RECYCLING OF AMINES IN THE REVERSE FLOTATION OF IRON ORES

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ABSTRACT

The reverse flotation of iron ores is the usual process for the beneficiation of iron ores. In this case the silica particles are floated with amines. Usually this pulp, including the main part of amines, is deposited in the tailing dams. In the best case a part of the amine free water is reused as process water after a long residence time. The recycling of the amines has not yet been investigated until now. This was the starting point for the investigations forced to prove the possibility of recycling the amines from the float product. As a result of the first tests it was discovered that the float product contains a significant concentration of amines, which can be generally recycled for the further flotation process. By using the recycled amines, the same flotation results were noticed by saving nearly 50 % of the collector amount.

INTRODUCTION

Brazilian iron ore deposits are the six major deposits in the world. However the Fe- high ores (hematite ores with 60 to 67 % Fe and Itabirite ores with 50 to 60 % Fe) place Brazil at an even higher classification in terms of Fe-rich countries. World iron ore production in 1998 was about 1 billion tons. Brazil was classified with 199 million tons or 19,2 % and rated as the second largest producer. Maybe it is the first producer of iron ores that are beneficiated. From this production 75,5 % were produced in Minas Gerais state. In 1998, 150 million tons of iron ore and pellets were exported which produced 3.25 billions dollars (DNPM 1999). The rich iron ores normally are only comminuted and classified. The low-grade iron ores like Brazilian Itabirite have to be concentrated using gravity and magnetic concentration or flotation. Froth flotation is the most important concentration method, which is utilized in poor iron ore concentration. This method can be classified in direct flotation when the iron oxides are floated or reverse flotation when the silica's gangue are floated (Houot 1983). Normally the reverse flotation with amines is used (Iwasaki 1983 and Numela & Iwasaki 1986) These amines ionize in water solution by protonation, like in the reaction below,

$$RNH_2(liq.) + H_2O \rightleftarrows RNH_3^+ + OH^- \quad (1)$$

At saturated systems,

$$RNH_2(s) \rightleftarrows RNH_2(liq.) \quad (2)$$

The amine salts not substituted are totally dissociated at acid neutral and weak basic medium. Quaternary ammonium salts are established and completely dissociated at all pH values (Leja 1983). The amine solubility is reversibly related to the hydrocarbon size chains. The amines with a carbon chain up to 12 atoms, generally are liquids. The ether group introduction and the amines partial neutralization using acetic acid and hydrochloric acid cause the amine solubility increase.

The adsorption of etheramine acetate on to hematite and quartz surfaces at pH 10.5 happens by electrostatic attraction of etheramonium cation, on mineral surfaces that have negative surface charges in this pH value, and van der Walls bonds between etheramonium ions and etheramine molecules. The quartz adsorption density of this reagent is greater than the hematite adsorption density because the quartz negative surface charge in this pH value is greater than the hematite negative surface charge for the same pH value, but the differences are not enough for a selective flotation. For this corn starch, as a depressant for hematite, is used. (Lima and Brandão, 1998).

In the case of reverse iron ore flotation, probably the main part of the amines, which were used, remains in the float product. They are absorbed on the surface of the floated silica particles or are dissolved in the pulp. Usually this pulp is deposited in tailing dams. In the best case after a long residence time a part of water free of amines is reused as process water.

The recycling of the amines makes total sense and it is important because of ecological as well as economical aspects. The expensive amines are a potential threat to the surface waters and money is also wasted in the tailing dam. This was the starting point for the investigations done in the Mining Department at the

Federal University of Ouro Preto, Minas Gerais. The aim was to test the feasibility of recycling the amines from the floated product and to use these amines again for the flotation process.

Samples from the Samarco mine, Mariana, Minas Gerais were used for these investigations. The flotation is carried out as a reverse flotation with starch as a depressor of iron minerals and amines as a collector for silica. The float including the main part of silica and amines of the process is deposited together with other tailings of the mine in a tailing dam at present. The cleared water of this dam is reused as process water in the beneficiation plant (figure 1).

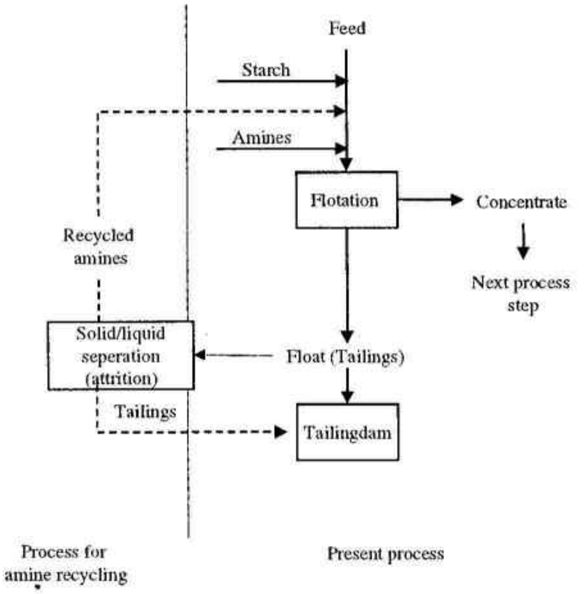

Figure 1: Present process of reverse iron ore flotation including the feasible process of amine recycling

The recycling of the amines in the float has not yet been investigated until now. Also, no related publication about this theme is known. This paper will present the first results of the investigations.

ORE SAMPLE CHARACTERIZATION

The sample was collected at the hydrocyclones underflow that feeds the conventional flotation step. The sample is characterized by following particle size distribution.

The sample size analyses were carried out by wet screening.

Table 1: Particle size distribution

Particle size range [mm]	Amount [%]
+ 0,296	0,91
0,296 – 0,148	7,96
0,148 – 0,074	27,23
0,074 – 0,037	29,32
- 0,037	34,58
Total	100,00

Approx. 100 % of the flotation feed is undersize <0,3 mm. The sample contained approx. 35% undersize < 0,037 mm, see particle size analyses in table 1.

Table 2: Results of Chemical Analysis done by AAS

Elements /.Compounds	Content [%]
Fe	44.44
SiO_2	34.91
Al_2O_3	0.210
P	0.026
Mn	0.017
TiO_2	0.010
CaO	0.012
MgO	0.012

The results of the chemical analysis were done by AAS and are presented in table 2. The content of silica with nearly 35 % is very high. The content of iron is 44 % at the low end of the normal range of the Minerals of Samarco Mining.

TEST PROCEDURE

The flotation tests were carried out with a 2 l Wemco agitation cell containing 1150 g of ore, which results in 45 % of solids in the pulp. The used amounts of reagents refer to 1 t-dried mineral.

pH:	10,5
Collector:	Amine Flotigam EDA B (different concentrations),
Depressor:	Starch 300g/t
Condition times:	3 min dispersing of ore, 5 min conditioning of starch, 3 min conditioning of amines, 5 min flotation.

The tests were carried out according to the following course scheme (figure 2):

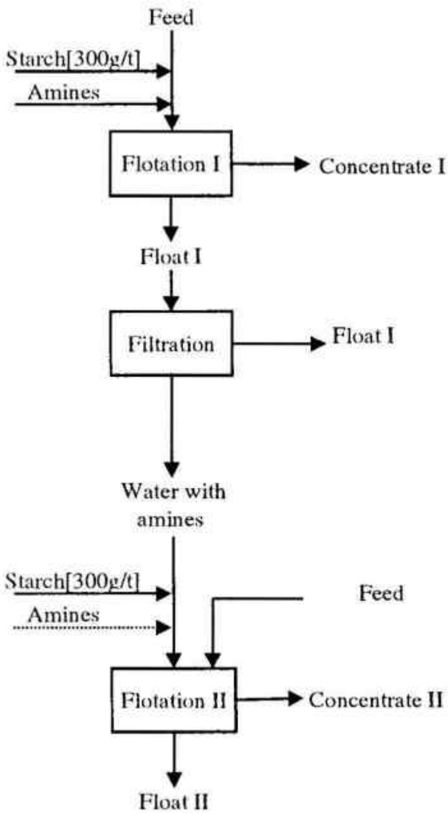

Figure 2: Course scheme of the flotation tests

In this work three different test series were realized. In the first series, flotation tests under varying amine collector amount (30, 40, 50, 80 g/t) were carried out. The concentrates and floats of these tests were dewatered by vacuum filtration. The dried products were analyzed in order to make the mass and metallurgical balances. The filtrate of the float expected contained a more or less large part of the used amines was collected. The water with amines from the first step filtration was used without any new amine addition. The second flotation was made with a new ore sample under the same conditions as in the first flotation step.

In the second test series all tests at the first flotation step were carried out with the same amine concentration (40 g/t). It was chosen because this amine concentration normally is used at the industrial plant. For the second flotation step the filtrate from the first flotation step was used with a new amine addition (0, 10, 20, 30, 40 g/t).

The third series had the aim to study the possibility to recycle the amines more than once. The first flotation step was carried out with 40 g/t amines. The filtrate plus 20 g/t of new amines was used for the second flotation. The float product from the second flotation was also dewatered in a vacuum filter. The water was collected and then used for a third flotation step. For this third flotation this filtrate plus 20g/t new amines were used.

RESULTS

In figures 3 to 5 the iron and silica contents in the concentrate and the iron recovery are presented. It can be observed that the iron content increased from 54% to 66 % with increasing concentration of collector. The opposite happens with the silica content that decreases from 21% at 30 g/t to 3.6 % at 80 g/t collector amount. The recovery of iron decreases with the increasing amount of collector, see curve "new amines". The relatively lower concentrations of iron and higher concentrations of silica in the concentrate can be explained by the high concentration of silica in the feed.

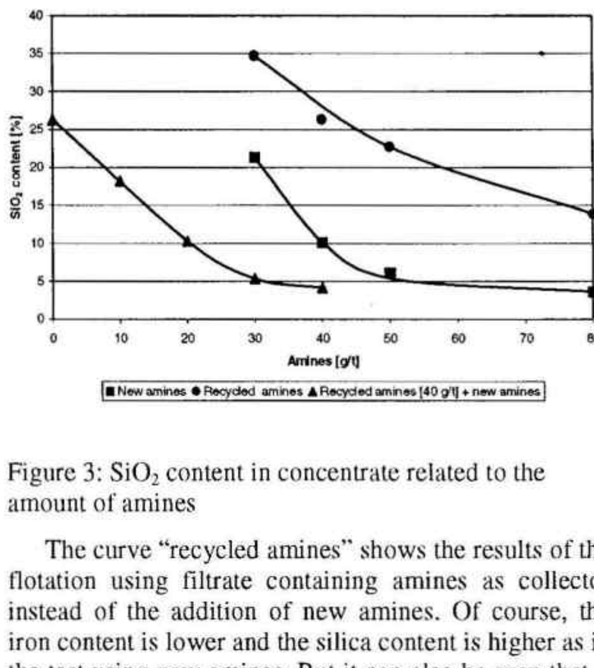

Figure 3: SiO_2 content in concentrate related to the amount of amines

The curve "recycled amines" shows the results of the flotation using filtrate containing amines as collector instead of the addition of new amines. Of course, the iron content is lower and the silica content is higher as in the test using new amines. But it can also be seen that it is possible to float the ore without the addition of any new collector, which shows evidence that the filtrate of the float really contains amines. In other words, with the filtrate from the test using 80 g/t amines we are almost considering the same result as that using 30 to 40g/t of new amines. This tendency can be seen in the concentration of amines up from 40 g/t used in the first step of flotation. This can be observed for the silica content, the iron content and the recovery of iron.

The results of the second series of testes are shown in the curve named "Recycled amines [40g/t] + new amines". By comparing this curve with the results of new amines curve it can be seen that by using the filtrate from the first flotation step (40g/t amines) plus 20 g/t new amines nearly the same result as by using 40g/t new amines was acquired. Under this condition, the concentrate contents are of 10.3 % silica and 61.1 % iron where the recovery of iron is 90.2 %.

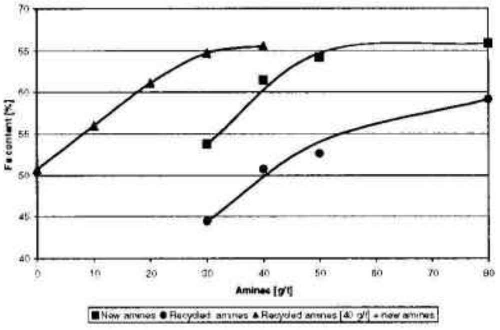

Figure 4: Fe content in concentrate related to the amount of amines

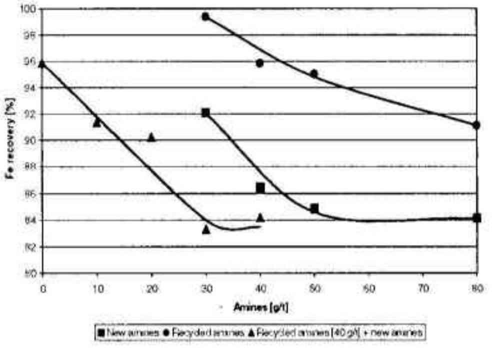

Figure 5: Fe recovery related to the amount of amines

By using the filtrate of the first flotation plus 40 g/t new amines nearly the same results can be reached as using 80 g/t new amines. The contents are of 4.1 % silica and 65.6 % iron where the recovery of iron is 84.2 %. Therefore, nearly the same results can be reached by using only 50 % of the collector when the recycled amines are used.

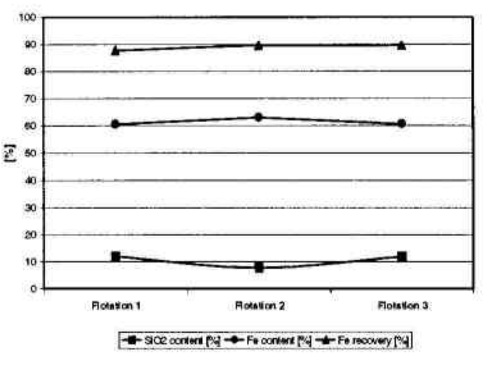

Figure 6: SiO_2 content, Fe content and Recovery of Fe for a 3-step flotation with the recycling of amines two times

Figure 6 shows the results of a 3-step flotation. In the first step 40 g/t amines, in step 2 and 3, the filtrate of the float from the step before plus 20g/t was used. It can be observed that in the third step the same results as in the first step can be obtained. The Silica content at the third step is 11.9 %, iron content is 60.6% and the recovery of iron is 89.55 %. In the first step, with 40 g/t amines, we received a Silica content of 11.9 %, an iron content of 60.5 % and an iron recovery of 87.8 %. It was discovered that it is possible to receive the same result by using the recycled water and only 50% of the amount of collector also in a third time using the recycled amines.

CONCLUSIONS AND OUTLOOK

As a result of the first tests created to prove the possibility to recycle the amines from the float produced in reverse flotation of iron ore by using amines, it was discovered that the float contains a significant concentration of amines, which can be generally recycled for the further flotation process. By using the recycled amines nearly the same flotation results were noticed by saving 50 % of the collector amount. It can be concluded that there is a great opportunity to save the environment and to decrease reagent costs.

In the future, different ores and collectors have to be tested. Especially because of the high silica content of the tested ore. Another aspect is the influence of the amine residence time in the process under special attention of the amine biodegradation. A method to measure the concentration of the amines in the pulp, water, and at the surface of the products also has to be researched. This method is been developed together with the chemical department of the UFOP.

Other important parameters and conditions that have to be tested are the possibility to separate the amines from the surfaces of the flotation product. For this, an attrition process can be used. For this purpose, different attrition conditions, pH and residence time have to be tested. Other aspects that have to be studied are the influence of ultra fine particles from solid/liquid separation. Subsequently, this research is still at the beginning.

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