Phosphorus K-edge XANES analysis of environmental samples: critical steps and limitations

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Phosphorus K-edge XANES spectroscopy has rapidly become an often used method for P speciation in environmental samples. Here we discuss the critical steps and considerations that need to be taken during data collection, analysis and interpretation, to ensure the best possible quality of the data. Three examples of crtical steps are:

(a) Frequent checks of the energy calibration during the beamtime and carefully made corrections for shifts/drift are of vital importance for the quality of the results

(b) Standards for linear combination fitting (LCF) should be processed at the same beamline as the samples and cover all types of P species of interest. At present, our own database at BL-8, SLRI, Thailand, includes > 30 standards that include P sorbed to Al and Fe hydrous oxides, Ca, Al and Fe phosphates, and organic P model compounds.

(c) Consistent normalization on the P K edge requires the use of relatively small pre- and post-edge ranges, and is aided by careful sample preparation that stabilizes the baseline [1, 2]

With these and other precautions, we have determined P speciation in a large variety of soils as well as in slag materials, and the results are shown to agree well with those expected from theory and from wet chemical extractions.

There are, however, certain problems that remain concerning the use of the K edge for XANES-LCF speciation, three of which are: (i) Organic P species are not well separated due to their relatively featureless spectra; (ii) For samples in which Fe- and Al-sorbed P represent a small part of total P, it is difficult to determine the relative importance of these phases; and (iii) The “real” uncertainty of LCF is difficult to assess given the lack of specificity of this analysis for P species, but it is certainly much larger than that reported with LCF analysis by XAFS software, e.g. Athena.

[1] Werner & Prietzel (2015) *Environ. Sci. Technol.* **49,** 10521-10528, [2] Eriksson et al. (2016) *Sci. Total Environ.* **280,** 29-37.