EUROPEAN SYNCHROTRON RADIATION FACILITY

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ESRF

Experiment Report Form

ESRF	Experiment title: In situ XANES analysis of the organic compounds that drive the mineralogical changes related to environmental conditions in larval shells of the oyster shells	**Experiment number**: EC545
Beamline: ID21	**Date of experiment**: from: 20/01/2010 to: 25/01/2010	**Date of report**: 11 feb 2010
Shifts: 15	**Local contact(s)**: Murielle Salomé	*Received at ESRF:*

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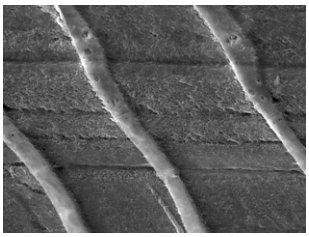
Report:

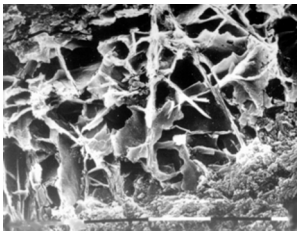
This proposal was a part of a research project (Calmaro, ITN) covering the microstructural, mineralogical and chemical changes along the growth of the oyster, associated with a molecular biological study of the mantle cells secreting the shell layers. Understanding the consequences of the early developmental stages of cultivated shells of commercial interest is a key-question for fisheries and environmental analyses related to ocean acidification and climatic changes.

Previous sessions dedicated to XANES maps of S aminoacids and SO4 acidic polysaccharides were present in mollusk shells. In mollusk shells composed of calcitic and aragonitic layers, S was more abundant in calcite. In both minerals, sulphated sugars were more abundant than S aminoacids related to proteins, but S aminoacids are more abundant in aragonitic layers than in calcitic layers. However, no shell composed of several calcitic layers, the structure of which differs, were studied.

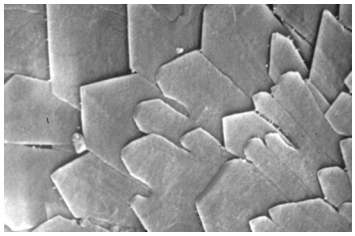
Oyster shells have two valves; one of the specificity of oyster shells is that the two valves comprise different structures. One valve comprised an outer thin prismatic layer, inter-bedded chalky zones and a inner foliated layer. The other valve has no prismatic layer. Thus both valves have to be analyzed. We have studied two sets of left and right valves, in juvenile and adult shells. Previous electron microprobe analyses have shown that the S content of the prismatic layer of *Crassostrea* is twice that of the foliated layer (unpublished data).



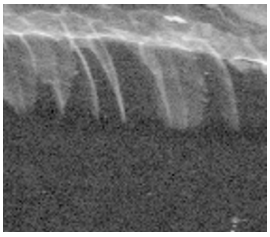
Prismatic layer (prism diameter ~15 µm)

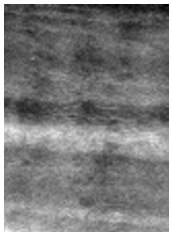


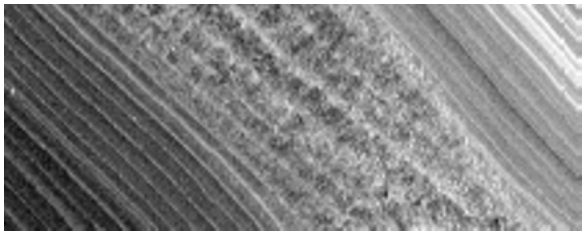
Chalky layer



Foliated layer







From left to right: prismatic and foliated layers, right valve, 4 weeks old (60x60 µm); detail of the foliated layer (40x55 µm); XANES maps of the sulphated sugars. Chalky layer in the foliated layer of the right valve of an adult shell, XANES map of organic sulphate.

In this first step of the expriments (farming oysters in various pCO2 sea waters), only shells growing in normal sea water have been used as reference.

Despite a common mineralogy: calcite, the diverse structural layers of the oysters have different sulfur contents: the calcitic prismatic layer has a higher S content than the foliated layer in the juvenile shells. The boundaries of the prisms are organic, and clearly visible on the maps. Growth lines are visible in the foliated layers. Despite the discontinuous structure of the chalky layers, growth zones are also present in organic sulphate maps. Limits and boundaries of the structural units and layers are stronger in the adult shells. The high sulfate content of the thin prismatic layer in adult shells confirms the quantitative results obtained with electron microprobe.

A detailed examination of Mg and P maps obtained with the multi-channel detector will be done in the next months to compare with microprobe quantitative data.

These results will be integrated in the PhD memoir of F. Bagusche (ITN program Calmaro).

Because of some problems at the Argenton station, we have not been able to use cultivated oyster shells in modified sea waters.

Thus, we have tested the ability of the ID21 to map S amino acids and sulfate in mollusk shells composed of two layers with different structures but with the same mineralogy: aragonite. As for the calcitic oyster shell, differences between layers are clearly visible in the aragonitic shells. Additional data will be acquired using a confocal microscope (LIONS, CEA) to try to correlate the organic matrix and organic sulphate.

Experiment at ID21 was done at the end of January 2010, so it is too soon for submitted our published papers related to this session.