Characterization of Microfabric and Mineralogy of Marine Sediments by Electron Microscopy

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Marine sediments were examined by transmission and scanning electron microscopy in order to determine their mineralogy, microfabric, and pore geometry. These parameters affect fluid flow through marine sediments, consequently the migration and accumulation of economic resources. Because this study focused on samples in their natural condition, sample preparation was critical to achieve good results.

The samples investigated are marine chalks with various degree of cementation from the the northwestern margin of the Demerara Rise (offshore French Guyana) and marine clays from the northwestern Gulf of Mexico continental slope.

We developed a new method for sample preparation that takes advantage of cold microwave technology for dehydration and impregnation, to preserve the natural microfabric, achieve stability under the electron beam and allow polishing of sample surface. Samples were dehydrated by water substitution with methanol, conducted in steps and aided by microwaving at low temperature. Resin infiltration was also conducted in steps, with the use of propilene oxide as transient fluid. Microwaving was adopted to facilitate resin infiltration. The basic steps are based on previously published methods [1-2-3] of sediment sample preparation, but we took advantage of cold microwave technology to complete substitutions in a shorter time, and infiltrate low-permeability samples with an epoxy resin. The use of epoxy resin, rather than LRWhite [2-3] gives the samples stability under the electron beam for extended periods of time and good polishing characteristics.

Observation by TEM shows very good quality samples, with the resin filling the space between particles (Fig1-2-3). The chalk samples showed various mineralogy with carbonates, mostly fossilferous in origin, but also abundant clay minerals (Fig.1-2). The TEM was very useful to detect clay minerals that were missed from preliminary observations or analyses. Observation by SEM shows, that at a larger scale, oozes and chalks from the Demerara Rise are composed of a complex mixture of whole and broken coccolith spheres, micrite crystals, with forams (Fig.5). Most interestingly, large pores are present in these sediments that are completely filled by opal lepispheres and clinoptilolite (Fig.4). The mineralogy of the Gulf of Mexico clays is more homogeneous, with prevalent clay minerals, mostly smectite and illite. As their mineralogy indicates, Gulf of Mexico clays have extremely low permeability, nonetheless, we obtained good infiltration of resin and high quality samples (Fig. 3). An example is given in Fig. 3, where a particle of illite shows two sets of lattice fringes, one at 10 Å and the other at 3.3 (Fig. 3).

References

[1] R.H. Bennett et al., J. of Sed. Petr. 51 (1981) 217.

[2] R.T. Giberson and R.S. Demaree J, Microwave Techniques and Protocols, Humana Press Inc., Totowa, 2001

[3] J.W. Kim et al., Clays and Clay Min. 43 (1995) 51.



Fig. 1. The distribution of carbonates and Al-silicate minerals in a TEM micrograph (left), is clearly shown by EDS maps of Ca, Si and Al.



Fig. 2. Calcite crystal with correpsonding diffraction pattern (bottom left)

Fig. 3. Illite particle sectioned across the 001 plane. The magnified area (A) shows two sets of lattice fringes that also cause two peaks i n the associated Fourier Transform (bottom image) corresponding to spacing of 10 Å, typical of illite, and spacing of 3.3 Å. The higher order maximum is accompanied by a less intense reflection off-axis (at 3.47 Å) indicative for a planar rotation .





Fig. 4. Pore conduit in sediment filled by opal and clinoptilolite (top left) as shown by SEM. The bottom micrograph shows the morphology of opal lepisheres in great details, while the right micrograph shows the association and intergrowth of clinoptilolite and opal.



Fig. 5. BSE map of a polished sample of marine sediment, impregnated with resin showing pore and grain size distribution as well as variations in composition.