Study of the transformation of boehmite into alumina by Rietveld method

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INTRODUCTION

The transformation of boehmite into alumina involve a complex sequence of transitional phases of aluminas:
Boehmite/amorphous Al_2O_3 → γ-Al_2O_3 → δ-Al_2O_3 → θ-Al_2O_3 → α-Al_2O_3.

This transition depends strongly on the chemical routes of synthesis, atmospheric conditions, degree crystallinity, heating rate, impurities, moisture, alkalinity, thermal history of the material, etc.

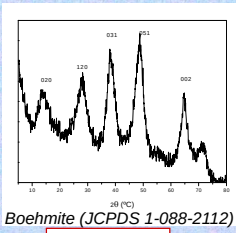
The aim of this paper is to study the conversion of boehmite into alumina, by means of the application of Rietveld refinement method to the X-ray diffraction patterns of samples obtained by thermal treatment of boehmite. Boehmite was synthesized by a hydrothermal method from an aluminium industry waste.

EXPERIMENTAL

Raw material: Aluminium industry waste
↓ Hydrothermal treatment
Precursor: Boehmite
↓ Thermal treatment
Furnace (Air)
T = 1300, 1400, 1500ºC; V_h =20ºC/min; t = 2, 7, 12h
↓
Alumina
↓
TG/DTA | TEM | XRD (Rietveld refinement method)

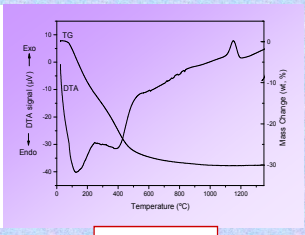
CARACTERIZATION

Precursor: Boehmite



Boehmite (JCPDS 1-088-2112)

XRD



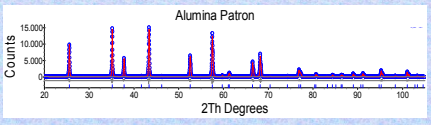
TG/DTA

Calcined precursor: Alumina

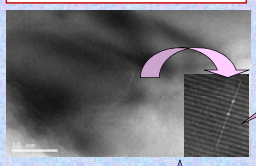
X-ray diffraction

Identification: **SIEMENS D 5000 diffractometer equipped with X-ray Cu tube and a diffracted beam monochromator. A current of 30 mA and a voltage of 40 KV were employed as tube setting.**

Quantification: **Rietveld method (TOPAS 4.0 Bruker AXS) As the diffraction patterns were obtained on a diffractometer working with parallel beam is necessary to consider the peculiarities introduced by this configuration when using the method. Therefore, we performed an empirical parameterization of the functions that establish the contribution of the computer. By analyzing the shape of the diffraction peaks of the standard sample of corundum, measured under the same conditions.**



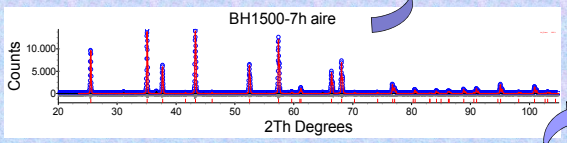
Crystallographic planes



The average interplanar spacing is 0.359 nm could correspond to the plane of indices 012 hkl of the corundum

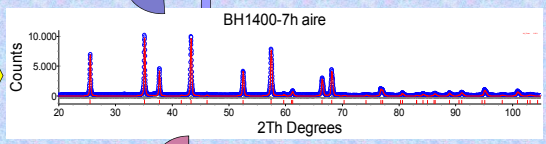
a = 0.4766 nm
c = 1.3009 nm

α-Al_2O_3	79.2%
Amorphous	19.80%

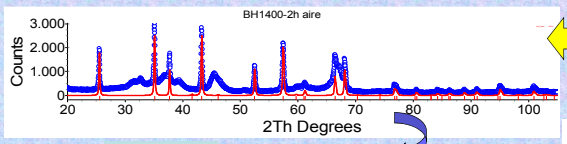


α-Al_2O_3	81.50%
Θ-Al_2O_3	0.7%
δ-Al_2O_3	1.7%
Amorphous	16.0%

a = 0.4766 nm
c = 1.3009 nm
a = 1.1790 nm
b = 0.2907 nm
c = 0.5618 nm
β = 103.78º
a = 0.5651 nm
c = 2.4067 nm



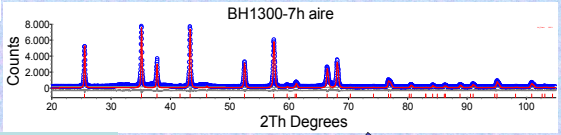
XRD (Rietveld refinement method)



a = 0.4765 nm
c = 1.3006 nm
a = 0.4624 nm
c = 1.3006 nm

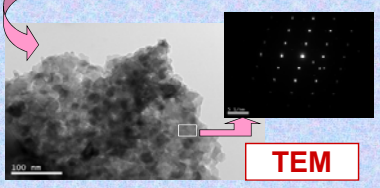
α-Al_2O_3	21.2%
γ-Al_2O_3	36.8%
θ-Al_2O_3	9.2%
δ-Al_2O_3	12.0%
Amorphous	20.8%

a = 1.1838 nm
b = 0.2890 nm
c = 0.5608 nm
β = 103.57º
a = 0.5567 nm
c = 2.4948 nm



a = 0.4766 nm
c = 1.3008 nm
a = 0.5642 nm
c = 2.3460 nm
a = 1.1790 nm
b = 0.2907 nm
c = 0.5618 nm
β = 103.78º
a = 0.5607 nm
c = 2.4911 nm

α-Al_2O_3	63.3%
γ-Al_2O_3	12.5%
θ-Al_2O_3	3.5%
δ-Al_2O_3	19.80%
Amorphous	18.0%



TEM

CONCLUSION

- **Rietveld method allowed to determining the lattice parameters and the mass fraction of the alumina phases.**
- **The temperature favours the transformation of metastable aluminas into corundum.The increasing of the corundum percentage comes from the changes in the transition aluminas, because the percentage of amorphous phase does not change during calcination (~20%).**
- **At 1500ºC and 7h, corundum was identified as the only crystalline phase with 19.8% of amorphous phase.**

REFERENCE

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