



	Experiment title: Speciation of rare earth elements in bauxite and bauxite residues using HERFD-XANES	Experiment number: 16-01-804
Beamline: ID16	Date of experiment: from: 07/07/2021 to: 15/07/2021	Date of report: 16/07/2021
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Report:

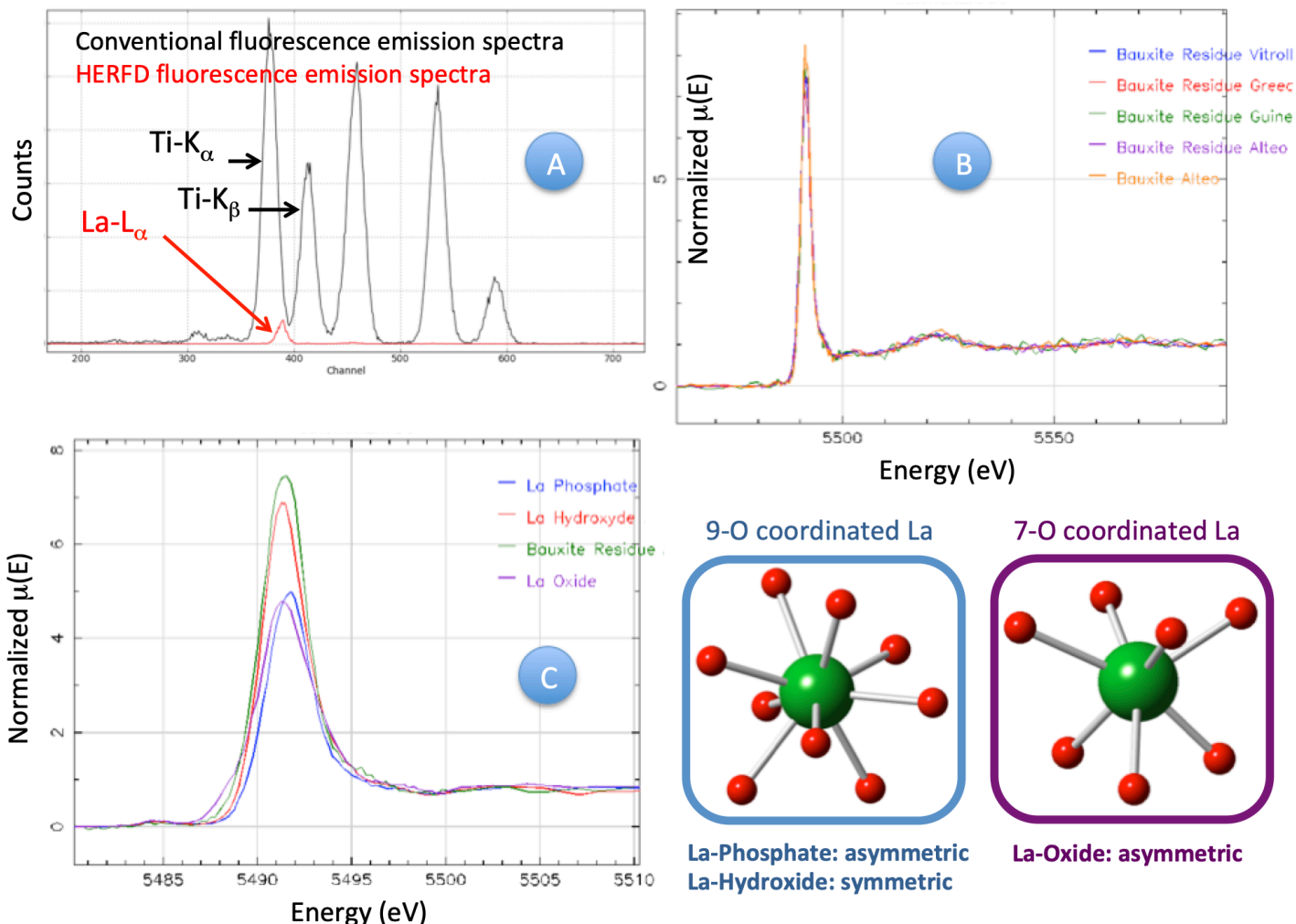
This experiment request is part of a larger project including two PhD projects that aims at developing sustainable processes for the recovery of iron and critical metals from bauxite residue. The project is built on the hypothesis that a good knowledge of waste composition and REE speciation will guide the extraction protocol and help minimizing the environmental impact of the extraction by reaching a better extraction efficiency and selectivity. Therefore, our project addresses the following question: What is the chemical speciation of REEs in bauxite residue and associated bauxite ore? In particular, this project aims at answering the following questions: (1) What is the bulk speciation of REEs in bauxite residues? (2) Is the speciation of REEs affected by the origin of the bauxite (lateritic vs karstic)? (3) Is the Bayer process, used to extract alumina modify the speciation of REEs and if yes, to what extent?

To answer these questions, measurements of 4 REEs on different bauxite residues and bauxite samples were initially planned: First, Lanthanum (La), as a Light REEs (LREE) proxy, Neodymium (Nd) speciation because of its importance in many key technologies, Gadolinium (Gd) given its position in the lanthanide series at the frontier between LREEs and Heavy REEs (HREEs) and finally, Ytterbium (Yb) as a HREE proxy.

Main Results:

- XAS conducted at the La-edge benefits from HERFD-XANES compared to traditional XANES as initially anticipated given the high concentration of Ti in the bauxite residue samples (Figure, A).
- Bauxite residue from different origins (Guinea, Greece and France) exhibit similar Lanthanum speciation despite different type of bauxite ore (lateritic (Greece, Vitrolles) or karstic(Guinea, Alteo)) (Figure, B).

- Lanthanum speciation in bauxite is similar than in bauxite residue indicating no or insignificant change of La-speciation during the Bayer process used to extract aluminium from bauxite (Figure, B).
- For model compounds, most differences were observed in the absorption edge and seems to be related to Lanthanum site symmetry and O coordination number based on preliminary calculations performed using FDRNES (Figure, C)
- If the speciation determination will be a challenge to address given the high similarities between the model compounds, a first rapid analysis seems to exclude the presence of significant amount of La-phosphates that is suspected for HREEs.



Issues encountered:

Several technical problems were encountered including an issue with the piezo of the monochromator and a leak of He from the spectrometer box that have caused a lost of 7 shifts (as estimated by the beamline staff). As a consequence, only the Lanthanum edge has been fully characterized for our samples and few spectra only were recorded at the Neodymium edge. The initially planned experiments at the Gd and Yb edges were not performed.

Perspectives

Linear Combination Fitting will be performed and additional XANES calculations will be done to investigate the effect of coordination number and symmetry on the shape of the white line. In addition, a proposal will be submitted for next call to finish the experiment. In particular, we would like to assess whether the proportion of phosphate-REEs increase as the atomic number increases from La to Yb.