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X-Ray Microanalysis of Fully Wet Samples Using WETSEM™ Technology

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Characterization of chemical microstructures is one of the most important applications of the scanning electron microscope (SEM) equipped with an energy dispersive x-ray spectrometer (EDS). However, one of the challenges this technique was facing is the ability to apply it to wet samples. Typical examples include creams, solutions, suspensions and other liquid containing samples.

In this work we show that the recently developed WETSEM™ technology provided the solution and enabled direct observation and chemical analysis of samples in their native wet state. WETSEM™ uses conventional scanning electron microscopes equipped with a standard EDS detector. The technology was used to study the chemical composition of samples from a variety of fields of use, including material science and bio-science. Samples of creams, solutions, and other liquid containing specimens such as suspensions of metal, ceramics and mineral particles were analyzed.

When using WETSEM™ technology, the sample is placed in the sealed specimen capsule (the QX capsule), and is isolated from the vacuum by a thin, electron-transparent membrane. A metal grid is mechanically supporting the membrane. The thin membrane is used as a window through which imaging and x-ray analysis is carried out. The contribution of the membrane to the sample EDS spectrum was investigated and shown to be negligible. Since the thickness of the membrane is a few hundreds of nanometers and the x-ray signal comes from a much thicker layer the membrane does not interfere with the EDS measurement and its contribution to the EDS spectrum is manifested only by the carbon peak. The grid consists of 330x330 μm windows. When an EDS spectra is generated from an area within such window the elemental signature of the grid is negligible. This work shows results that validates these conclusions

For identification of particles, X-ray spectroscopy (EDS) was applied to a particle of unknown composition. Selected area microanalysis was also used in order to distinguish between adjacent areas in the sample and evaluate homogeneity.

Fig.1 shows an SEM image (a) and related selected-area spectra of the membrane (darker area in the image) and supportive grid (bright cross), fig. 1(b) and 1(c) respectively. The capsule was filled with double distilled water. The grid elemental composition is evident only when the analyzed area contains parts of the grid. The spectra analyzing the membrane and the capsule contents shows the carbon contribution of the membrane and the oxygen peak produced by the water.

Fig. 2 shows the WETSEM image (a) and the full frame EDS spectrum (b) of dead sea mud courtesy of AHAVA®. Fig. 2c shows a spot analysis of a single particle denoted in the image by (C). The full frame spectrum provides the average chemical composition of the area seen in the image while the spot analysis gives the elemental composition of a specific particle.

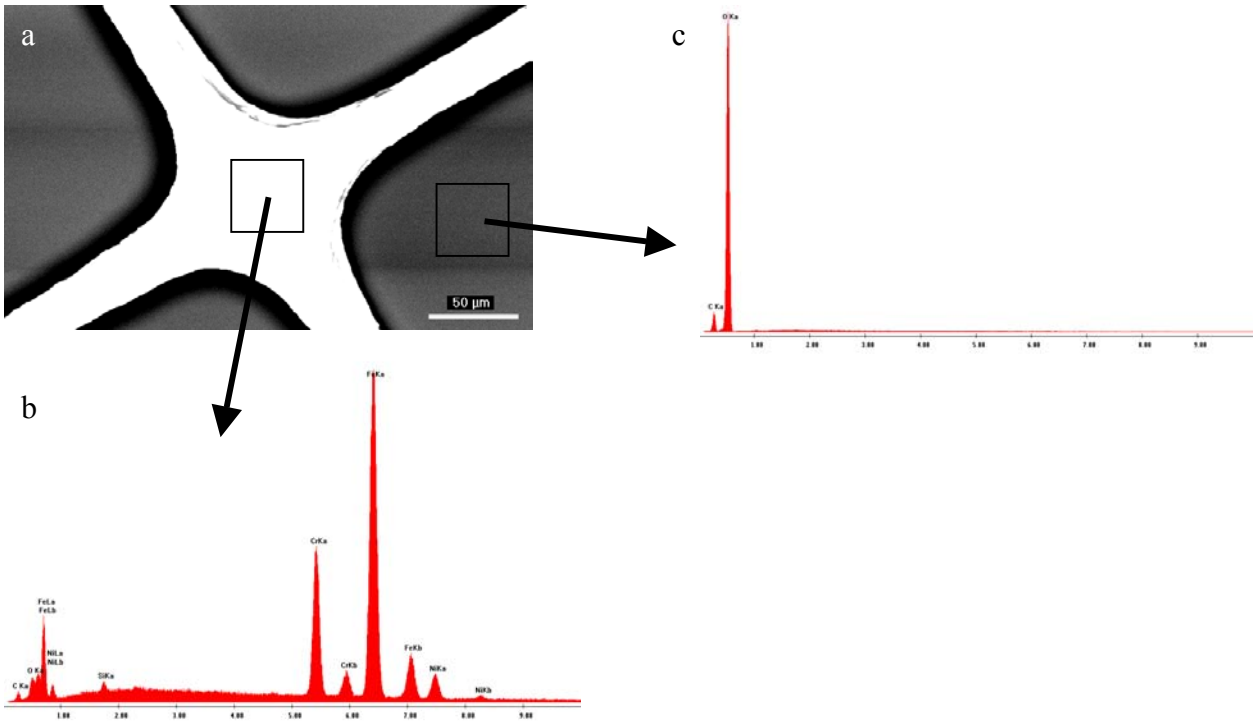

Fig. 1 SEM image and EDS spectra of QX capsule filled with water.

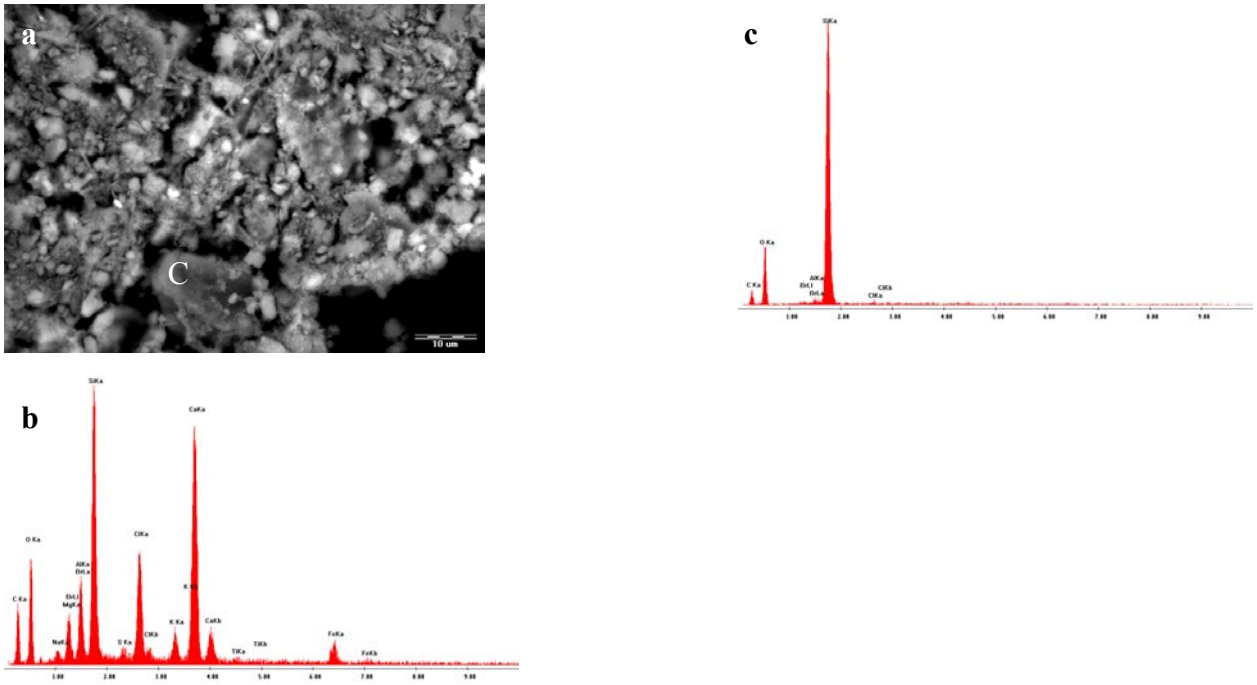

Fig. 2 SEM image (a) and EDS spectra of Dead Sea mud, full frame (b) and spot analysis (c).

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