ESRF	**Experiment title:** High resolution X-ray powder diffraction on exotic sepiolites.	**Experiment number**: 25 01 624
Beamline: BM25A	**Date of experiment**: from: 14-4-2006 to: 18-4-2006	**Date of report**: 29-5-2006
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NOTE THAT THIS REPORT IS THE SAME FOR EXPERIMENT 25-01-606 AND ITS CONTINUATION 25-01-624

Report:

Sepiolite is a fibrous clay minerals which differ from laminar clays by having channels in its structure. This is the result of the inversion of the tetrahedra layers every 8 octahedral positions (giving channels of 5.7 × 11 Å). These channels can hold zeolitic water and other molecules. Sepiolite is a trioctahedral phyllosilicate (all octahedral positions are occupied, in this case by Mg in the "ideal" sepiolite) and the theoretical formula is Si6Mg4O15(OH)2•6 H2O. Theoretical models of the crystallographic structure for sepiolite and palygorskite have been proposed. However, sepiolites vary depending on the origin, formation conditions, chemical environment, etc., therefore they can separate from the theoretical model and this is the first time that a systematic and comparative study on sepiolite is make.

Experimental method

High-resolution X-ray powder diffraction patterns were collected at beamline BM25A with a fixed wavelength about 15 keV (0.8 Å) at room temperature. Powdered samples were placed inside a 1 mm diameter capillary, which were rotated during exposure. Data were collected in a continuous 2 -scan mode from 3° to 40°. 23 different samples coming from most important deposits of the word were studied. They are well characterized by electron microscopy and other laboratory techniques, and the chemical formula is being obtained by Analytical Electron Microscopy using a Transmission Electron Microscope.
Samples were selected taking into account their high content in sepiolite, but small quantities of some impurities have been found in certain of them.

Two different types of sepiolite have been studied (if size of the fibre is considered):

a- macroscopic sepiolite: fibres of several milileters or centrimeters in long.
b- clay sepiolite *s.s.*: natural powdery samples.

Both group, macroscpic and *clay*-sepiolite, correspond to two different types of conditions of formation: hydrothermal and sedimentary ambiences respectively, and first objective is to know if differences in the diffractions patterns can be found for two group. Hydrothermal conditions imply higher temperature and more homogeneous compositions of the precursor solution than sedimentary conditions, and consequently *better* crystals (in composition and crystallinity) should be formed.

Results obtained

Diffractograms corresponding to the samples studied are similar, but differences in peak position and relative intensities have been found.

As can be seen in Figure 1 the width and position the main peak (110) are different. The width is related to crystallinity of the samples and ranges from the hydrothermal samples (the best crystallised) to the sample coming from Eskisehir (Turkey) which presents very broad diffraction bands.

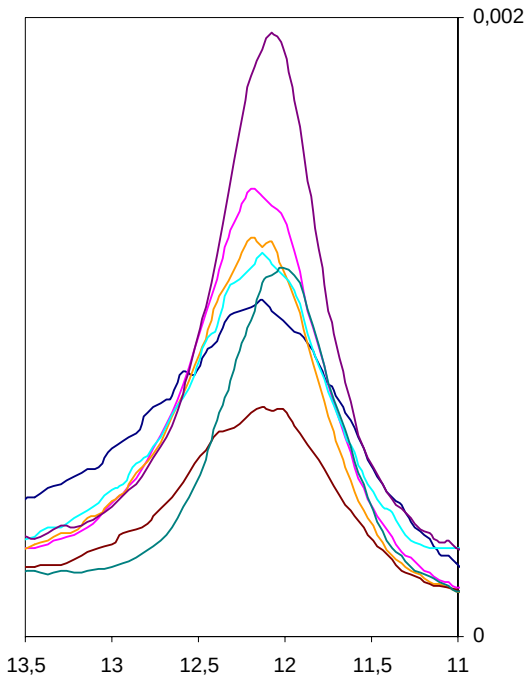

Figure 1: 110 peak of several samples studied in same conditions..

Position (d-sapacing) of 110 reflection is not related to origin (crystallinty) of the samples, as can be seen in figure 2 were hydrothermal samples are ploted in different colour. On the contrary, variations in d200 reflection correlates directly with the origin of the sepiolite and the higher d-spacing corresponds to macroscopic fibres (figure 3).

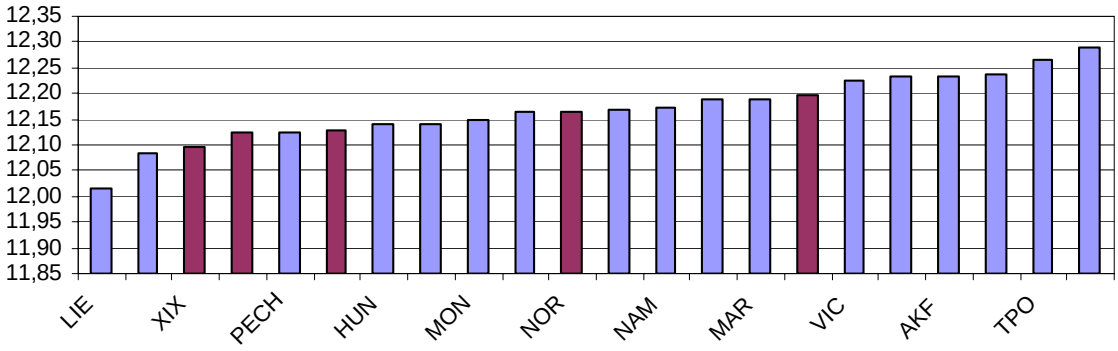

Figure 2: d 110 value. Blue colour correspond to “clay” samples and magenta to macroscopic ones.

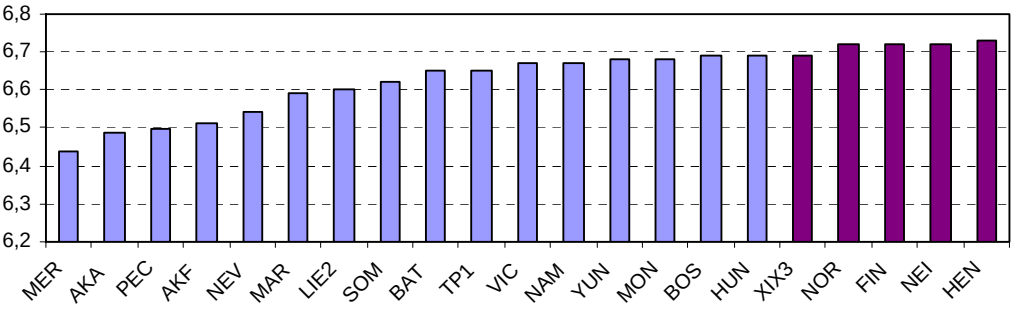

Figure 3: d 200 value. Blue colour corresponds to “clay” samples and magenta to macroscopic ones.

Differences related to crystallinity have been found as can be seem in figure 4, corresponding to 17-22 Å, the relative intensities of the diffractions peaks

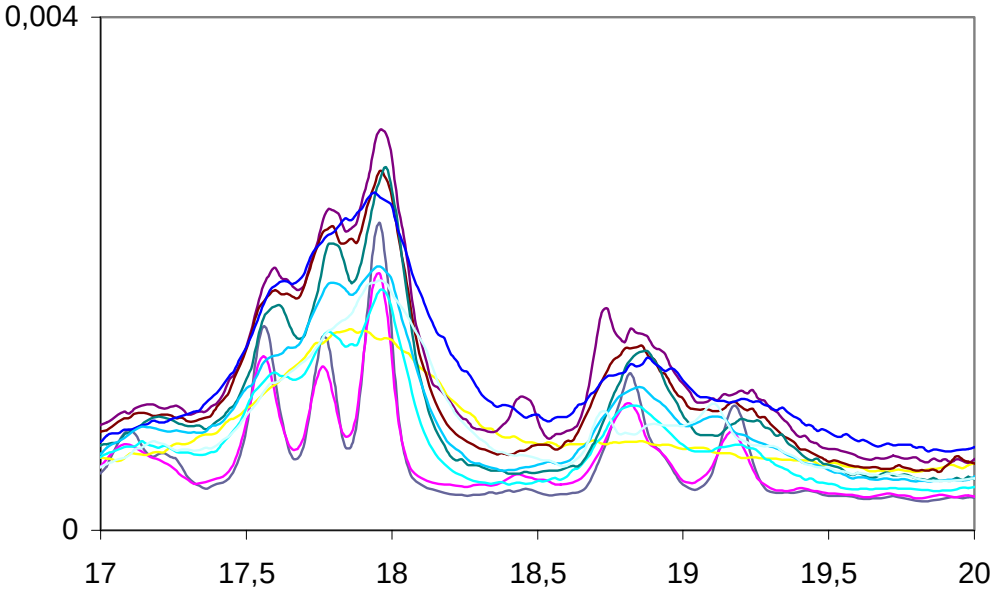

Figure 4: Different crystallinity of the measure samples is evident in the 17-20 deg. zone

In conclusion, we successfully measured high resolution XRD on a large collection of sepiolites. Work is in progress to relate the differences found in the diffractograms to the crystal structure.

We would like to thank the beamline team for its frienly welcome, disponibility and efficient assistance.

The beamline worked perfectly. We suggest that the Powder XRD station where me made the experiment would be equipped with more detectors (only one at the moment) to reduce significatively the measuring time and allow users to measure more samples and/or reduce the number of runs.