by

Luis R. M. Bittencourt Magnesita S.A. Research and Development Center Belo Horizonte, Brazil

Chen L. Lin and Jan D. Miller Department of Metallurgical Engineering University of Utah Salt Lake City, Utah 84112, U.S.A.

Abstract. Recently research efforts have been made to obtain a high-purity gibbsite concentrate, suitable for first-class alumina refractories, from a Brazilian bauxite ore containing gibbsite (50%), quartz (35%), and kaolinite (15%). Detailed polished-section microprobe analysis was used to describe the liberation characteristics of the ore. Experimental results suggest that the kaolinite liberation characteristics can be described by King's liberation model and that only about 11% of the kaolinite is expected to be liberated at a particle size of 75  $\mu$ .

Bench-scale experiments for the flotation of gibbsite/kaolinite from quartz using alkyl sulfates as collectors were particularly successful at pH 2, and under appropriate conditions a concentrate containing 93.0% Al<sub>2</sub>O<sub>3</sub> (calcined basis) was obtained at a gibbsite recovery of about 96%. Subsequently, the more difficult separation of kaolinite from gibbsite was accomplished by kaolinite flotation from the alumina concentrate with a quaternary ammonium salt at pH 6 to produce a high-purity gibbsite product, 97.4% Al<sub>2</sub>O<sub>3</sub> (calcined basis), at an overall gibbsite recovery from the bauxite flotation feed of about 90%.

# INTRODUCTION

Aluminum is the third most abundant element in the earth's crust. In nature it occurs only in combination with other elements and is part of the crystal structure of many rock-forming minerals. Bauxite, the most important commercial ore of aluminum, consists of mixtures of aluminum hydroxide minerals and impurities. The three aluminous minerals that form bauxite are gibbsite  $(A1(OH)_3)$ , boehmite (YA10.OH), and diaspore (aA10.OH), any of which may at times be dominant. Besides its main use as an aluminum ore, bauxite is used to manufacture refractory products, alumimous chemicals, abrasives, and miscellaneous applications such as building stones and road surfacing. Also, the demand for high-purity alumina in advanced materials is significantly increasing in many industrial applications. These applications include advanced aluminum alloys for aerospace, *β*-alumina solidstate electrolytes, high-temperature ceramic

Table 1.						
Raw	Materials Used to					
Manufacture	High-Alumina Refractories					

Raw Material	<b>%</b> Al <sub>2</sub> O <sub>3</sub> (Calcined Basis)
Andalusite	58-61
Kyanite	58-61
Silimanite	58-61
Gibbsitic clays	55-72
Synthetic sintered or	
fused mullite	70-74
Bauxites	85-90
Brown fused alumina	96-97
Tabular alumina (sintered)	99
White fused alumina	99

engines, and first-class refractories. There are many different types of raw materials used to manufacture high-alumina refractories, as shown in Table 1.

Tabular alumina and white fused alumina are based on chemical-grade Bayer-type alumina for which the production cost is very high. Brown fused alumina and fused mullite are materials manufactured from regular bauxites by electrofusion, but the price is still very high. Andalusite, silimanite, and gibbsitic clays are raw materials which are used to manufacture 60 to 72% Al<sub>2</sub>O<sub>3</sub> refractories, but they cannot be used in the higher alumina refractory classes (80 to 95% Al<sub>2</sub>O<sub>3</sub>). The first-class alumina refractories which contain more than 90% Al<sub>2</sub>O<sub>3</sub> are used mainly in sliding-gate nozzle systems, alumina graphite tubes for continuous casting, blast furnace bricks, and other demanding applications.

Regular refractory bauxites with a typical analysis of 1.5 to 2.0%  $Fe_2O_3$ , 3.0 to 4.0%  $TiO_2$ , 6.0 to 7.0%  $SiO_2$ , and 87 to 89%  $Al_2O_3$  (calcined basis) are mined and sintered in Guyana and China, and sold all over the world. These materials are much cheaper than tabular or electrofused aluminas (US \$150 per ton for sintered bauxite compared to US \$850 per ton for electrofused alumina); however, due to the association of oxide impurities, they cannot be used as raw materials for first-class refractories. Magnesita S.A. (the largest refractory company in Latin America) owns a very special bauxite deposit known as Rio Pomba bauxite. This bauxite deposit has, as its major impurities, quartz and kaolinite. Other impurities such as  $Fe_2O_3$ ,  $TiO_2$ , and alkali occur in very small percentages (<0.5%). If most of the quartz and kaolinite can be removed by concentration methods, it will be possible to obtain a material with an  $Al_2O_3$  content higher than 95% (calcined basis), which is suitable to be used as raw material for first-class high-alumina refractories, competing with much higher-priced raw materials such as fused and tabular alumina.

#### FLOTATION SEPARATIONS

More than 90% of the world bauxite production is treated by the Bayer process to produce alumina which is reduced to aluminum by the Hall-Herault electrolytic process. The majority of the bauxite mines have been developed to supply bauxite for aluminum production, and in some cases specialgrade bauxites have been produced for other industrial uses. Selective mining of the bauxite is usual for these special grades, and occasionally they require some additional concentration by washing or by flotation.

# Quartz Flotation

and the second of

Quartz flotation is one of the most wellstudied flotation systems due to its common occurrence as gangue material, its availability in pure form, and its low solubility in water. Selective flotation of quartz frequently can be obtained with amines as collectors. These cationic collectors have been widely used in flotation plants to separate quartz from valuable minerals. Many investigations on the study of quartz flotation by amine have been reported and the results summarized in several publications such as Fuerstenau (1976), Leja (1982), and Fuerstenau, Miller and Kuhn (1985). However, most of this work has considered only the weak base primary, secondary, and tertiary amines. Much less attention has been given to the study of quartz flotation with quaternary ammonium salts. 9-24

Also, the anionic flotation of quartz using fatty acids or their alkali soaps has been reported by Gaudin (1957). In this case, the quartz must be activated by polyvalent cations such as iron, calcium, barium, lead, or aluminum. Extensive study of quartz activation by polyvalent cations has been reported in the literature (Fuerstenau, Miller, and Kuhn, 1985).

# Gibbsite/Alumina Flotation

The use of tallowamine as a gibbsite collector has been reported by Huang et al. (1980) and Cheng et al. (1982) for the beneficiation of a Taiwanese bauxite ore where gibbsite is floated from hornblende. Andreev et al. (1972, 1973a, 1975) have discussed the adsorption of laurylamine on gibbsite and chamosite at pH 4 to 8. However, the flotation of gibbsite using amines as collectors cannot be applied in the case of the Rio Pomba bauxite, since a significant amount of quartz (about 35%) is present, which is not the case of the Taiwanese and the Soviet ores mentioned above.

Anionic flotation of gibbsite using fatty acids as collectors has also been investigated by Andreev et al. (1973b). It has been determined that the best flotation response of gibbsite with sodium oleate as collector is achieved at pH 8. Similar behavior has been observed by Hinds et al. (1985). In this case, gibbsite is floated with sodium oleate from a mixture of quartz, kaolinite, and iron and titanium oxides at pH values between 10 and 11.

The use of alkyl sulfates as collectors for minerals which contain aluminum has been widely studied. Modi and Fuerstenau (1960) have observed that corundum can be floated at pH 4 and 6 with sodium dodecyl sulfate (SDDS). However, corundum flotation recovery decreases significantly at pH 9.3, with depression occurring at pH 11, indicating that pH effectively regulates the flotation of corundum through its control of the surface charge. The effect of long-chained neutral molecules (decyl alcohol) in the flotation of corundum with SDDS has been described by Fuerstenau and Yamada (1962). The addition of decyl alcohol increases the flotation recovery of corundum because of the coadsorption of neutral molecules with the collector anions. In another study, Yoon and Salman (1976) concluded that lauryl sulfate chemisorbs at the surface of Y alumina at pH 4.5.

Only recently have in-situ spectroscopic investigations elucidated some of the mechanisms involved in the alumina/SDDS system (Chandar, Somasundaran and Turro, 1987; Miller and Kellar, 1988; Kellar, Cross, and Miller 1989, Somasundaran et al., 1989). In particular it has been demonstrated (Miller and Kellar, 1988; Kellar, Cross, and Miller, 1989) that adsorbed SDDS species have no preferred orientation at the alumina surface. This finding contradicts traditional beliefs that wellordered molecular aggregates termed "hemimicelles" are present on mineral surfaces. Also, it has been found that, at pH values less than 3, the adsorption density of SDDS increases dramatically to form several multilayers of adsorbed collector, reminiscent of collector surface precipitation reactions common to semisoluble salt flotation systems. Such a finding is further substantiated by the batch flotation results discussed later in this paper.

#### Separation of Gibbsite from Kaolinite

Differential flotation for the separation of kaolinite from gibbsite has been reported to be achieved mainly by two different methods. The first one is the anionic flotation of gibbsite using fatty acids or alkyl sulfates as collectors as described above. The depression of kaolinite can be achieved with hexametaphosphate. Balashova and Kuznetsov (1970) have reported a bauxite flotation process where gibbsite is floated with a mixture of oleic acid, machine and tall oils, and 85 to 90% of the kaolinite is depressed with sodium hexametaphosphate at pH 9. Black and Low (1950) have suggested that the depression of kaolinite by phosphate is due to the surface replacement of silicon tetrahedra by phosphorus tetrahedra.

The second method is the cationic flotation of kaolinite using amines as collectors. Brehler et al. (1970) have reported the flotation behavior of kaolinite using amine hydrochloride as collector. They observed that kaolinite can be floated at pH values lower than pH 8 and that a decrease in the hydrocarbon chain length increases the kaolinite content of the concentrate. Kellogg (1946) has also reported the flotation of kaolinite with amines (differential flotation from quartz). In this case, laurylamine hydrochloride was used as collector (pH 3) which was speculated to adsorb at the kaolinite surface by chemical reaction to form laurylammonium silicate.

#### EXPERIMENTAL RESULTS AND DISCUSSION

Characterization and Liberation Studies

The Rio Pomba bauxite is composed basically of gibbsite (50%), quartz (35%), and kaolinite (15%). The impurities such as Fe<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, and alkali occur in very small percentages. Detailed polishedsection microprobe analysis was used to analyze the association between the mineral phases and also to describe the liberation characteristics of the ore. These studies were accomplished using an electron microprobe manufactured by Cameca (model SX-50) where X-ray scanning images were made in order to obtain the elemental distribution of the sectioned particles. Detailed microcompositional information was obtained from these scans. It was observed that quartz occurs mainly as relatively coarse grains (>300  $\mu$ ) and as macroscopic veins. Because kaolinite occurs only as fine grains disseminated in a gibbsitic matrix, there is essentially no association between kaolinite and quartz. Another interesting feature of the Rio Pomba bauxite is the fact that all minor impurities (mainly Fe<sub>2</sub>O<sub>2</sub> and TiO<sub>2</sub>) are only associated with the kaolinite.

The X-ray scanning micrographs were also used to determine the linear intercept distribution of the kaolinite grains in order to apply King's liberation model (King, 1979) which is based on linear intercept analysis as determined from polished sections. Experimental results have suggested that about 11% of the kaolinite is expected to be liberated at a particle size of 75  $\mu$ . Details of the liberation characteristics will be discussed in a separate paper.

#### Flotation Procedures

The main objective of the flotation studies Was to obtain a high-alumina content gibbsite concentrate to be used for the production of firstclass alumina refractories. In this regard, a high-purity gibbsite concentrate must be made by the necessary rejection of quartz and kaolinite. In view of the liberation characteristics described above and because there are no significant differences in physical properties (density, magnetic and electrostatic susceptibility, etc.) between the major mineral phases (gibbsite, kaolinite, and quartz), flotation was selected as the most suitable method for the recovery of a high-purity gibbsite concentrate. Preparation of the feed for flotation is discussed in subsequent sections.

The analysis of flotation products was accomplished by X-ray fluorescence (X-MET 820, Outokumpu Electronics). This instrument uses a radioisotope for its X-ray source, and the measurements are based on energy dispersive X-ray fluorescence. Using 21 standard samples of the Rio Pomba bauxite with known chemical analysis ( $Al_2O_3$  content varying between 5 and 97% on calcined basis), calibration curves were established in order to determine the chemical composition of unknown samples of flotation products. The analysis involves an error of about 5%.

#### Flotation of Gibbsite/Kaolinite from Quartz

Previous research (Magnesita S.A., 1986) has established the optimum conditions for the flotation of quartz from gibbsite for the Rio Pomba bauxite using a quaternary ammonium salt as collector. However, a considerable sensitivity to slimes was detected in this system. Therefore, a desliming stage prior to flotation became essential. The material was deslimed at 37  $\mu$  after being ground to minus 177  $\mu$ . A concentrate containing 94% Al<sub>2</sub>O<sub>3</sub> (calcined basis) was obtained with 47% mass recovery and 83% alumina recovery. However, considering that the minus 37- $\mu$  slimes represent a 33% weight loss of feed, the mass recovery and alumina recovery are actually 32% and 53% respectively.

In this regard, two samples of the Rio Pomba bauxite were prepared in order to establish the optimum conditions for the flotation of gibbsite using alkyl sulfates supplied by Henkel Corp. as collectors. The first sample was ground in a laboratory rod mill to minus 177 µ and deslimed by sieving at 37  $\mu$  (same size range used in the previous research). The second sample was ground to minus 75  $\mu$  and deslimed with a cyclosizer at 10  $\mu$ . The minus  $10-\mu$  slimes, in this case, represented 18.2% by weight of the feed material. Most of the flotation testing was done with the coarse sample. After grinding and desliming, all samples were analyzed by X-ray fluorescence as shown in Table 2. It can be observed that the slimes contain more alumina and less silica than the deslimed material. showing that selective grinding occurs due to the

Table 2.										
Alumina	and	Si	lica	Cont	ent	of	the	Río	Pomba	
Bauxite S	Sampl	es	Used	for	Flo	tat	ion	Expe	riment	s

	% Wt	% A1203	% Si0 <sub>2</sub>
Fine Sample			
-75 +10 μ	81.8	33.7	47.0
-10 µ slimes	18.2	55.6	22.1
	100.0	37.7	42.5
Coarse Sample			
-177 +37 μ	66.9	32.1	48.7
-37 µ slimes	33.1	48.9	30.1
	100.0	37.7	42.5

difference in grindability between gibbsite and quartz. Usually, for the Rio Pomba material and for bauxite ores in general, the slimes contain a higher percentage of  $Al_2O_3$ , as is evident from the data presented in Table 2.

Preliminary Experiments. The bench-scale flotation tests were typically performed on the deslimed samples in a 1-liter flotation cell (Denver Equipment Co.) at 1000 rpm and 10% solids. Hydrochloric acid and sodium hydroxide solutions were used to control pH. A rougher and scavenger flotation stage was done for each test with the addition of the same quantities of collector in each stage. Initially, sodium dodecyl sulfate (SDDS) was used as collector for gibbsite flotation tests on the fine sample. Five minutes of conditioning time was used for each reagent added in the flotation tests. Figure 1 shows the effect of pH on the alumina content and recovery in the rougher concentrate as well as the silica content and recovery in the scavenger tailings product. It can be noted that the best selectivity was achieved at pH 2 where a rougher alumina recovery higher than 80% and a scavenger tailings product containing more than 95% silica were obtained. The selectivity decreases significantly when the pH approaches the gibbsite PZC value of pH 9 and then increases again at higher pH values.

Effect of Sodium Dodecyl Sulfate Concentration and Particle Size. The effect of the SDDS concentration on the flotation of the deslimed fine and coarse samples at pH 2 is shown in Figures 2 and 3. It can be observed that the concentrate from the coarse feed  $(-177 + 37 \mu)$  is richer than the concentrate from the fine feed  $(-75 + 10 \mu)$ . In both cases, tailing products containing more than 95% silica were obtained, but with significantly better recoveries of silica in the tailings for the case of the coarse feed. These results show that particle size distribution has a major effect on



Effect of pH on the flotation of gibbsite/kaolinite from the Rio Pomba bauxite (-75 +10  $\mu$ ) using 200 g/t per stage of sodium dodecyl sulfate (SDDS) as collector.



Effect of sodium dodecyl sulfate (SDDS) addition on gibbsite/kaolinite recovery in the concentrate during rougher flotation of the Rio Pomba bauxite at pH 2.



Figure 3. Effect of sodium dodecyl sulfate (SDDS) addition on quartz rejection to the tailings during scavenger flotation of the Rio Pomba bauxite at pH 2.

the separation efficiency. The effect of temperature and sodium silicate concentration was also studied in this system, but no significant effect was detected in either case.

Effect of Hydrocarbon Chain Length. In order to study the effect of the hydrocarbon chain length on gibbsite flotation with alkyl sulfates, sodium decyl sulfate (SDES) and sodium octyl sulfate (SOCS) were also tested using the coarse feed (-177 +37  $\mu$ ), as shown in Figures 4 and 5. It can be noted that the longer the hydrocarbon chain length, the smaller the collector concentation required to achieve a given recovery. High-silica tailings were



Effect of alkyl sulfate addition on gibbsite/kaolinite recovery in the concentrate during rougher flotation of the Rio Pomba bauxite (-177 +37  $\mu$ ) at pH 2 for sodium dodecyl, decyl, and octyl sulfates as collectors.



Effect of alkyl sulfate addition on quartz rejection to the tailings during scavenger flotation of the Rio Pomba bauxite  $(-177 + 37 \mu)$  at pH 2 for sodium dodecyl, decyl, and octyl sulfates as collectors.

155 44 3

obtained with SDDS and SDES, but a tailings grade of 80%  $SiO_2$  could not be achieved in the case of SOCS. A concentrate containing 55.7%  $Al_2O_2$  was obtained with SDES, which, of the three collectors, provided the best separation efficiency.

Effect of Flotation pH. In order to optimize the conditions using SDES as collector, the pH effect was studied as presented in Figure 6, which shows the alumina content and recovery in the rougher concentrate as well as the silica content and re-

covery in the scavenger tailings as a function of pH. Similar to the SDDS case, a higher recovery as well as a higher alumina content in the concentrate was achieved with SDES at pH 2. The same decrease in recovery and grade near the PZC of gibbsite was detected, but there was not significant increase in selectivity at higher pH.

These results support previous indications (Yoon and Salman, 1976; Miller and Kellar, 1988; Kellar, Cross, and Miller, 1989) that alumina has a rather strong adsorption potential for alkyl sulfates under acidic conditions, conditions under which the alkyl sulfate collector appears to be stabilized at the alumina surface as a chemisorbed and surface-precipitated species. For example, the adsorption density of SDDS by alumina was determined as a function of pH by FTIR/IRS as shown in Table 3 (Kellar and Cross, 1989). It is evident that the SDDS adsorption density is relatively constant at pH 3 and pH 7 but increases dramatically at pH 2. The increase in the adsorption density is substantial, increasing by almost an order of magnitude. These results are indicative of multilayer adsorption and suggest a surface precipitation of



Figure 6.

Effect of pH on the flotation of gibbsite/kaolinite from the Rio Pomba bauxite (-177 + 37  $\mu)$  using 800 g/t per stage of sodium decyl sulfate (SDES) as collector.

Table 3. Adsorption Density of Sodium Dodecyl Sulfate at the Surface of Alumina after 20 Minutes Equilibration as Determined by In-Situ FTIR/IRS Measurements for Various pH Values and an Equilibrium SDDS Concentration of 1x10<sup>-3</sup> M

Hq	Adsorption Density (mol/cm <sup>2</sup> )
2	9.02x10 <sup>-10</sup>
3	$1.55 \times 10^{-10}$
7	$0.97 \times 10^{-10}$

SDDS has occurred at the alumina surface. This phenomenon apparently accounts for the strong flotation response of gibbsite during flotation of the Rio Pomba ore at pH 2.

Effect of Percent Solids. Since all flotation tests mentioned before were performed at 10% solids in the pulp, the effect of the pulp density was also studied as shown in Figure 7. It can be noted that there is a significant increase in alumina recovery when the pulp density is increased from 10% to 15%, and for densities higher than 15% a plateau is established. Also, for pulp densities higher than 15% solids, a tailings product containing more than 98% silica was obtained reaching 99.5% silica at 30% solids.

Cleaning the Rougher Concentrate. Figure 8 shows the cumulative grades and recoveries in the concentrate after cleaning the rougher concentrate. The alumina content in the concentrate increases significantly up to the first cleaner stage, after which the grade increases slowly and reaches a plateau. After five cleaner stages, a cleaner concentrate containing 58.7% Al<sub>2</sub>O<sub>3</sub> and 8.2% SiO<sub>2</sub> was obtained with 97.0% alumina recovery and 90.0% of the total silica removed. However, considering the desliming stage during the feed preparation, the alumina recovery is actually 55.3%.

In order to minimize the losses to slimes in the desliming stage, a third sample was prepared by grinding to minus  $177 \mu$  and desliming at 3  $\mu$ . In this case, the minus  $3-\mu$  slimes represented 10.6%weight loss of feed containing 56.5% Al<sub>2</sub>O<sub>2</sub> and 21.3\% SiO<sub>2</sub>. The flotation of the material -177 +3  $\mu$  containing 35.6 Al<sub>2</sub>O<sub>3</sub> and 44.9\% SiO<sub>2</sub> was performed at 20\% solids, pH 2, and using SDES as collector as presented in Figure 9, which shows the effect of the collector concentration on mass recovery, alumina grade, and alumina recovery in the





Effect of pulp density on the flotation of gibbsite/kaolinite from the Rio Pomba bauxite (-177 +37  $\mu$ ) at pH 2 using 800 g/t per stage of sodium decyl sulfate (SDES) as collector.



Figure 8.

Cumulative grades and recoveries for the flotation of gibbsite/kaolinite from the Rio Pomba bauxite (-177 +37  $\mu$ ) at 20% solids, pH 2, and using 800 g/t of sodium decyl sulfate (SDES) as collector for rougher and scavenger stages.



Figure 9. Effect of sodium decyl sulfate (SDES) addition on the gibbsite/kaolinite flotation from the Rio Pomba bauxite (-177 +3  $\mu$ ) after one scavenger, one rougher, and five cleaner stages at 20% solids and pH 2.

concentrate after one scavenger, one rougher, and five cleaner stages. At 200 g/t of SDES added to the rougher and scavenger stages, a concentrate containing 63.2% alumina (approximately 93.0% calcined basis) was obtained, representing 46.4% by weight of the feed material with an alumina recovery of 79.9%. Considering the desliming stage, the mass recovery and alumina recovery in the concentrate are 41.5% and 67.3% respectively. This concentrate was submitted for X-ray diffraction analysis, which revealed the presence of only gibbsite and kaolinite, indicating that practically all the quartz had been rejected. 150 1 x 31

89.214

Flotation of Kaolinite to Produce High-Purity Gibbsite 1.12

The differential flotation of kaolinite from gibbsite in the SDES alumina concentrate was studied in order to produce a high-purity gibbsite concentrate. The concentrate obtained with SDES at pH 2 (Figure 9, 200 g/t) containing only kaolinite and gibbsite was used as feed material after being filtered and dried. Only one flotation stage was performed in each test. Table 4 shows the composition of the concentrate  $(-177 + 3 \mu)$  used as feed material for the tests of differential flotation of kaolinite from gibbsite. A pulp density of 20% solids was used in all tests."

Firstly, the flotation of gibbsite using oleic acid as collector and depression of kaolinite with sodium hexametaphosphate at pH 9 was tested as suggested by Balashova and Kuznetov (1970). Different concentrations of both reagents mentioned above were tested, but no selectivity was achieved. It was observed that sodium hexametaphosphate depressed both gibbsite and kaolinite even at low concentrations.

Secondly, the cationic flotation of kaolinite using amine as collector was tested as suggested by Brehler et al. (1970) and Kellogg (1946). A quaternary ammonium salt (cetyltrimethylammonium chloride) supplied by Hoechst was used as collector at different pH values as shown in Figure 10. It can be observed that selectivity was achieved for pH values lower than 6. In this acidic region, the silica grade in the tailings is higher than 6.4% (the feed material contains 3.1% SiO<sub>2</sub>), and 76.9% of silica recovery in the tailings product was reached at pH 6 where a maximum alumina grade in the concentrate (64.95%) was obtained with an alumina recovery of 64.80%. For pH values between 6 and 8, some selectivity remains, but a decrease in grade and recovery takes place. For pH values higher than 8, the silica grade in tailings and consequently the alumina grade in the concentrate become approximately the feed grades, indicating the lack of any selectivity. These results support the results obtained by Brehler et al. (1970) where the flotation of kaolinite with amine hydrochloride as collector was observed only at pH values less than pH 8.

# Table 4.

Composition of the Alumina Concentrate Used for the Production of High-Purity Gibbsite

Chemical (	Composition	Mineralogical Composition			
% Al <sub>2</sub> 03	63.2	% Gibbsite	92		
\$ Sio	3.1	🖇 Kaolinite	7		
۶ Fe <sub>2</sub> 0 <sub>3</sub>	0.7	🖇 Impurities	1		
% Ti02	0.1				
🕻 Alkali	- 0.1				



Effect of pH on the single-stage rougher flotation of kaolinite from gibbsite in the alumina concentrate (-177 +3  $\mu$ ) prepared from the Rio Pomba bauxite using 200 g/t of quaternary ammonium salt 1 m as collector.

In order to optimize the quaternary ammonium chloride concentration for pH values lower than 6, some flotation tests were performed at pH values of 2, 4, and 6 as shown in Figure 11. It can be noted that the higher alumina grades in the concentrate were obtained at pH 6 where a value of 65.13% Al203 (approximately 98.8% Al202 calcined basis) was obtained at 400 g/t of quaternary ammonium chloride. However, the grade curves have a tendency to become horizontal for additions of the quaternary ammonium chloride collector higher than 300 g/t. This limiting behavior is due to the liberation characteristics of the feed material.



#### 14 Figure 11.

Effect of quaternary ammonium salt addition on the gibbsite recovery in the rougher concentrate (-177 2. +3  $\mu$ ) at pH values of 2, 4, and 6.

1957 1.37 05

In the acid pH range, the pH effect decreases with a decrease in the collector concentration and becomes insignificant at collector additions lower than 100 g/t. At this concentration (100 g/t), a concentrate containing approximately 64.7% Al<sub>2</sub>O<sub>2</sub> (97.4% Al<sub>2</sub>O<sub>2</sub> calcined basis) with an alumina recovery of about 90% was obtained. Taking into consideration the flotation of a gibbsite/kaolinite alumina concentrate from quartz at pH 2 using 200 g/t of SDES as collector as a first stage and the flotation of kaolinite from gibbsite at pH 6 using 100 g/t of quaternary ammonium salt as collector as a second stage, a final concentrate containing 64.71%  $Al_2O_2$  can be obtained with a final alumina recovery in the concentrate of 71.59% and a mass recovery of 40.62%. If the losses during the desliming stage are taken into consideration, the alumina recovery and mass recovery are 60.25% and 36.31% respectively. However, it is important to remember that alumina recovery does not mean gibbsite recovery for this system, since kaolinite also has aluminum and is being considered as a tailing material. Therefore, the gibbsite recovery is substantially higher than the alumina recovery, and it is estimated to be about 90% from the flotation feed. If the gibbsite losses in the 3-µ fraction (due to desliming) are taken into consideration, the overall gibbsite recovery is estimated to be about 78%.

## SUMMARY AND CONCLUSION

Based on the results from this research program, a process has evolved for the production of a high-purity gibbsite product from the Rio Pomba bauxite ore. The liberation characteristics of this ore, composed basically of gibbsite, kaolinite, and quartz, were studied through detailed polished-section microprobe analysis. It was observed that kaolinite occurs as fine grains disseminated into a gibbsitic matrix and quartz occurs mainly as coarse grains and macroscopic veins. King's liberation model (King, 1979) was applied to describe the kaolinite liberation characteristics, and it was estimated that about 11% of the kaolinite is expected to be liberated at a particle size of 75 µ.

Flotation was selected as the most suitable method for the recovery of a high-purity gibbsite concentrate from the Rio Pomba bauxite ore. Prior to flotation, the ore was ground to minus 177  $\mu$  and deslimed. The flotation strategy (Figure 12) is composed of two flotation steps where different flotation conditions were used. The first step involves the flotation of gibbsite/kaolinite from quartz using alkyl sulfates as collectors. One scavenger, one rougher, and five cleaner stages were used with addition of collector only in the rougher and scavenger stages. Optimum conditions were established with the addition of 200 g/t of sodium decyl sulfate (SDES) at pH 2 and 20% solids. For these conditions, a gibbsite/kaolinite concentrate, the alumina concentrate, was obtained containing 63.2% Al<sub>2</sub>O<sub>3</sub> (93.0% calcined basis) with an alumina recovery of 79.9%.

25 0 4 M 24

The second flotation step involves the flotation of kaolinite from gibbsite in the alumina concentrate using an amine as collector. Only one flotation stage was performed in this case. Optimum conditions were established with the addition of 100 g/t of cetyltrimethylammonium chloride at pH 6 and 20% solids. For these conditions, a final gibbsite concentrate was obtained containing 64.7% Al<sub>2</sub>O<sub>3</sub> (97.4% calcined basis) with a final alumina recovery and mass recovery of 71.6% and 40.6% respectively. The final gibbsite recovery is estimated to be about 90% from the flotation feed and 78% if the gibbsite losses in the minus 3  $\mu$  fraction (due to desliming) are taken into consideration.

Table 5 shows a comparison between the chemical compositions of the concentrate obtained by the flotation process described in this paper and some raw materials used to manufacture high-alumina refractories. It can be noted that the flotation concentrate has an alumina content comparable to fused aluminas and significantly higher than the Guyanian bauxite. Actually, in view of the application of these materials in the refractory industry, the flotation concentrate has a chemical





Flotation strategy for the production of a highpurity gibbsite concentrate from the Rio Pomba bauxite ore

# Table 5. Chemical Composition (Calcined Basis) of the High-Purity Gibbsite Concentrate and Some Raw Materials Used to Manufacture High-Alumina Refractories

		High-Purity Gibbsite Concentrate	Guyanian Bauxite	E Brown Fused <u>Alumina</u>	White Fused Alumina	
6	A1203	97.4	88.0	96.1	99.3	
6	Si02	1.9	6.5	1.2	0.1	
6	Fe203	0.3	1.7	0.4	0.1	
6	Ti02	0.1	3.5	2.0	0.0	
6	Alkali	0.1	0.1	0.1	0.3	

84

composition better than the brown fused alumina, especially with regard to the TiO<sub>2</sub> grade, since iron and titanium oxides and alkali are highly harmful to refractory properties. Importantly, it should be noted that the production cost of the flotation concentrate is considerably lower than the production cost for fused alumina, which provides an excellent opportunity for the application of this high-purity gibbsite concentrate in the refractory industry.

#### ACKNOWLEDGEMENTS

The authors gratefully acknowledge the financial support provided by Magnesita S.A. for this research project, graduate studies, and technical services. Support from DOE Grant No. DE-FE-02-84ER13181 has also contributed to the success of this work.

The authors extend their thanks to Amilcar G. Morato for analytical and technical assistance and to Jon J. Kellar and William M. Cross for helpful discussion.

#### REFERENCES

- Andreev, P. I., Anischenko, N. M., and Kirichenko, T. F., 1972, "Mechanism of the Interaction of Cationic Reagents during the Flotation of Chamosite-Gibbsite Bauxites," <u>Tsvetnye Metally</u>, Vol. 15, pp. 12-16.
- Andreev, P. I., et al., 1973a, "Beneficiation of Chamosite-Gibbsite Bauxites in the Belenikhinsk Deposit of the Kursk Magnetic Anomaly," <u>Obo-</u> gashch. Polez. Iskop., Vol. 12, pp. 16-19.
- Andreev, P. I., Anishchenko, N. M., and Mishakenkova, N. P., 1973b, "Mechanism of the Anionic Flotation of Chamosite and Gibbsite," <u>Tsvetnye</u> Metally, Vol. 16, pp. 16-20.
- Andreev, P. I., Anishchenko, N. M., and Mishakenkova, N. P., 1975, "Mechanism of the Action of Amines during the Flotation of Bauxite Ore Minerals," Tsvetnye Metally, Vol. 18, pp. 13-17.
- Balashova, G. G., and Kuznetsov, V. P., 1970, "Flotation Concentration of Kaolinite-Hydrargillite Bauxites," Tsvetnye Metally, Vol. 11, pp. 84-85.
- Black, C. A., and Low, P. F., 1950, "Reactions of Phosphate with Kaolinite," <u>Soil Science</u>, Vol. 70, pp. 273-289.
- Brehler, B., Clement, M., and Trondle, H. M., 1970, "Flotation of Kaolinite and Feldspar with Amines as Collectors," <u>Interceram</u>, Vol. 19, pp. 185-191.
- Chandar, P., Somasundaran, P., and Turro, N. J., 1987, "Fluorescence Probe Studies on the Structure of the Adsorbed Layer of Dodecyl Sulfate at the Alumina-Water Interface," <u>Journal of Colloid</u> and Interface Science, 117, 1, pp. 31-46.
- Cheng, T. W., and Wang, C. T., 1982, "The Beneficiation of Gibbsite from the Tatun Volcanic Area," Kuang Yeh, Vol. 26, pp. 121-132.

- Fuerstenau, D. W., and Yamada, B. J., 1962, "Neutral Molecules in Flotation Collection," <u>AIME</u> <u>Trans.</u>, Vol. 223, pp. 50-52.
- Fuerstenau, M. C. (editor), 1976, <u>Flotation --</u> <u>A. M. Gaudin Memorial Volume</u>, <u>SME</u> Publication, <u>New York, NY.</u>
- Fuerstenau, M. C., Miller, J. D., and Kuhn, M. C., 1985, <u>Chemistry of Flotation</u>, SME Publication, New York, NY.
- Gaudin, A. M., 1957, <u>Flotation</u>, McGraw-Hill Book Company, Inc., New York, NY.
- Hinds, S. A., Husain, K., and Liu, N., 1985, "Beneficiation of Bauxite Tailings," <u>Light Met.</u>, Vol. 54, pp. 17-30.
- Huang, B. H., and Ray, D. T., 1980, "Study on the Beneficiation of Bauxite on the Tatun Volcanic Area, Taiwan," <u>Kuang</u> Yeh, Vol. 18, pp. 69-81.
- Kellar, J. J., and Cross, W. M., 1989, Private communication, University of Utah.
- Kellar, J. J., Cross, W. M., and Miller, J. D., 1989, "Adsorption Density Calculations from In-Situ FTIR/IRS Data at Dilute Surfactant Concentrations," to be published November/December 1989, Applied Spectroscopy, 43, 8.
- Kellogg, H. H., 1946, "Flotation of Kaolinite for Removal of Quartz," <u>AIME Trans.</u>, Vol. 169, pp. 548-555.
- King, R. P., 1979, "A Model for the Quantitative Estimation of Mineral Liberation by Grinding," <u>International Journal of Mineral Processing</u>, Vol. 6, pp. 207-220.
- Leja, J., 1982, <u>Surface Chemistry of Froth Flota-</u> tion, Plenum Press, New York, NY.
- Magnesita S.A., 1986, "Estudo de Beneficiamento do Bauxito Rio Pomba -- MG," Internal Report, REL-CPqD-86-0163.
- Miller, J. D., and Kellar, J. J., 1988, "Quantitative In-Situ Analysis of Collector Adsorption Reactions by Internal Reflection FTIR Spectroscopy," <u>Challenges in Mineral Processing</u>, Sastry, K. V., ed., SME-AIME, Littleton, Colorado, pp. 109-129.
- Modi, H. J., and Fuerstenau, D. W., 1960, "Flotation of Corundum -- An Electrochemical Interpretation," AIME Trans., Vol. 217, pp. 381-387.
- Somasundaran, et al., 1989, "Excited State Resonance Raman Spectroscopy as a Probe of Alumina-Sodium Dodecyl Sulfate Hemimicelles," <u>Langmuir</u>, 5, 1, pp. 215-218.
- Yoon, R. H., and Salman, T., 1976, "Chemisorption of Sodium Lauryl Sulfate on Y-Al<sub>2</sub>O<sub>3</sub>," <u>Colloid</u> <u>and Interface Science</u>, vol. 3, Kerker, M., ed., Academic Press, New York, pp. 233-246.