Advances in phosphorus speciation in environmental samples by Synchrotron-based X-ray absorption Near-Edge Spectroscopy

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In developing countries P from agro-industrial byproducts can be either a significant source of freshwater pollution or a valuable fertilizer for soil amendment. For both cases, it is important to speciate the P in common byproducts. We have investigated dry (COD) and wet (COW) coffee, sisal (SIS), barley malt (BEB) and sugarcane processing (FIC) byproducts, and filter cakes of linseed (LIC) and niger seed (NIC) with sequential P fractionation, solution ^{31}P nuclear magnetic resonance (NMR) spectroscopy, and X-ray absorption near edge structure (XANES) spectroscopy at the P *K*- and $L_{2,3}$-edges. Sequential fractionation recovered 59 to about 102% of total P (P_t), and more than 50% of P_t was extracted by H_2O and $NaHCO_3$ in five out of seven samples. Since sequential fractionations only provided a first hint to potential P mobilization but no direct speciation, we investigated the residue after each extraction step by P *K*- and $L_{2,3}$-edge XANES. Semiquantitative evaluation of P *K*-edge spectra of by linear combination (LC) fitting as well as the $L_{2,3}$-edges spectra provided unequivocal evidence for the enrichment of Ca-P along the extraction sequence and its complete removal by the H_2SO_4-treatment for the FIC sample. The LC fitting was unsuccessful for the NIC samples, most likely because of the absence of significant proportions of Ca-P as confirmed by both P XANES methods (Kruse et al., submitted). In the bulk samples of agro-industrial byproducts, the results of sequential fractionation, especially the proportions of inorganic and organic P were confirmed by ^{31}P NMR spectra. Again, only the combined application of P *K*- and $L_{2,3}$-edge XANES provided unequivocal evidence for the abundance of Ca-P phases in a few samples (Negassa et al. 2010). The results strongly called for the combined use of all these four analytical methods for a comprehensive P speciation in environmental samples but P-XANES at the P *K*- and the $L_{2,3}$-edges provided the best information on mineral P phases. Furthermore, for environmental protection and P nutrition of field crops the byproducts should be applied to soils instead of dumping.

Kruse, J., W. Negassa, N. Appathurai, L. Zuin, and P. Leinweber. Phosphorus speciation in sequentially extracted agro-industrial byproducts: Evidence from X-ray absorption near edge structure spectroscopy. J. Env. Qual. (submitted).

Negassa, W., J. Kruse, D. Michalik, N. Appathurai, L. Zuin, and P. Leinweber. 2010. Phosphorus speciation in agro-industrial byproducts: sequential fractionation, solution ^{31}P NMR and P *K*- and $L_{2,3}$-edge XANES spectroscopy. Environ. Sci. Technol. 44:2092–2097.