Speciation of 3d-Metals in Slags via Laboratory-Based X-Ray Absorption Near Edge Spectroscopy

Alena Wittkowski[1], Ursula Elisabeth Adriane Fittschen[1]

[1] Clausthal University of Technology, Institute of Inorganic and Analytical Chemistry, Arnold-Sommerfeld Straße 4, 38678 Clausthal-Zellerfeld, Germany

alena.wittkowski@tu-clausthal.de

Chemical recycling of slags is essential for regaining valuable and critical materials. In pyrometallurgical processes like the production of steel or the recycling of batteries, slags are formed to remove certain impurities from the alloy. During the conversion, not only impurities migrate into the slag but also valuable elements with a high chemical affinity to oxygen like vanadium or lithium.

Today, the recycling of slag is limited to the production of cement or for construction works. To put a chemical recycling into practice and separate valuable materials from the slag, new strategies need to be applied. One is to create engineered artificial minerals (EnAM) by controlling the formation of the slag via the formulation and the cooling conditions. Ideally, the target element forms a recoverable crystalline phase, which differs physically or chemically from the rest. This makes it easy to separate. For this, it is essential to characterize the slag.
The crystalline structure and elemental distribution is easily characterized by X-ray diffraction (XRD) and electron micro probe analysis (EMPA). To investigate the elemental species also in the amorphous material we are using X-ray absorption near edge spectroscopy (XANES) as a complementary method to XRD. This method allows us to study the chemical environment as well as the coordination sphere of redox sensitive elements like vanadium, manganese, iron, and chromium, which is essential to identify the corresponding oxides in the slag.

Until now, XANES is mainly performed using synchrotron radiation sources. In this study, we are using a novel laboratory-based XANES instrument.
The concentration of the element oxides studied by XANES is in the range between 5 and 50 weight percent. The lowest concentration at which a speciation can be done will be determined for each element. Due to the calcium rich matrix, background absorption is fairly high, and the sample preparation needs to be adapted to obtain meaningful spectra.

Here we will present preparation strategies, first laboratory-based XANES results in evaluating a convenient concentration range, and first measurements of slag-like calcium silicates containing 3-d elements.