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|                          | <b>Experiment title:</b><br>insights into Yttrium speciation in bauxite residue before and after unconventional leaching experiments | <b>Experiment number:</b><br>EV-471  |
| <b>Beamline:</b><br>BM30 | <b>Date of experiment:</b><br>from: 15/06/2022 to: 20/06/2022  | <b>Date of report:</b><br>16/07/2021 |
| <b>Shifts: 15</b>        | <b>Local contact(s):</b> Isabelle KIEFFER  | <i>Received at ESRF:</i>             |

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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 in the context of circular economy. 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 questions: (1) What is the bulk speciation of Y in bauxite and bauxite residue ? (2) Is the speciation of Y in bauxite residue affected by the origin of the bauxite (lateritic vs karstic)? (3) What is the speciation of Y leftover in the residue after leaching using either aqueous solution of organic acids or deep eutectic solvents as leaching media?

**Main Results:**

To answer question 1 and 2, measurements of Y speciation on a set of various bauxite residues and bauxite samples were measured from different origin (karstic (France, Greece) and lateritic (France, Guinea) and age (fresh and stockpiled or stored in laguna for up to hundred years).

- Bauxite residue from different origins (karstic vs lateritic) exhibit different Yttrium speciation (Figure 1). The other variables (storing conditions and age) does not seem to affect Y speciation
- From linear combination fitting results, Y speciation can be described as mostly Yttrium Phosphate in lateritic samples (Figure 2). Regarding karstic samples, Y speciation is different but not yet identified. Best fit were obtained using an Y-doped apatite and Y sorbed on hematite. The latter is consistent with the presence of iron oxy-hydroxyde in the bauxite residue as observed by XRD.

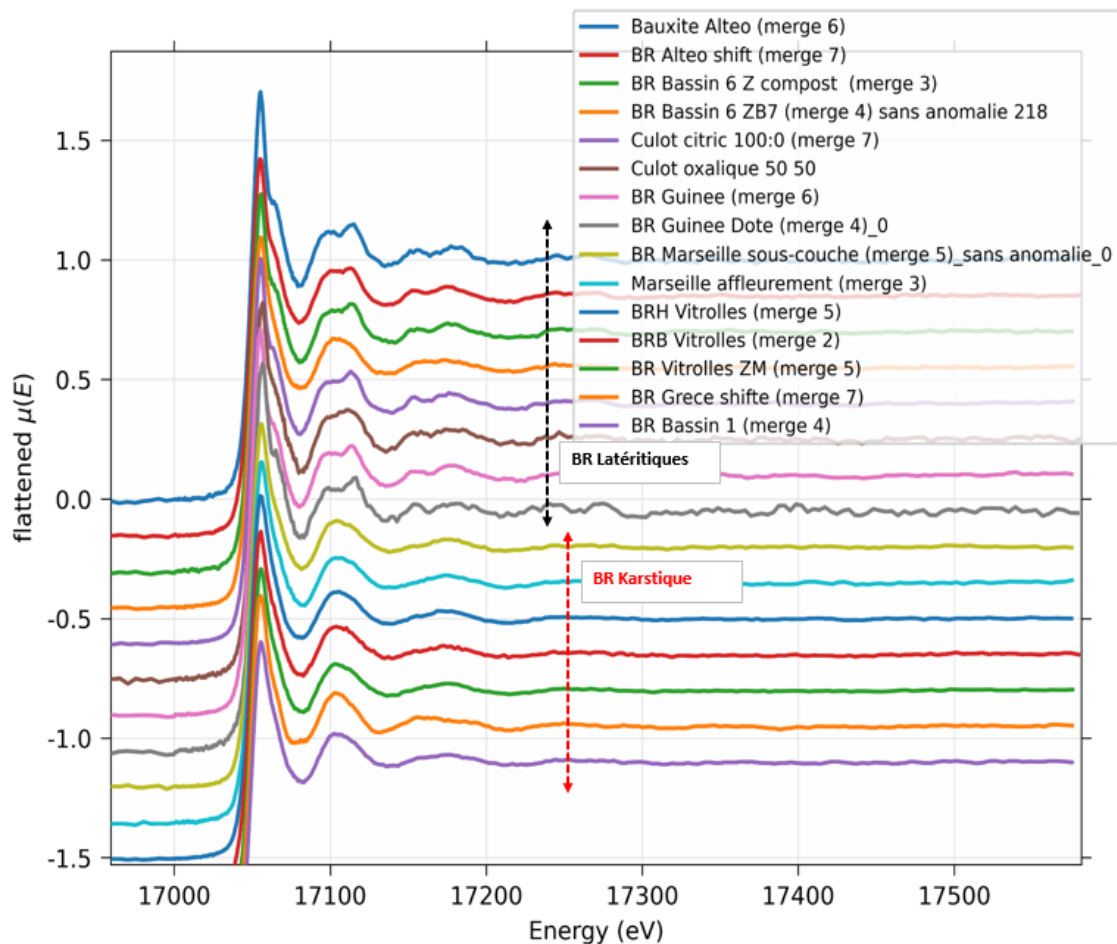


Figure 1. Yttrium XANES spectra for a set of bauxite residue with different ore origin (karstic vs lateritic), storing conditions (stockpile, laguna) and age (0-100 years).

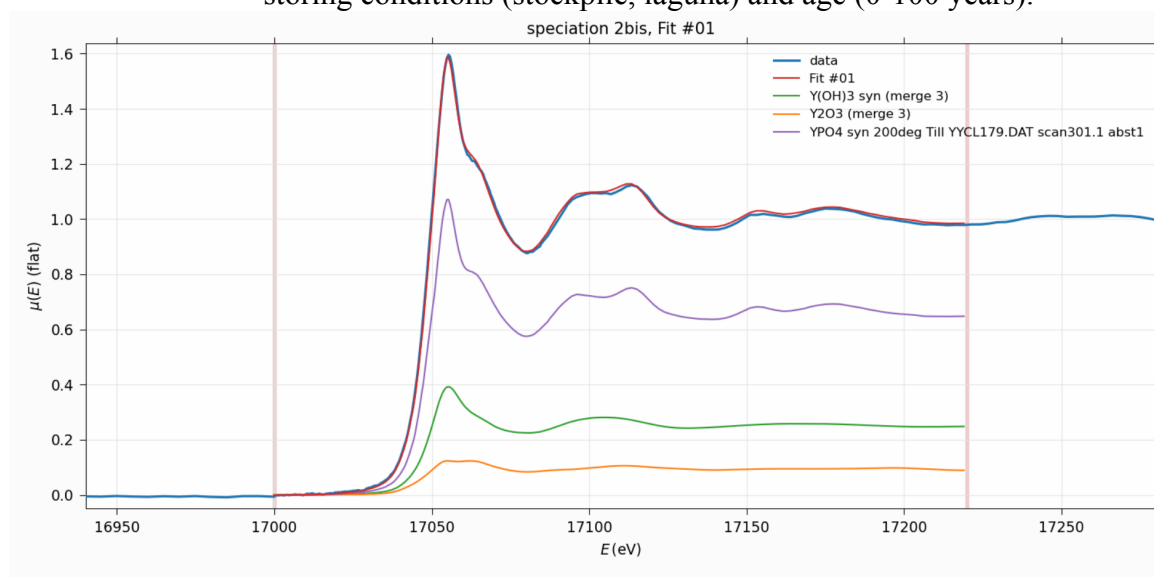


Figure 2. Linear Combination Fitting of the bauxite residue from Alteo (Gardanne, France)

Regarding question 3, leaching residues obtained from different conditions were analyzed:

- (i) organic acids in water at pH 4 (citric acid and oxalic acids). Speciation in the leaching residue is very similar to the initial speciation in the bauxite residue and consist mostly in yttrium phosphate phases.
- (ii) Because of the complexity of the DES systems and the poor knowledge of dissolution mechanisms in these systems we finally decided to look at the speciation of Y in modeled system that mimick the speciation of Y in bauxite residue. Thus, yttrium phosphate dissolution was tested in choline chloride :

lactic acid DES system and both the supernatant and the leaching residue after extraction were analyzed by XAS. No differences of speciation was observed in the supernatant and in the solid residue and consisted in yttrium phosphate. The suspected dissolved Y present in the supernatant consisted in small yttrium phosphate particles that were not separated during the centrifugation step.

### **Issues encountered:**

No particular technical issues were encountered.

### **Perspectives**

Further analysis of XANES and EXAFS spectra of the different bauxite residues will be performed to be published in a peer-reviewed article that will be part of the PhD project of J. Couturier and C. Lallemand

Regarding the DES systems, other model systems will be explored with light REEs and other modeled minerals including REE -oxide, -hydroxide and -carbonate to cover the different REEs speciation that can be found in the environment.