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Redox transformations in wildfire ashes studied by micro X-ray absorption spectroscopy

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Analysis of wildland-urban interface (WUI) fire ashes collected from the 2020 LNU Lightning Complex Fire perimeter in Northern California, USA, has been performed using Micro X-ray fluorescence (µXRF) imaging and micro X-ray absorption fine structure spectroscopy (µXAFS). Changes in metal(loid) speciation (e.g., oxidation states of Cr, Mn, As, and Ti) were addressed as a result of combustion processes and high-temperature reducing microenvironments. These experiments were conducted at beamlines 4-BM at the National Synchrotron Light Source II (Brookhaven National Laboratory) and 13-ID-E at the Advanced Photon Source (Argonne National Laboratory). The µXRF elemental mapping enabled the identification of high- and low-concentration spots of different metal(loid) and correlations among them. Our findings demonstrate that a mixture of both oxidized and reduced species are present in the WUI fire ashes. The spatially resolved X-ray microprobe analyses provide a comprehensive picture of the end-member species and intermediates that were not detected using bulk XAFS spectroscopy. The µXAFS analysis allows us to establish reliable reference spectra for linear combination fitting analysis of bulk XAFS measurements. Overall, these results have demonstrated complex transformations of metal(loid)s in WUI fire ashes. In particular, the reduction of TiO₂ to the sub-stoichiometric Magnéli phases (TiO_{2n-1}), As(V) to As(III), Mn(IV) to Mn(II), and the oxidation of Cr(III) to Cr(VI). These metal(loid) transformations help to explain the higher toxicity of WUI fires compared to particulate matter from other sources, and the importance of understanding the pollution characteristics and health risks of WUI fire ashes and associated particles, which are currently poorly understood on the micro- and submicrometric scale.