

SOME ASPECTS OF THE MINERALOGY OF THE KIBI BAUXITE

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ABSTRACT

The mineralogy of the Kibi bauxite was investigated using XRD, IR and Thermoanalytical methods. Five pit samples were used in the study. Results indicated that the gibbsite, boehmite and goethite minerals are present in a well crystallised state as coarse dispersions. The aluminium substitution in the goethite lattice was high, varying between 20-29 mol per cent.

It can therefore be predicted that aluminium recovery at low temperature digestion conditions will be low and the aluminium content of the goethite will remain undigested under such conditions.

Keywords: mineralogy, crystallinity, particle size, aluminium substitution, goethite lattice, digestibility

INTRODUCTION

A knowledge of the mineralogy of an ore body is essential to the prediction of the behaviour of the mineral components and is therefore a tool in the choice of process technology. Answers could also be given to some problems that may arise during the treatment of the ore.

Apart from the different phases present in ore, data on the crystallinity, grain size, micromorphology, and the specific surface, among others, are also important.

The X-ray diffractometry (XRD), Infrared (IR) spectroscopy and thermal methods are extensively used for the phase analysis of bauxites. Scanning electron microscopy has been helpful in the study of the micromorphology and grain size.

For heterogeneous solid-liquid reactions, such as the digestion of bauxite in caustic soda, the rate of reaction is affected by both crystallinity and particle size of the mineral, among others. Less crystalline, amorphous minerals are more easily digested than the crystalline ones. As the particle

size of the mineral decreases the reaction rate increases and hence an increase in the digestibility of the mineral.

In the Bayer process technology, aluminium incorporated in the goethite lattice is not leachable under normal conditions of digestion, and therefore constitute digestion losses. The aluminium ions can only be freed when the goethite lattice disintegrates under the action of certain digestion additives.

In the present study, as part of a comprehensive investigation of the Kibi bauxite, emphasis is laid on the crystallinity, particle size of the different minerals, and the degree of aluminium substitution in the goethite lattice, as properties that could affect the digestibility of these minerals.

ANALYTICAL METHODS

X-ray Diffractometry

In addition to the usual qualitative and quantitative determination by X-ray diffractometry, deductions on the crystallinity and grain size of gibbsite, boehmite, diaspor, kaolinite, hematite and anatase could also be made from the quality of the characteristic peaks of these minerals [1,2]. The half-width ($\Delta\theta$) values and the ratio of the half-width to height of the peaks are used. The lower the ratio the higher the degree of crystallinity.

A relationship has been established between the shift in the goethite reflection and the extent of aluminium substitution in the goethite lattice [3,4]. The greater the shift the more the aluminium substitution.

Infrared Spectroscopy

With IR spectroscopy the position and quality of the absorption bands allow deductions to be made on the phases present and the degree of crystallinity of the minerals [1,4]. The ratio of the extinctions of the O-H stretching bands of gibbsite at 3460 and 3530 cm^{-1} have been proven to give information on the degree of crystallinity and therefore the digestibility of the gibbsite [1,5]. Similarly, the wave number of the Al-O-H bending frequency or deformation band between 1075 and 1090 cm^{-1} related to the strength of the hydrogen bond between the OH groups and therefore to the crystallinity of boehmite mineral [6].

However, the position of the bending frequency does not give information on the age, microcrystallinity and hardness of the crystals, nor does it indicate if there had been recrystallization into larger and harder crystals. Information on this can

be inferred from the quality of the absorption bands.

The shift in the goethite Fe-O-H deformation band from 900cm⁻¹ towards 100cm⁻¹ means an increasing degree of aluminium substitution in the goethite lattice [1].

Thermoanalytical Methods

Thermoanalytical methods such as Differential Thermogravimetry (DTG) and Differential thermoanalysis (DTA) could be used in the determination of gibbsite, boehmite, diaspore kaolinite and goethite. The overlap of the endothermic peaks of gibbsite and goethite at 290-350°C, and boehmite and diaspore at 350-560°C can be resolved by selective dissolution [4,8].

In general the peaks of the coarser, crystalline minerals shift to higher temperature on the DTG and DTA.

The peak temperature (290-390°C) of the dehydration of goethite to hematite depends on the degree of crystallinity and grain size of the goethite. Aluminium substitution in the goethite lattice also causes a shift to a higher temperature.

XRD, IR, DTA, and DTG all have their own advantages, but it is the combined application of these together with other methods such as electron microscopy, specific surface area, that gives a better insight into the overall properties of the minerals.

EXPERIMENTAL

Five different pit samples were ground in laboratory ball mill to particle sizes less than 320µm. They were then prepared for XRD, IR and thermal analyses.

The X-ray diffractograms were taken using Muller Mikro III X-ray generator and Philips goniometer with CuKα radiation and discrimination.

The derivatograms were taken with MOM Q-1500D type Derivatograph using 1000mg sample at a sensitivity of 200mg.

For the IR studies 1.5mg sample was measured in 800mg KBr, pressed into a disc and the spectra recorded on Zeiss UR-10 type spectrophotometer in the range of 400-3800cm⁻¹. Selective dissolution of sample for IR studies was also carried out with 8 weight per cent HCl solution in water bath at 80°C for 8 hours.

RESULTS AND DISCUSSION

The phase composition of the samples, determined by the combined use of data from XRD, thermal and chemical analysis, is shown in Table 1.

XRD Studies

The X-ray diffractogram of one of the samples (Sample No.1) is shown in figure 1. The X-ray diffractograms indicate the presence of gibbsite, boehmite, hematite, kaolinite, anatase, rutile and quartz in the sample. With the exception of sample No.3, which was rather clayey, boehmite, goethite and hematite peaks of the other samples were sharp. Whereas a highly crystalline mineral would have sharp and narrow peaks on a diffractogram, the less crystalline ones show broad peaks. The ratio of half-width (HW) to peak height (PH) decreases with increasing crystallinity. For the determination of the crystallinity of the bauxite minerals, the characteristic gibbsite, boehmite, hematite, goethite and kaolinite peaks at the lattice constant (d) values of 4.36, 6.11, 2.51, 4.14 and 7.13 respec-

Table 1: The phase compositions of the bauxite sample

	Sample No.1, %	Sample No.2, %	Sample No.3, %	Sample No.4, %	Sample No.5, %
Al ₂ O ₃ in gibbsite	31.9	17.5	40.0	36.1	42.0
boehmite	8.4	0.6	9.8	4.8	12.5
diaspore	0.3	-	0.5	0.2	0.2
kaolinite	2.8	13.3	1.4	3.4	0.9
halloysite	-	1.1	-	-	-
goethite	2.0	2.3	1.8	1.5	0.8
hematite	0.2	-	0.2	0.2	0.1
Fe ₂ O ₃ in kaolinite	3.4	15.6	1.8	4.0	1.0
halloysite	-	1.3	-	-	-
quartz	1.1	12.1	-	-	-
TiO ₂ in anatase	2.6	2.6	3.2	2.4	2.4
rutile	1.1	-	0.8	0.4	1.7

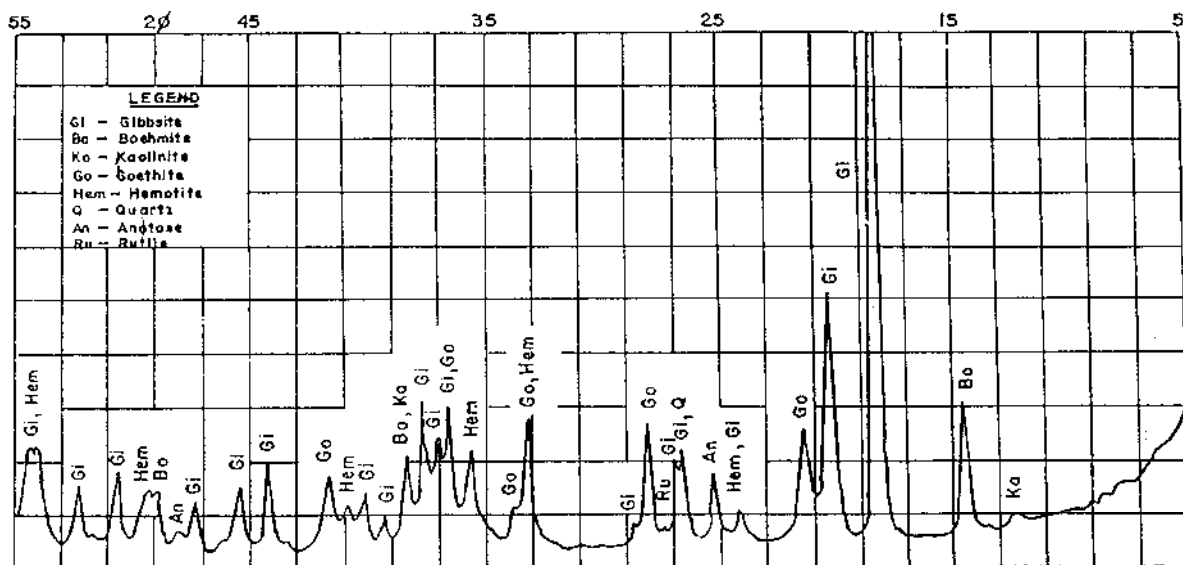


Figure 1: The X-ray diffractogram of sample No.1

Table 2: The half-width values (HWV) and the ratio of half-width to peak height (HW/PH) of the characteristic peaks on the X-ray diffractograms.

Sample No	Gibbsite peak at $d = 4.36$ HWV, HW/PH mm	Boehmite peak at $d = 6.11$ HWV, HW/PH mm	Goethite peak at $d = 4.14$ HWV, HW/PH mm	Hematite peak at $d = 2.51$ HWV, HW/PH mm
1	4.0 0.034	2.5 0.029	5.5 0.103	4.0 0.077
2	6.0 0.069	ND* ND*	10.5 0.160	ND* ND*
3	4.0 0.017	3.0 0.037	4.5 0.058	4.5 0.164
4	4.0 0.045	3.0 0.095	4.5 0.060	3.5 0.079
5	4.0 0.015	3.0 0.023	4.5 0.086	4.0 0.154

*ND - not determinable

Table 3: The lattice constants (d) of the goethite in the samples

SAMPLE NO.1	SAMPLE NO.2	SAMPLE NO.3	SAMPLE NO.4	SAMPLE NO.5
4.139	4.145	4.149	4.158	4.144

tively were used. The half-width values and the ratio of half-width to peak height are given in Table 2. The lattice constant (d) values of goethite peaks around 4.14 are given in Table 3.

Results in Table 2 show that with the exception of clayey sample No.2 the value of the ratio of half-width to peak height are generally low. This indicates a high degree of crystallinity of the gibbsite, boehmite and goethite minerals.

The d values (Table 3) show a substantial shift from the value of 4.20 (when there is no aluminium

in the goethite lattice). The values of 4.139, 4.145, 4.149, 4.158 and 4.14 correspond to an aluminium substitution of 28.5, 25.8, 24.0, 19.5 and 26.0 mol per cent respectively.

IR Spectrophotometric Studies

The characteristic wave numbers for the gibbsite and boehmite absorption bands are given in Table 4. The IR spectrogram for sample No.1 is shown in figure 2. The specific extinction (ϵ) values, calculated per gram sample, for gibbsite (at 3460 and

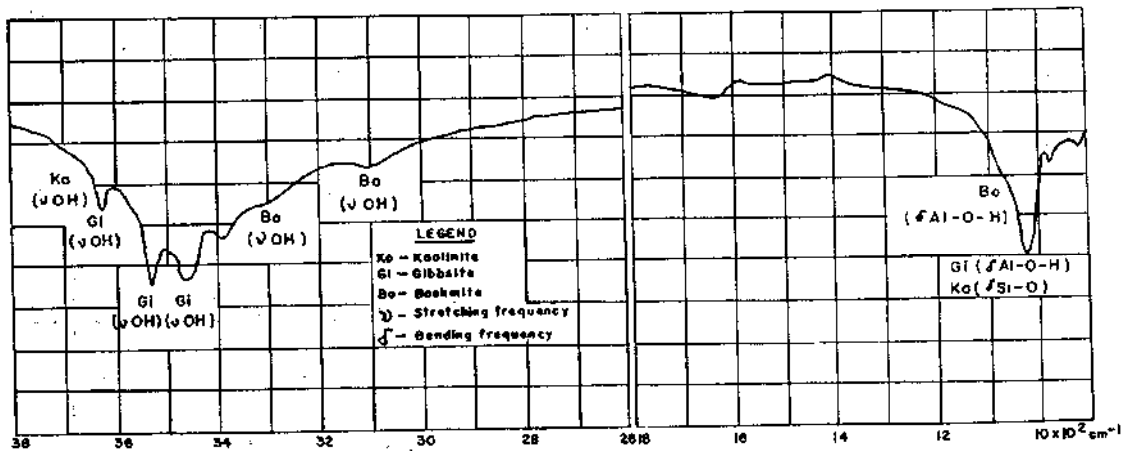


Figure 2: The IR Spectrogram of sample No.1

Table 4: The position of the characteristic absorption bands on IR spectra

Sample No.	Characteristic gibbsite bands, cm^{-1}	Characteristic boehmite bands, cm^{-1}
1	3630; 3532; 3462	3290; 3100; 1075
2	3630; 3530; 3455	ND* ND* ND*
3	3628; 3532; 3462	3300; 3100; 1075
4	3628; 3530; 3465	3300; 3105; ND*
5	3628; 3530; 3465	3300; 3100; 1072

* ND - not determinable

Table 5: The specific extinctions (ϵ) and the ratio of the extinctions (R) of the gibbsite bands at 3460 and 33530 cm^{-1} .

Sample No.	ϵ of boehmite bands at		ϵ of boehmite bands at		R
	3460 cm^{-1}	3530 cm^{-1}	3100 cm^{-1}	3300 cm^{-1}	
1	88	123	16	19	0.72
2	51	57	ND	ND	0.89
3	194	189	15	17	0.98
4	135	200	8	13	0.68
5	219	200	19	29	1.09

R-ratio of the extinctions at 3460 and 3530 cm^{-1} for gibbsite

Table 6: The characteristic gibbsite, boehmite and goethite peaks on the DTG, DTA

Sample No.	Gibbsite peak on DTG, $^{\circ}\text{C}$		Boehmite peak on DTA, $^{\circ}\text{C}$		Goethite peak on DTG, $^{\circ}\text{C}$
	DTG, $^{\circ}\text{C}$	DTG, $^{\circ}\text{C}$	DTA, $^{\circ}\text{C}$	DTA, $^{\circ}\text{C}$	
1	320	330	540	550	390
2	295	300	545*	560*	330
3	320	330	530	540	390
4	320	330	530	548	390
5	320	330	530	540	390

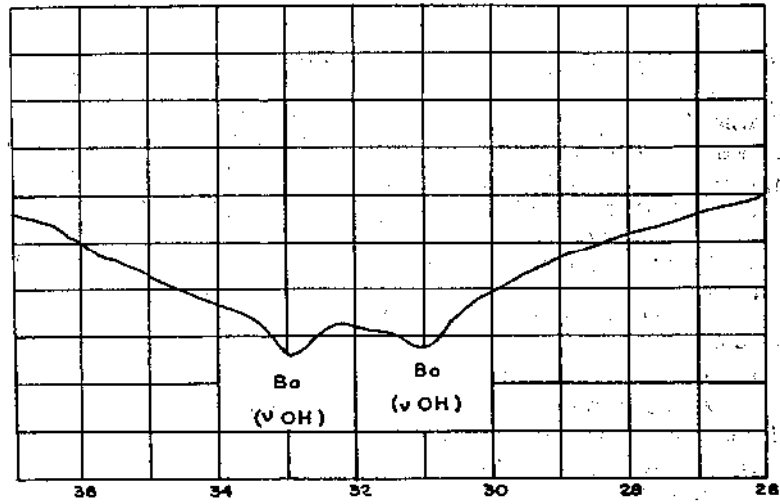


Figure 3: The IR Spectrogram of sample No.1, between 2600 and 3800cm⁻¹, after selective dissolution

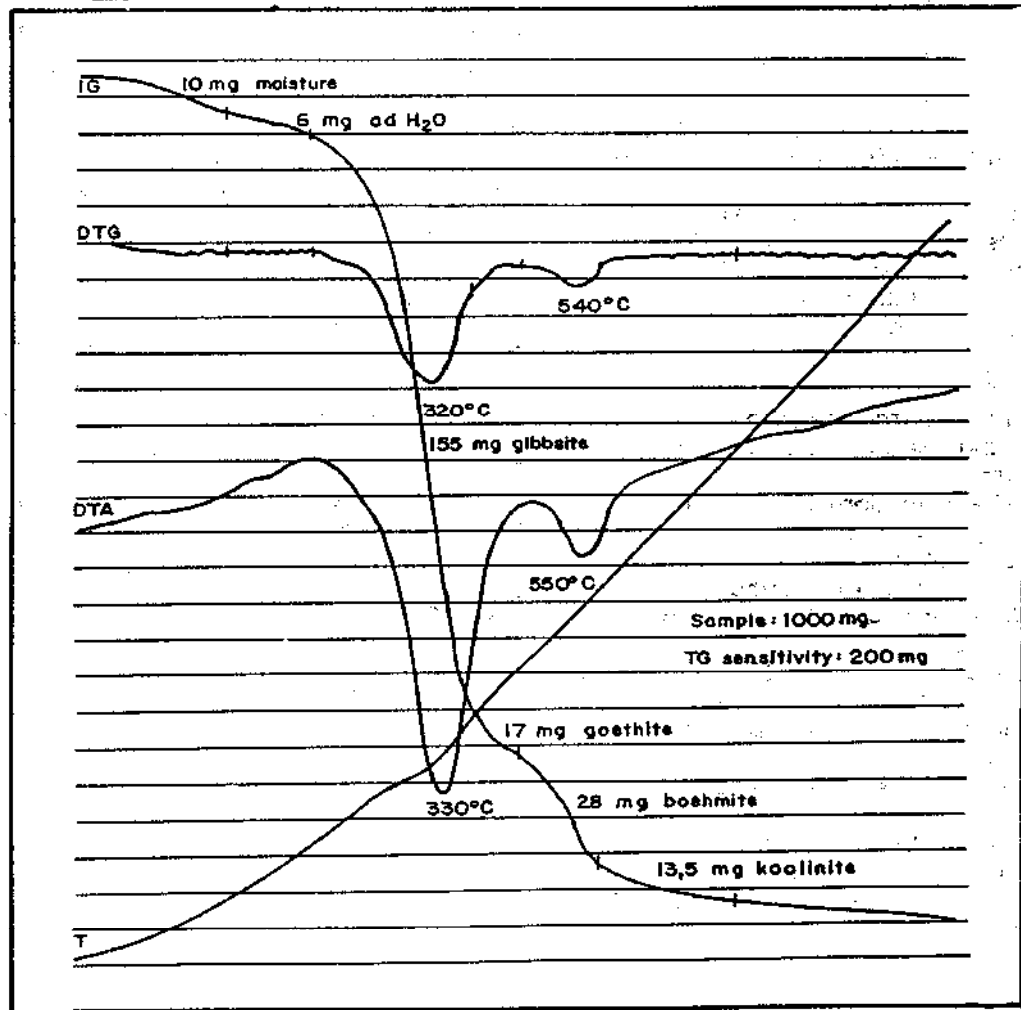


Figure 4: The Derivatogram of sample No.1

3530cm⁻¹) and boehmite (at 3100 and 3300cm⁻¹), together with the ratio of the extinction value at 3460 and 3530cm⁻¹ are given in Table 5.

The specific extinction values are very low, indicating a highly crystalline gibbsite and boehmite. The low value of the ratio of the extinction at 3460 and 3530cm⁻¹ for gibbsite confirms this fact.

The wave number of the Al-O-H bending frequency of the boehmite of all samples is between 1072 and 1075cm⁻¹. This means that the hydrogen bonds between the OH groups in the crystal lattice are not strong. However, the boehmite absorption bands at 3100 and 3290cm⁻¹ are very broad and flat.

Even after longer times of grinding in microvibratory ball mill, the shape hardly changed, showing that the boehmite crystals are coarse and hard. This property of the boehmite was further confirmed after selective dissolution with dilute acid. The crystals remained undissolved shown by the broad absorption bands in Figure 3.

Thermoanalytical (DTG DTA) Investigations

The derivatogram of sample No.1 is presented in Fig.4, and the positions of the gibbsite, boehmite and goethite bands on the DTG, DTA, are given in Table 6.

The endothermic bands of gibbsite and boehmite show a shift towards higher temperatures. This confirms the observations made with the XRD and IR, thus the degree of crystallinity of the minerals is high and they are present in coarse dispersions.

CONCLUSIONS

XRD, IR and the Thermoanalytical methods were used to study the mineralogy of five bauxite samples from the Kibi deposits.

The results of the investigations showed that the aluminium minerals (gibbsite and boehmite) and goethite are highly crystalline and exist in the ore as coarse dispersions. The aluminium substitution in the goethite lattice (alumogoethite) is high, as much as 20-29 mo per cent.

As a consequence of the high crystallinity of the minerals and their presence as coarse dispersions the aluminium recovery can be expected to be low at low temperature digestion conditions. The aluminium content of goethite will also remain undigested.

However, because of the coarse dispersion of the minerals, especially the undigested iron minerals, no difficulties will arise in the separation of red mud from digested liquor.

ACKNOWLEDGEMENTS

The author wishes to thank Dr. Jonas Klara of the University of Veszprem for her interest and guidance in starting this work. He is also grateful to Dr. Szucs Ferenc, Dr. Solymar Karoly and the team at the Alumina Research Laboratory of ALUTER-FKI, Hungary, for the use of some facilities at their laboratory. Finally thanks are due to Dr.K.E.N. Tsidzi.

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