BL-11B/2013G606

Phosphorus speciation in agricultural byproducts determined by P K-edge XANES

Yohey Hashimoto[1*] and Noriko Yamaguchi[2]
[1]Tokyo University of Agriculture and Technology, Tokyo 184-8588, Japan
[2] Natural Institute for Agro-environmental Sciences, Tsukuba 305-8604, Japan

1 Introduction

To understand availability and chemical speciation of P in various fertilizers and soil samples, macro- and molecular-scopic techniques have been employed. A method of sequential fractionation has widely been used, which classifies P using difference chemical fluids. However, the results of chemical fractionation are defined by operationally-based P classification and are not directly determine P speciation. In combination with chemical fractionation techniques, the use of X-ray adsorption near edge structure (XANES) spectroscopy provide more detailed and precise information on P species in the environmental samples. Phosphorus K-edge XANES spectroscopy is a non-destructive method and can distinguish inorganic forms of Al, Fe and Ca phosphates in manure samples [1-2]. The objective of this study was to investigate chemical speciation of P in various P fertilizers derived from agricultural byproducts using P K-edge XANES spectroscopy.

2 Experiment

Agricultural byproducts including poultry litter, bone powder and fish lees powder were air-dried and analyzed for the P K-edge XANES. The study was conducted using the beamline BL11B equipped with Si(111) monochromator in a fluorescence mode at ambient temperature under a vacuumed condition. The XANES spectra were collected with a step wise in 0.1 eV increments with 1 s/point from 2130 to 2190 eV and in 0.5 eV increments with 1 s/point from 2190 to 2220 eV. Multiple scans across the P K-edge were averaged. Known reference P compounds such as hydroxyapatite were also measured and their XANES spectra were compared with those of agricultural byproducts.

3 Results and Discussion

Figure 1 showed selected XANES reference spectra that are representative of soil P species. Calcium phosphate references including hydroxyapatite were characterized by a well-defined shoulder on the high energy side. Compared to these P compounds, organic phosphate references including Ca-phytate exhibited featureless spectra. Strengite was characterized by an intense white-line peak in a narrow energy range. Strengite exhibited a pre-edge between -5 and -2 eV, which can be a distinctive features to differentiate them from other phosphate compounds.

The XANES spectrum of bone powder was characterized by a shoulder at 3 eV and a small peak at 11 eV, which corresponded to the spectral features exhibited in hydroxyapatite. The samples of poultry litter and fish lees also had a shoulder around 3 eV, suggesting the presence of hydroxyapatite-like compounds. Samples like fish lees were abundant with organic material, and thereby organic P may also be present. Due to featureless XANES spectra, however, organic P was difficult to differentiate from other inorganic P species in these samples.

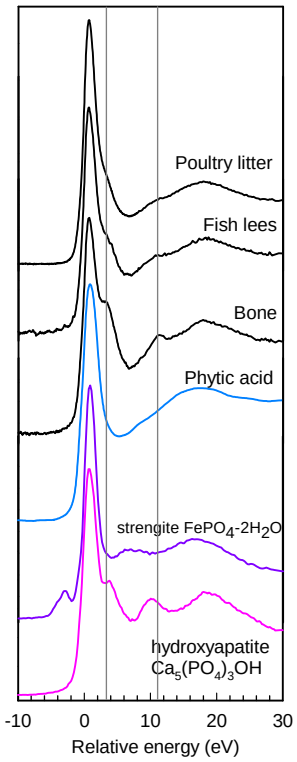

Fig. 1: P K-edge XANES spectra of byproduct samples and P references

Acknowledgement

The authors are grateful to Dr. Y. Kitajima for supporting the experimentation at the beamline. The samples of agricultural byproducts were kindly provided by Dr. K. Murakami (Mie Prefecture).

References

[1] Negassa et al. Environ. Sci. Technol. 44, 2092 (2010)
[2] Shober et al. J. Environ. Qual. 35, 1983 (2006)

* yhashim@cc.tuat.ac.jp