ESRF	**Experiment title:** Development of functionalised biogenic magnetic nanoparticles for the sustainable extraction of critical metals from waste	**Experiment number**: EV-535
Beamline: BM 28	**Date of experiment**: from: 05.04.2023 to: 11.04.2023	**Date of report**: 04.10.2023
Shifts: 18	**Local contact(s)**: Dr Laurence Bouchenoire	*Received at ESRF:*

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Report:

Critical metals are under intense global demand due to their usage in renewable technologies. The development of new extraction methods for metals such as cobalt, nickel, and lithium is thus gaining importance. In this project, we are developing magnetic nanoparticles (MNPs) as functionalized sorbent materials for recovering these critical metals from waste. We anticipate that biogenic MNPs will be more effective than abiogenic alternatives due to the presence of reactive organic molecules, which will promote metal complexation. After carefully conducting the batch experiments in the laboratory of the School of Earth Sciences, we found out that abiogenic MNPs work best at pH 7 for Co and Ni recovery from solutions. For both materials, Co(II) and Ni(II) sorption followed a Freundlich rather than Langmuir type isotherm.

At the BM28/XMaS beamline, we successfully coupled X-ray absorption near edge spectroscopy (XANES) and extended X-ray absorption fine structure (EXAFS) to determine the oxidation state and bonding environment of abiogenic MNPs before/after sorption to Co(II) and Ni(II). Preliminary analysis reveals a clear association between metals and MNPs.

We mainly measured 1. XANES and EXAFS at Fe K-edge for the magnetite samples sorbed with and without different concentrations of Co(II) or Ni(II), 2. XANES at Co K-edge and 3. XANES at Ni K-edge. We also conducted XAS of Fe, Ni, and Co foils and references.

Preliminary investigations showed that we could successfully distinguish the emission lines of Fe Kα1, Kα2, Kβ, and Co Kα for collecting the Fe (XANES+EXAFS; transmission mode) and Co (XANES, fluorescence mode) data. The collected XANES data is compiled in Figure 1.

Further evaluation of EXAFS data is ongoing to understand which Fe site is coordinated with the critical metal upon sorption reactions. Shell-by-shell fitting of EXAFS data can provide such information, including bond distances between Fe-Fe, Fe-O, and/or Fe-O-Co/Ni.

In the April 2023 round of our beamline visit, we obtained extensive knowledge about the facility and available resources from beamline scientist Laurence Bouchenoire and Postdoc Olga Filimonova. In further rounds, we will build on this knowledge and utilize both transmission and fluorescence detectors to measure the XAS spectra of our samples.

A manuscript is being prepared based on the data collected at the BM28 in this round.

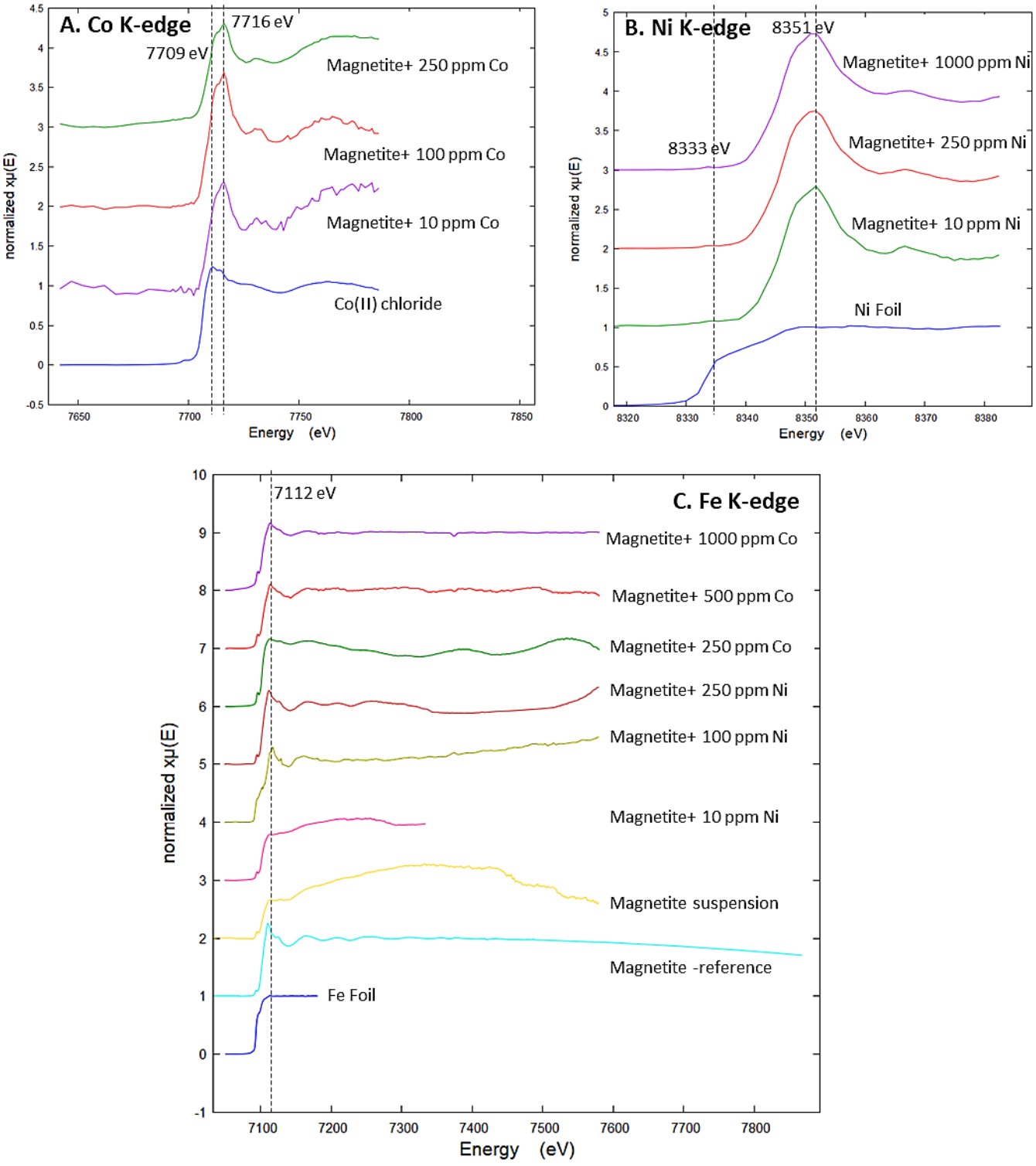

Figure 1. (A) Co K-edge XANES spectra of Co sorbed onto abiogenic magnetite. (B) Ni K-edge XANES spectra of Ni sorbed onto abiogenic magnetite. (C) Fe K-edge XANES spectra when (selected treatment of) Co or Ni sorbed onto abiogenic magnetite. Solid (drid in glovebox after synthesis) and suspended magnetite (5 g/L), Fe foil, Ni foil, Co(II) chloride were used as reference.