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High-resolution imaging of soil colloids in aqueous media with a compact soft X-ray microscope

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Abstract. Colloids play an important role when describing parameters of and processes within soils, sediments or aquifers due to their abundance and their high specific surface area. It is of great importance to visualize the morphology of the structures formed by these particles as close as possible to environmental conditions. With X-ray microscopy colloids from the environment can be imaged directly in aqueous media with high spatial resolution. We demonstrate the first use of a compact laboratory x-ray microscope for studies of colloids from the environment, namely aqueous suspensions of clays and soils. The microscope is based on a high-brightness laser-produced-plasma X-ray source, a multilayer mirror and diffractive optics. The experiments show that such compact X-ray microscopes are reaching the image quality and operational maturity to make a significant impact in fields like environmental sciences.

1. Introduction to colloidal structures in soils
Soils are penetrated by atmosphere, hydrosphere and geosphere. Its fabric is expressed in the pore system of soils, where the distribution of the pore radii shows a wide range from mm to sub-μm. This pore system determines the inner surface of a soil. It affects many important soil parameters and processes as turnover of nutrients, adsorption of toxicants, bioavailability of these substances, water storage capacity and water flow. Pore radii of some μm or less are the most frequent, thus contributing to the largest part of the inner surface. These micro pores are mainly built up by inorganic colloids such as clays, quartz particles, iron and aluminium oxides, or organic colloids as bacteria, fungi, or humic substances. To better understand parameters and processes it is necessary to have a close look at the microstructure built up by these colloids. Due to their large specific surface area (e.g. clays with up to 800 m²/g) and their small size many physical, chemical and biological processes in soils are influenced by these particles. Due to this particle size and as nearly all chemical reactions occur in aqueous phase, X-ray microscopy is a technique well suited for these purposes.

2. Compact X-ray microscopy
Successfully running X-ray microscopes have been installed at synchrotron radiation facilities, the most commonly used source for X-ray microscopes today. However, only few electron storage rings
exist worldwide and the accessibility is limited, hampering the dissemination of X-ray microscopy as a useful technique. Using a high-brightness and minimum-debris regenerative liquid-jet laser-plasma source, a normal-incidence spherical multilayer condenser mirror and a micro zone plate, colloids from the environment in aqueous media have been imaged at $\lambda=3.37\text{nm}$. A micro zone plate with $d_m=25\text{nm}$ has been used as microscopic objective, and the resulting resolution in the images is of about the same value [1, 2]. Image and illustration in figure 1 show the arrangement of the compact laboratory soft X-ray microscope at the KTH. The images shown in this paper have been taken with exposure times of up to 600 seconds.

3. Clay dispersion
A dispersion of nontronite clay has been chosen to demonstrate the necessity for imaging colloidal particles in aqueous suspension. Nontronite consists of small particles stacked together forming open structures within the soil solution. When falling dry, the delicate structures collapse. This change in morphology will make it impossible to assess the real internal surface under ambient conditions, i.e. when small pores within the soil are filled with water.

4. Soil colloids
Figure 2 shows images of samples from a chernozem soil show different appearances of colloidal particles, forming clusters. The trained eye quickly identifies in each sample particles of similar shapes. Small, optically dense clay particles are the majority of the particles visible. The cluster OF the center in the left image can be identified as a larger central clay particle with smaller particles attached. Due to the frayed edges of the smaller features they could possibly be of organic origin. The image diameter is $\sim2000$ pixels, corresponding to 20 $\mu\text{m}$ in the sample plane.

5. Discussion and Outlook
It has been clearly demonstrated that it is possible to visualize the morphology of colloidal structures from the environment with a compact laboratory X-ray microscope. The pictures shown here have been taken with exposure times of 600 seconds, which is of course a major drawback in comparison to exposure times in the range of seconds or less using X-ray microscopes at electron storage rings. The
use of a state of the art laser with improved pulse energy, pulse duration and repetition rate will shorten the exposure time with about a factor 10. Equipping the microscope with a multilayer condenser mirror for the \( \lambda = 2.48 \text{ nm} \) source, thereby increasing the numerical aperture and the collection efficiency of the condenser WILL shorten the exposure time even further. Finally, the transmission for thicker, \( \sim 10 \mu\text{m} \), aqueous samples at this wavelength is \( \sim 5 \) times higher than for \( \lambda = 3.37 \text{ nm} \). This contributes also to shorter exposure times. All together these planned improvements lead to exposure times comparable to the time needed with synchrotron-based microscopes. Such short-exposure-time compact X-ray microscopes readily accessible in the laboratories of environmental sciences would make powerful tools for future studies.

**Figure 2.** Cluster of nontronite particles in a dry (left) and an aqueous state (right).

**Figure 3.** Colloid clusters from a Chernozem soil in aqueous suspension.

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