ESRF	**Experiment title:** **Probing electronic structure of uranium after adsorption on organofunctional Na-P1 zeolite by applying the U-L_3 edge HERFD-XANES/EXAFS spectroscopies**	**Experiment number**: CH-6759
Beamline: ROBL-II BM20	**Date of experiment**: from: **01.12.2023** to: **05.12.2023**	**Date of report**: 19th of March 2024
Shifts: 12	**Local contact(s)**: André Rossberg	*Received at ESRF:* **19th of March 2024**

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Report:

This report referes to the X-ray Absorption Spectroscopy (XAS) experiment carried out between 1st of December 2023 and until 5th of December 2023 at the ROBL-II BM20 beamline of the ESRF titled: “Probing electronic structure of uranium after adsorption on organofunctional Na-P1 zeolite by applying the U-L_3 edge HERFD-XANES/EXAFS spectroscopies" which was awarded the beamtime by the Committees based on the official letter received by the Main Proposer on 2nd of June 2023.

The experiment was carried out in the RCH-1 (research hutch 1) of the BM20 beamline in the dedicated glovebox EXAFS endstation using He-cooled cryostat for dedicated U-L_3 XANES/EXAFS spectroscopy sample holders. The analyses started at the BM20 beamline on 1st of December with calibration of the spectrometer, finding the X-ray beam, slits and mirror adjustments as well as with the placement of the sample in the dedicated sample holders in the RCH-1 of the beamline where the EXAFS station with He-cooled cryostate is located. Energy calibration of the spectrometer was done using Y foil (Kα line = 17,038 eV) measured simultaneously with each sample uring each scan. For the first batch of 5 samples (UN, UA, P3, P4, and P5, according to the IAF Form), 5 spectra for each sample were collected to achieve statistically good averaged spectra with low background noise. This batch ended around 1p.m. of 2nd of December. Afterwards, the Main Proposer and the

Local Contact changed the sample holder to another batch of four (4) samples. It is worth highlighting that we measured the samples in the following batches to distribute equally the gross amount of the samples to be measured throughout the allocated beamtime (5 samples, 4 samples, and 5 samples).

We decided to measure the following 5 samples duirng 2nd of October, 3rd of October and to terminate this batch early in the morning of 4th of December. Considering that second batch of 5 samples was crucial for the experiment, it was decided to increase the number of scans for each sample to 15. The measurement was undisrupted, and in the morning of 4th of December, a second change of samples took place. At 8 a.m. the final batch was subjected to the measurement with 5 spectra collected for each sample.

The measurements were undistorted, and initial EXAFS signal extraction took place at the beamline for the first batch of investigated samples (two U reference salts - U nitrate hexahydrate and U acetate, respectively, as well as the native Na-P1 zeolite, after U removal, prior organic modification using a biodegradable surfactant, at three pH_{eq} levels.

Currently, the data treatment take place with FT of the EXAFS signals for samples modified with biosurfactant (CB3, CB4, CB5), and a (C3, C4, C5, M3, M4, M5), as well as the samples with native material (Na-P1 coal-fly-ash zeolite). The initial assessment of the collected XANES spectra at U-L_3 edge with respective EXAFS signal extraction and FT analysis is shown as Figure 1.

It is worth highlighting the fact, that the solid-state characterization of Na-P1 zeolite and biosurfactant-modified zeolite is a part of the publication devoted to the U removal onto biosurfactant-functionalized Na-P1 zeolite, which is currently under construction, and is aimed to be submitted to Journal of Hazardous Materials in Q1/2024. The publication is aied to be the third manuscript devoted to U removal onto functional mineral materials, and is a part of the PhD dissertation prepared at the AGH University of Cracow Doctoral School by the Main Proposer.

The XANES/EXAFS spectroscopies will complement other spectroscopies such as FT-IR, SEM-EDXS , and XPS to elucidate how U binds to the newly engineered organo-mineral composite at the nano-scale level. The working title of the publication is as follows: **“Highly efficient uranium uptake by the eco-designed cocamidopropyl betaine-decorated Na-P1 organo-zeolite - elucidation via batch, laboratory, and synchrotron spectroscopies**”.

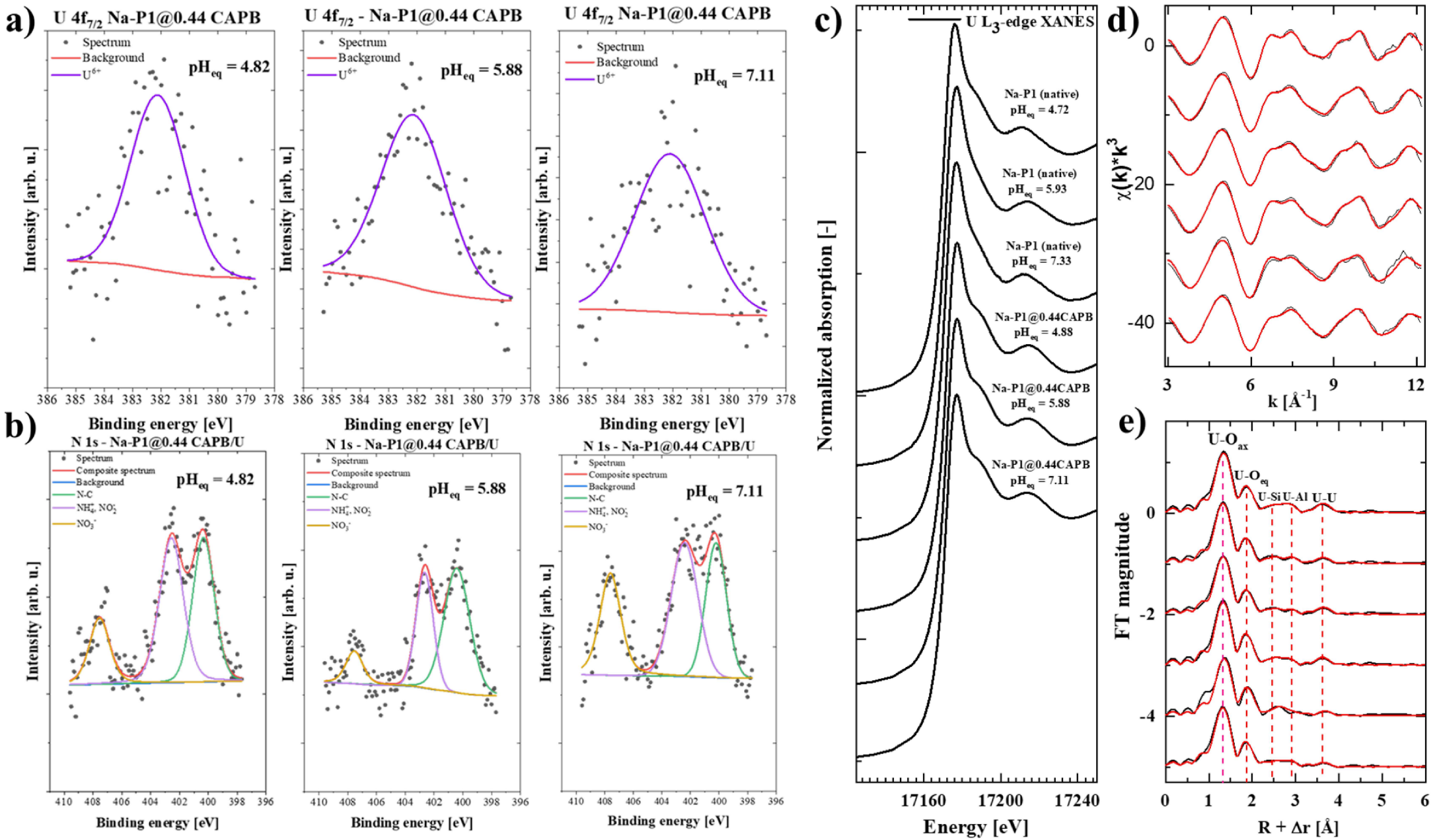

Figure 1. a) The high-resolution XPS spectra of U-4f$_{7/2}$ line, b) and N 1s line, c) U-L$_3$ edge XANES spectra for the native Na-P1 zeolite and Na-P1@0.44CAP B composites with d) EXAFS analysis and e) respective FT of the EXAFS signal revealing electronic structure of scavenged U.