equal size. The trials used one of the two lines.

3 RESULTS

3.1 Sample Employed on Bench Scale Tests

Chemical assays of the sample collected for this part of the research are shown in Table 3.

Table 3. Chemical assays for bench scale raw sample

Grades (%)					
Fe	SiO_2	Al_2O_3	P	Mn	LOI
64.48	4.30	1.64	0.063	0.045	1,80

This sample was them submitted to size separation at 0.15mm and the material passing this size was deslimed by a standard procedure the involves agitation and decantation for a pre-established period of time in order to remove particles below 0.010mm. Table 4 shows the results of the desliming of this sample.

Table 4. Laboratory desliming.

Mass Split		Grades (%)					Distribuition (%)				
Items	%	Fe	SiO_2	Al_2O_3	P	LOI	Fe	SiO_2	Al_2O_3	P	LOI
Analyzed head		60,59	8,06	2,65	0,087	2,503					
Calc. Head	100,00	61,29	7,99	2,35	0,074	2,288					
Deslimed	81,66	63,77	7,85	0,58	0,027	0,865	84,95	80,31	20,08	30,16	30,88
Slimes	18,34	50,27	8,57	10,25	0,282	8,620	15,05	19,69	79,92	69,84	69,12

The fraction deslimed was the feed of all bench scale flotation tests. The chemical characteristics of the deslimed feed are in accordance to normal plant practice at Vargem Grande flotation plant.
Size distribution of the slimes indicated a d_{95} of 0,012mm, a bit coarser than the value predicted by settling velocity.

3.2 Sample Used for Pilot Plant Test Runs

The chemical assays for the sample used during the pilot scale test program are shown in Table 5. Once again, the values for the sample are compatible with the ones normally practiced at Vargem Grande plant.

Table 5. Chemical assays for pilot plant sample

Grades (%)					
Fe	SiO_2	Al_2O_3	P	Mn	LOI
63.13	7.21	1.02	0.040	0.127	1.15

3.3 Bench Scale Tests

At bench scale, tests were performed with the following reagents:

- Montanol 800;
- Yserol 60;
- Benewet 685P.

For each chemical tests were performed varying the replacement ratio of collector at levels 10%, 15%, 20% and 30% by weight. For instance, for 10% replacement, the collector (etheramine) dosage was 36 g/t and the frother dosage was 4g/t, for a combined dosage of chemicals of 40g/t. For comparison reasons a curve, spanning from 28 to 40g/t of collector dosage was obtained. Each level of replacement was them compared graphically using several performance parameters. These results are shown in Figures 1 to 6.

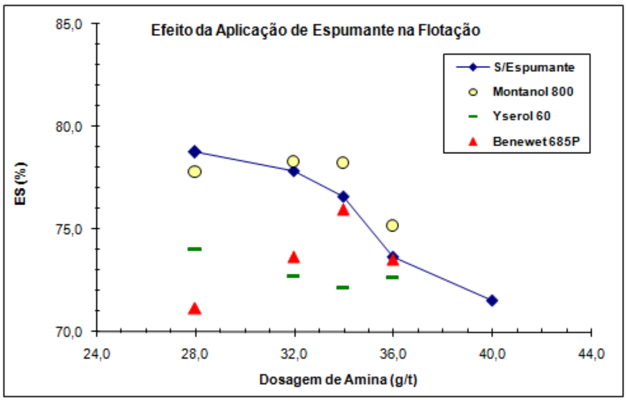

Figure 1. Separation Efficiency (espumante = frother)

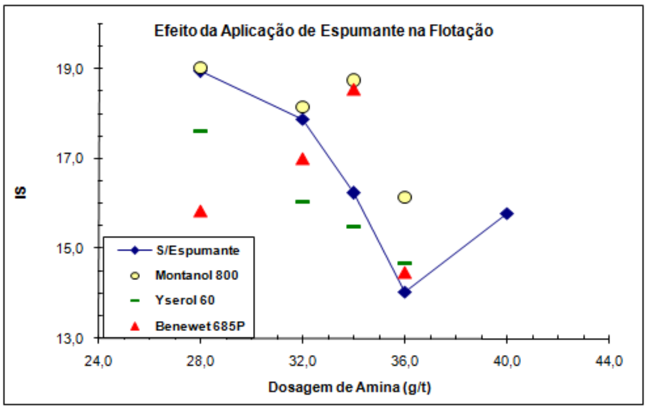

Figure 2. Selectivity Index (espumante = frother; s/espumante = without frother)

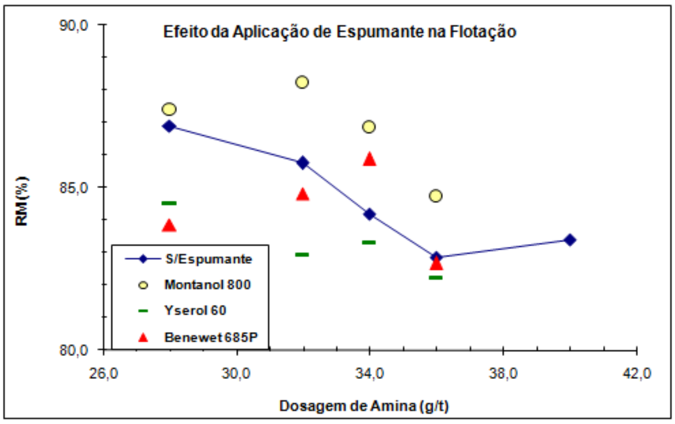

Figure 3. Yield (RM) to concentrate

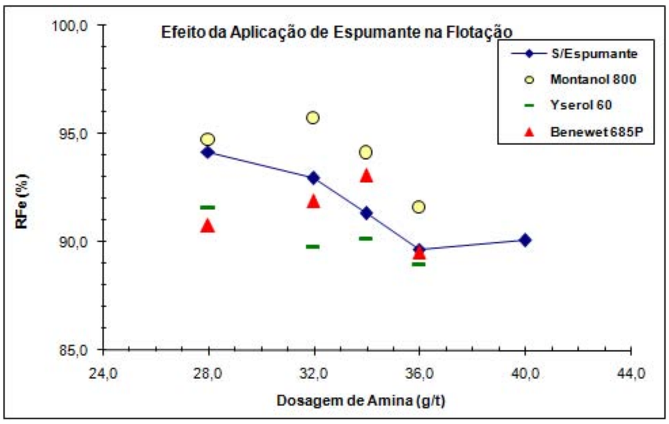

Figure 4. Iron recovery (R Fe)

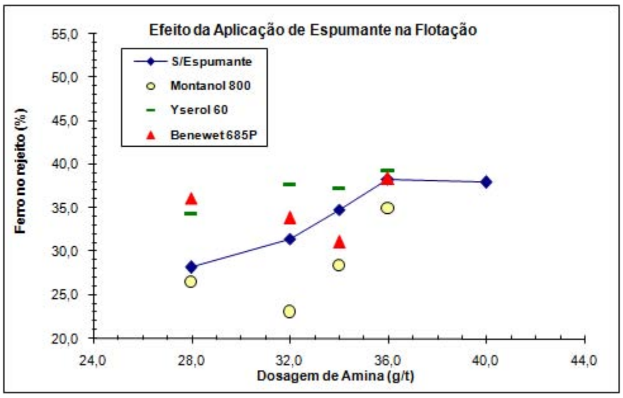

Figure 5. Fe grade in tailings

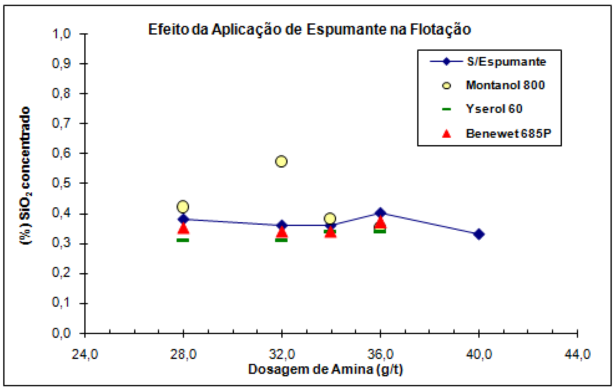

Figura 6. SiO_2 grade in concentrate

In terms of separation efficiency (SE), Montanol 800 presented the best results among the frothers tested. Better SE values were maintained even for levels of replacement of 20%. For Benewet, SE values were similar to the curve without frother substitution for collector. Good results were not obtained when Yserol was used to replace the collector.
Taking into account the selectivity index (SI), two of the chemicals tested resulted in improvement of this parameter, namely Montanol and Benewet. Montanol once again kept improving the selectivity for all replacement levels tested. For Yserol, SI improved up to 10% replacement level.
Comparing yield and iron recovery one more time Montanol gave the best results. Yserol, also once again, did not show any improvement now in terms of yield and iron recovery.
The assay of iron on the tailings confirmed once more the best performance of Montanol among the reagents tested. Benewet also reduced Fe of the tailings up to 15% replacement level. For Yserol, iron grades of the tailings increased.
Regarding the silica grade of the concentrates, they were kept low in all cases, well below the maximum allowed silica content of 1.5%.
Hence, Montanol 800 was selected as the reagent to be tested at pilot plant scale.

3.4 Pilot Plant Testing

The conditions selected for the pilot plant were similar to the conditions employed at bench scale. Frother replacement took place at 15%, 20% and 30% for a total dosage of 45g/t. Cornstarch additions were made at 720g/t and the pH was kept at 10.5 throughout the pilot runs. Test results are shown in Figures 7 to 10.

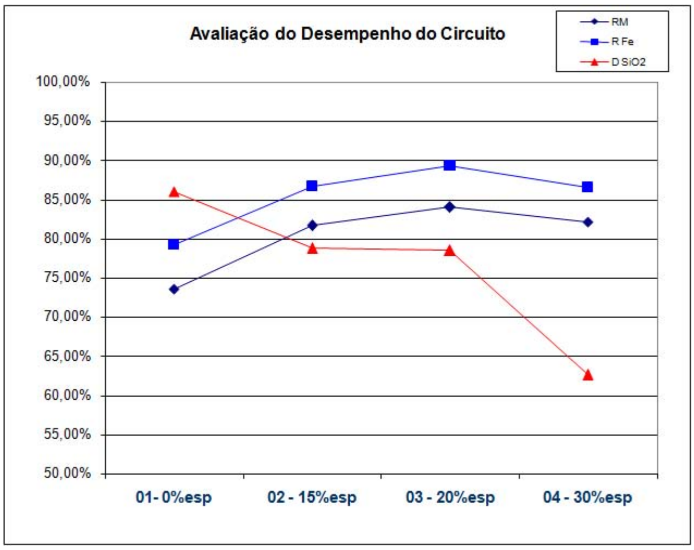

Figure 7. Yield (RM), iron recovery (R Fe) and sílica distribution to the froth phase (D SiO2)

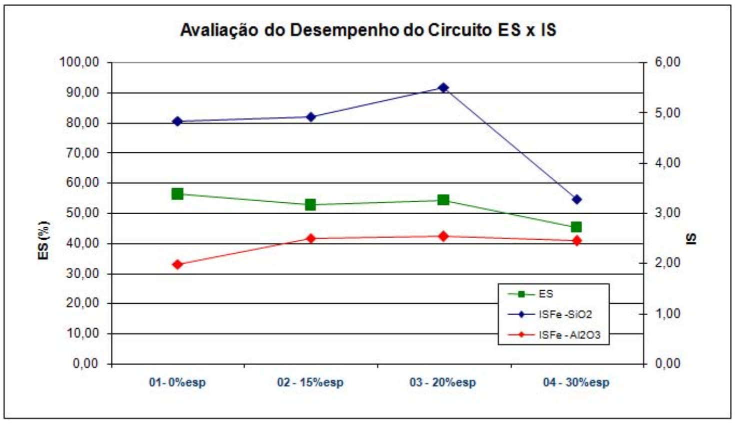

Figure 8. Separation Efficiency (ES) and Selectivity Index (IS)

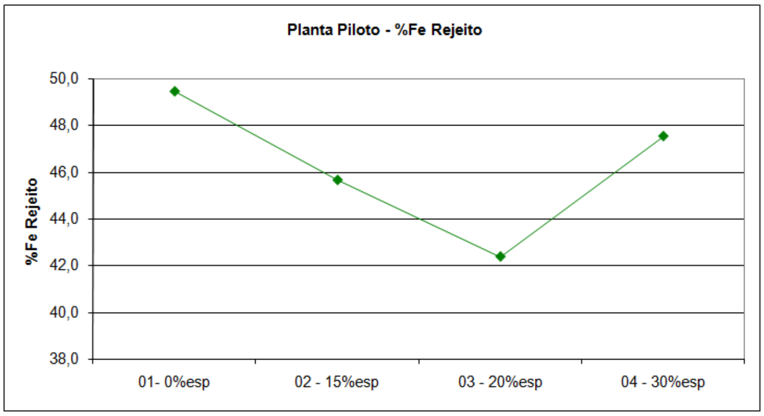

Figure 9. %Fe in tailings – Pilot Plant

Best results were encountered for replacement levels of 15% and 20%. The best level of substitution was found to be 20%.

Yield and iron recovery were both at greater levels for 20% replacement in comparison to all other conditions. Yield for 20% frother replacement reached 84.02 in comparison to 73.61% without frother. Iron recovery reached 89.29%, more than 10% points higher than in the condition without frother (79.24%). Selectivity was also improved for the tests with 20% replacement of collector by frother.

The best parameter reached for the test with 20% replacement was the tailings iron grade. On the other hand, silica in the concentrate increased as the amount of frother added also increased.

3.5 Industrial Plant Trials

Industrial tests were carried out at Vargem Grande Beneficiation Plant in two stages:

- First Stage – The collector used was etheramine acetate with 20% neutralization, supplied by Tomah (currently Air Products) PA 14 that was the standard collector during the months of April and May of 2006. The frother used in the tests was Montanol 800.
- Second Stage – At the time of the second trial (August to September of 2006) the plant was employing another etheramine as collector, also an etheramine but with 30% neutralization, supplied by Clariant (EDA 3B). The frother employed was the same as before, i.e., Montanol 800.

Tables 6 and 7 show some of the parameters for the trials during the first stage of industrial testing.

Table 6. Average Results of Plant Trials – First Stage (April-May of 2006)

Média	Feed		Concentrate		Tailings	
	%Fe	$\%SiO_2$	%Fe	$\%SiO_2$	%Fe	$\%SiO_2$
Without Frother	64,92	5,04	67,63	1,40	58,49	13,14
10% replacement	**65,43**	**4,91**	**67,74**	**1,46**	**54,15**	**19,15**
20% replacement	**64,83**	**5,08**	**67,44**	**1,39**	**59,44**	**12,07**

Table 7. Average of Evaluation Parameters – First Stage (April-May of 2006)

Average	Results			Re		SI	
	RFe	Yield	$DSiO_2$	ReFe	$ReSiO_2$	$Fe\text{-}SiO_2$	ES
Without Frother	67,96%	65,22%	79,93%	1,04	0,30	3,39	37,76
10% replacement	80,78%	77,82%	73,55%	1,04	0,33	4,16	44,96
20% replacement	63,62%	61,21%	80,22%	1,04	0,29	3,16	32,73

Analyzing the results from both tables, the best overall condition of that involving replacing the collector with 10% of Montanol 800.

Silica grade of the concentrates were similar for all conditions, with or without the utilization of frother. In terms of iron recovery, for the trial with 10% replacement the highest values were encountered in comparison with the trials without frother or with 20% replacement.

Analyzing other parameters such as separation efficiency (SE), coefficient of separation (CS) and selectivity index (SI) the conclusion is the same, i.e., the best overall performance took place for 10% replacement.

Thus, comparing the average results the iron recovery without frother was approximately 68% whereas for 10% replacement it reached, in average, approximately 81%, na increase of almost 13% points for basically the same outcome in terms of silica grade in the concentrate.

For the second stage of industrial trials, replacement of collector by frother was kept at the levels of 5%, 10% and 20%.

For this trial, the best results were obtained at 5% replacement. Yield and iron recovery increased significantly although silica grade also increased in terms of

average for the tests employing frother. Iron grade in the tailings decreased also significantly for 5% replacement level. These average results are summarized in Table 8. The increase in iron recovery can be shown to be statistically significant.
Other evaluation parameters such as separation efficiency and selectivity index also demonstrate that for the second stage of plant trial the best results were achieved for 5% replacement of the collector (also see Table 8).

Table 8. Average results – second stage of industrial tests.

Description / Average		Without	5% frother	10% frother	20% frother
Feed	%Fe	62,32	61,03	62,32	63,28
	$\%SiO_2$	8,55	9,90	8,48	7,23
Concentrate	%Fe	67,53	66,97	66,96	67,27
	$\%SiO_2$	1,30	1,71	1,92	1,69
Tailings	%Fe	55,83	50,13	56,70	58,95
	$\%SiO_2$	17,19	24,51	15,66	12,77
Recovery and Yield	RFe	53,02%	66,75%	53,72%	51,96%
	RM	49,03%	61,11%	50,10%	48,91%
	$DSiO_2$	91,60%	87,45%	86,74%	87,03%
Re	ReFe	1,09	1,10	1,08	1,06
	$ReSiO_2$	0,16	0,20	0,24	0,26
SI	$Fe\text{-}SiO_2$	4,14	4,54	3,14	2,97
ES		36,83	44,61	33,32	31,58

Tables 9 and 10 show selected results in order to highlight the differences obtained by partially replacing the collector with a frother. Tables 9 assembles the tests in which the silica feed grades with and without frother are similar. Table 10 puts together the tests in which concentrate silica grades are similar.
Analyzing the data on both tables 9 and 10 it is clear that iron recovery for 5% replacement, for a same concentrate silica grade, are indeed higher than for all other test conditions. In numerical terms, for the average condition without frother and with a silica grade of approximately 1.3%, iron recovery was 53%. The same concentrate was obtained by a combination of 5% frother and 95% collector at an iron recovery of over 67%, 14% points higher than without the assisted flotation condition with the frother.

Table 9. Second stage – Averages for the same feed silica grade

Description / Average		Without	5% frother	10% frother	20% frother
Feed	%Fe	61,31	61,03	62,32	63,28
	$\%SiO_2$	9,96	9,90	8,48	7,23
Concentrate	%Fe	67,48	66,97	66,96	67,27
	$\%SiO_2$	1,40	1,71	1,92	1,69
Tailings	%Fe	54,90	50,13	56,70	58,95
	$\%SiO_2$	18,52	24,51	15,66	12,77
Recovery and Yield	RFe	48,33%	66,75%	53,72%	51,96%
	RM	43,95%	61,11%	50,10%	48,91%
	$DSiO_2$	93,42%	87,45%	86,74%	87,03%
Re	ReFe	1,10	1,10	1,08	1,06
	$ReSiO_2$	0,15	0,20	0,24	0,26
SI	$Fe\text{-}SiO_2$	4,22	4,54	3,14	2,97
ES		35,66	44,61	33,32	31,58

Table 10. Second stage – Averages for the same concentrate silica grade

Description / Average		Without	5% frother	10% frother	20% frother
Feed	%Fe	62,32	61,63	62,71	63,32
	$\%SiO_2$	8,55	9,28	7,95	7,21
Concentrate	%Fe	67,53	67,28	67,43	67,54
	$\%SiO_2$	1,30	1,33	1,30	1,32
Tailings	%Fe	55,83	50,29	58,39	59,20
	$\%SiO_2$	17,19	24,36	13,14	12,61
Recovery and Yield	RFe	53,02%	67,28%	45,67%	49,97%
	RM	49,03%	61,92%	42,52%	46,94%
	$DSiO_2$	91,60%	88,24%	91,58%	90,27%
Re	ReFe	1,09	1,09	1,08	1,07
	$ReSiO_2$	0,16	0,18	0,19	0,20
SI	Fe -SiO_2	4,14	4,94	3,37	3,29
ES		36,83	45,66	30,43	31,96

4 DISCUSSION

The tests results of the first stage demonstrated good performance with substitutions of 10 % and the second stage with substitutions of 5%. This difference maybe explained by the type of amine that was used in each stage. In the first stage, an etheramine with 20% of neutralization (acetic acid) was used, that is, with a greater amount of active amine whereas in the second stage an etheramine with 30% of neutralization degree was employed.
The industrial application at Vargem Grande beneficiation plant demonstrated its fully feasibility and ease of application. In Table 11 and Figure 10, the possibilities of revenues for the Vargem Grande plant are presented. The data used as reference were obtained in the period spanning from January to September of 2006.
Considering a 10% replacement of amine by frothers it can be observed a direct savings of R$ 60,740 per year. When the PFF production increase is taken into account, reflected by the increase in flotation recovery, extra revenues of approximately R$ 11.7 million per year are reached. Figure 19 shows the increased revenue that can be directly associated with increases in yield (mass recovery).
The studies presented in the previous items had shown that the use of frother could promote an increase in yield (mass recovery) greater than 10% (see figure 10).
Increased recoveries when the collector was partially replaced by a frother as Montanol is on line with several other works performed in the field with other ores (4-14) and other collector-frother systems (15-20).

5 CONCLUSIONS

Selection of the best frother was carried out from the analyses of bench scale and pilot scale test programs
Industrial trials at two different operational conditions at Vargem Grande beneficiation plant have demonstrated that up to 10% replacement of collector by a frother can be achieved with improved overall results. Etheramine with smaller neutralization degree allowed for larger replacement than etheramine with higher degree of neutralization.

Table 11 - Revenue increase with frother assisted flotation at Vargem Grande Plant

Description		Without Frother	With Frother
Feed (t x 1000)	Flotation	2.696	2.696
Flotation yield		61,00%	71,00%
PRODUCTION	PFF	1.644	1.914
Specific consumption (g/t)	Surfactant	59,4	59,4
(%)replacing amine per frother		--	10,00%
specific consumption (g/t)	Amine	59,4	53,5
reagents consumption (kg)	Amine	160.121	144.109
specific consumption (g/t)	FROTHER	--	5,9
reagents consumption (kg)	FROTHER	--	16.012
PMU (R$/kg)	Amine	7,59	7,59
PMU (R$/kg)	FROTHER	--	3,79
Reagents costs (R$)	Total	1.214.809	1.154.069
direct savings for using the frother(R$)		60.740	
PFF sell value (R$/t)		43,20	
PFF -(Difference)		269.566	
PFF - R$ (Difference)		11.645.234	
TOTAL revenue increase utilizing frother (R$)		*11.705.974*	

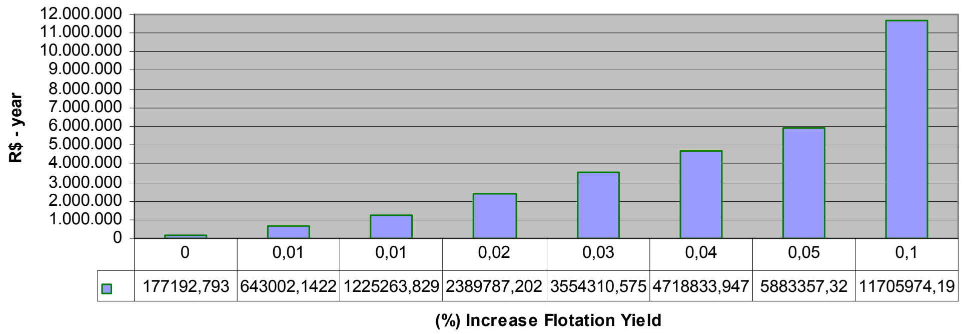

Figure 19. Yield increase impact on revenues

The industrial application of frother assisted amine flotation is feasible and its implementation is very ease once there is no need for separate preparation or conditioning of the frother.

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