ESRF	**Experiment title:** Zinc mobility and chemical stability in the biogenic tooth nanocomposite of dentine	**Experiment number:** MD1385
Beamline: ID21	**Date of experiment:** from: 08.06.2023 to: 14.06.23	**Date of report:**
Shifts: 14	**Local contact(s):** Hiram Castillo, Clément Holé	*Received at ESRF:*
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Report:

Zinc is a natural trace-element in the natural biocomposite dentine that makes up the bony part of teeth. In roots following root canal treatment, Zn can diffuse out of artificially placed filling materials, as occasionally reported for clinically-used dental biomaterials (e.g. root canal filling gutta-percha). Diffusion is extensive with concentrations that can percolate many hundreds of µm across the bulk of the tooth, as we recently observed. This diffusion may cause changes in the micro-structure and chemistry of dentine which have to be understood to determine benefits or possible degradation. Filling materials typically contain ZnO while natural Zn can either be incorporated in the apatite crystals or reside in the organic component, for example in MMP enzymes. To better understand the atomic environment surrounding Zn, we investigate dentine by micro and nano XANES. **We proposed to map the absorption edge of Zn to elucidate relationships between resident and diffusion species chemical states in treated healthy dentine, near filling material.**

What we planned:

Recently slaughtered, fresh bovine teeth are routinely used in dental research as alternatives to young, healthy human teeth. Such ca. 2 year old teeth do not have a history of dental treatment and are usually intact and abundant, allowing experimental reproduction. We prepared 7 samples with ~50 µm thickness fixed with double-sided carbon tape onto plastic slides:

3 slices of bovine tooth were treated with a ZnO rich paste (Life, KERR) artificially placed in the root canal for 1 year;

2 slices were taken from a bovine tooth that was root canal treated with conventional Gutta percha filling materials in sealer cement.

3 materials of reference: Life filling material, Gutta percha, Scholzite (Ca, Zn phosphate mineral).

Zn diffusion was previously characterized in the lab with µXRF at the BLiX laboratory of the Technical University of Berlin. In areas of diffusion, Zn K XANES measurements were performed at the interface between filling and dentine – collecting Zn K XANES spectra at increasing distances from the fillings as well as using Zn K XANES imaging.

Experimental Outcomes:

We collected point Zn K XANES measurements and XANES imaging on all samples. Prior to full XANES measurements, XRF mapping was performed in areas of diffusion, see figure 1.

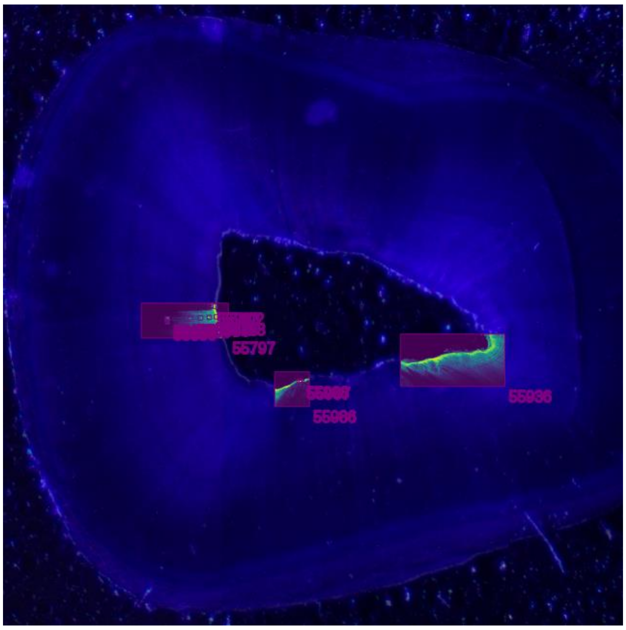

Figure 1: Overview microscopy image of a root cross section in areas of diffusion observed in the lab (22Life Bov-sl4)

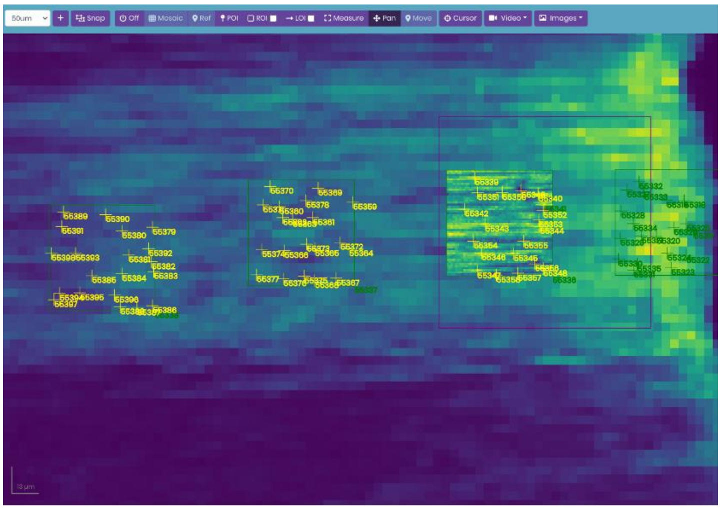

Figure 2: Zn K map of a bovine tooth slice treated with ZnO paste(18LifeBov_sl2). The pulp side where the biomaterial was placed is to the right. Diffusion of Zn along tubules can be clearly discerned. Four regions at increasing distances from the pulp were selected with ~ 20 points of measurement each for Zn K XANES spectra.

Subsequently, 50 x 50 µm² areas were mapped with high resolution at increasing distances from the pulp chamber, see figure 2. In each region approximately 20 positions (with high and low Zn K intensity) were identified and selected for the collection of XANES spectra. For several selected areas close to the pulp chamber, detailed XANES imaging maps with high resolution were collected, see figure 3.

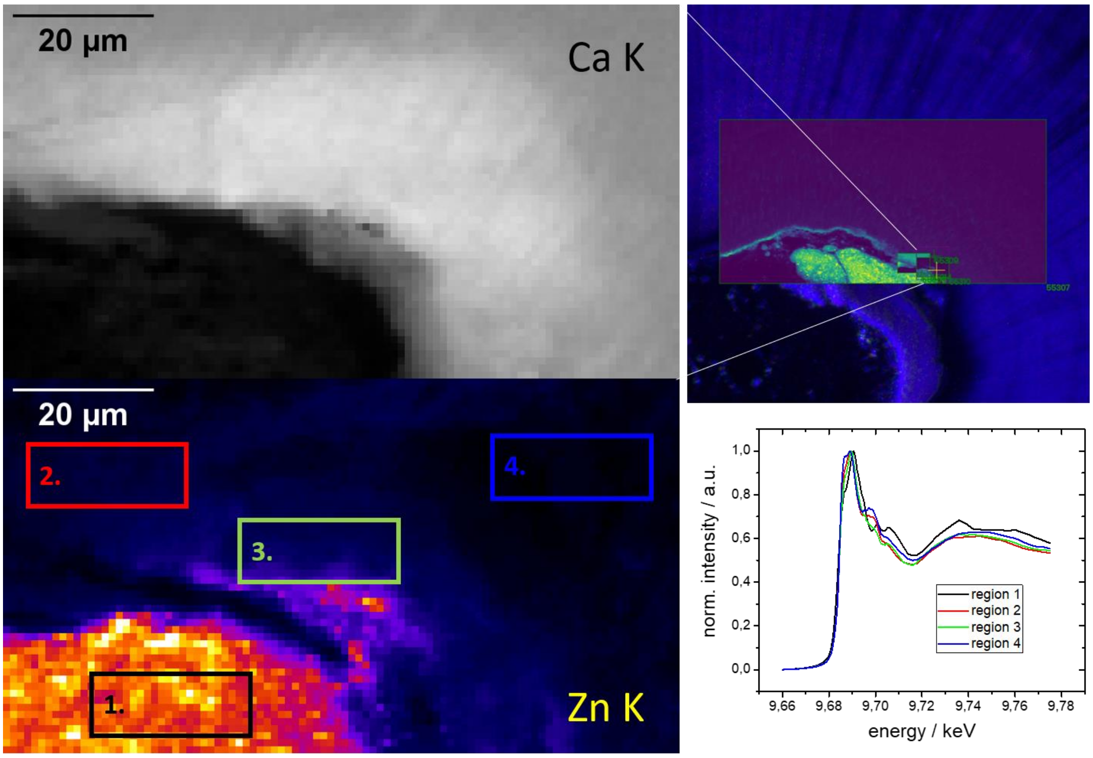

Figure 1: maps of X-ray scanned regions: left: Ca and Zn K intensity maps with 1 µm pixel size collected in the region identified in the microscope image on the right showing a microscope image overlaid with Zn K distributions;. bottom: Zn K XANES sum spectra of the four regions marked in the Zn Kα distribution.

After performing measurements, all samples cracked inducing movement only reaching a stable state after some hours in the beam. This is attributed to internal stress of the samples, which was released due to irradiation.