



	Experiment title: Changes of Cu and Zn speciation during sewage sludge incineration	Experiment number: 01-02-1160
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Report:

During this experiment, we completed a Cu and Zn K-edge XAS dataset for a study on the transformation of Cu and Zn during anaerobic digestion and sewage sludge incineration, which has been published recently (Wielinski et al., 2019).

In addition, selected samples resulting from the pyrolysis of digested sludge spiked with Cu and Zn in nanoparticulate or dissolved form were also investigated: The char samples were ground to a fine powder and pressed into 7 mm pellets for XAS measurements at the K-edge of Cu (8979 eV) and Zn (9659 eV). A closed-cycle He-cryostat set to 80 K was used to cool the samples. All samples were measured in fluorescence mode using a 9-element monolithic Ge fluorescence detector (Canberra, CT, USA). Reference spectra of ZnO (zincite), ZnS (sphalerite), gahnite (ZnAl_2O_4), CuO (tenorite) and CuS (covellite) were measured in transmission mode. Linear combination fit analysis (LCF) was used to evaluate Cu and Zn XANES (-20 to +120 eV around edge) and Zn EXAFS data (k-range 3 to 9 \AA^{-1}).

Zn XAS revealed a reduction in Zn-S coordination at temperatures $\geq 600^\circ\text{C}$ and a concurrent increase of the fraction of a (weakly crystalline) Zn(-Al) spinel phases, independent of the dosed Zn form. For Cu, XAS data suggested that pyrolysis reduces the fraction of CuS, which dominated in the sludge samples, and increases the fraction of a chalcopyrite-type phase. In contrast to Zn, no distinct (nanoparticulate) Cu enrichments were observed in the char using transmission electron microscopy. In line with our results for ashes from sludge incineration (Wielinski et al., 2019), also sludge pyrolysis results in a Cu and Zn speciation in char that is independent of whether Zn or Cu was spiked to sludge in dissolved or nanoparticulate form. These results are currently being prepared for publication in a peer-reviewed journal.

Publication

Wielinski, J.; Gogos, A.; Voegelin, A.; Müller, C.; Morgenroth, E.; Kaegi, R. Transformation of Nanoscale and Ionic Cu and Zn during the Incineration of Digested Sewage Sludge (Biosolids). *Environ. Sci. Technol.* **2019**. <https://doi.org/10.1021/acs.est.9b01983>.