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Characterization of inorganic-organic cement materials

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For environmental issues and energy consumption diminution, building materials with high performances are presently widely studied. These materials can be described as organo-mineral composites with a complex texture of the organic/inorganic hydrated co-matrix. As final material properties are governed by this complex structure, a characterisation of the geometrical features and the texture of each phases could help in formulating new durable construction materials. Furthermore these materials evolve in time, for example by hydratation processes leading to the formation of hydrates or by a filmfication of latex. It is thus necessary to characterize the evolution of the materials during time by a characterization of the fresh state and solid state.

To fully study their microstructure, a multiscale characterization (from mm to nm) is required. Several tools of electron microscopy can be envisaged for the analysis at these different scales: scanning electron microscope (SEM), environmental scanning electron microscope (ESEM), transmission electron microscope (TEM). Furthermore a 3D characterization can now be envisaged with the development of the 3D tomography in the Focused Ion Beam (FIB) microscope. Nevertheless, protocols have to be carefully defined so that the microstructural observation of the material may be feasible without hindering the material microstructure.

Different cements were characterized in this study and the latex used has been varied in terms of size and stabilization. A characterization at different scale reveals differences in the microstructure of the samples. SEM images showed that the spatial organization of hydrates can vary depending on the latex used: hydrates can intermingle and form clusters of flattened beads (mean diameter: 3 microns) (confirmed by TEM observation), while in other samples they were distributed in distinct zones (Figure 1). TEM characterization was also very useful to discriminate the C-S-H hydrates formed: foil-like (figure 2a), tubular structure (figure 2b) typical of the outer-product, fibrillar structure of the inner product and some thin sheets (figure 2d).

The advantage of FIB characterization is the possibility to have a 3D characterization of the sample. It was used to study the organization of the latex around the mineral grains. In the case of small latex particles, the filmfication is not completed and some of particles are well distributed around amorphous grains (Figure 3a). In the case of big latex particles, their presence can promote the presence of nanoporosity in the sample. This was observed by FIB 3D reconstruction (Figure 3b).

The coupling of SEM, TEM and FIB characterizations is of much interest to fully characterize the complex structure of cement. FIB technique and 3D reconstruction will allow to go further on the characterization and is very complimentary with SEM and TEM characterizations [1].

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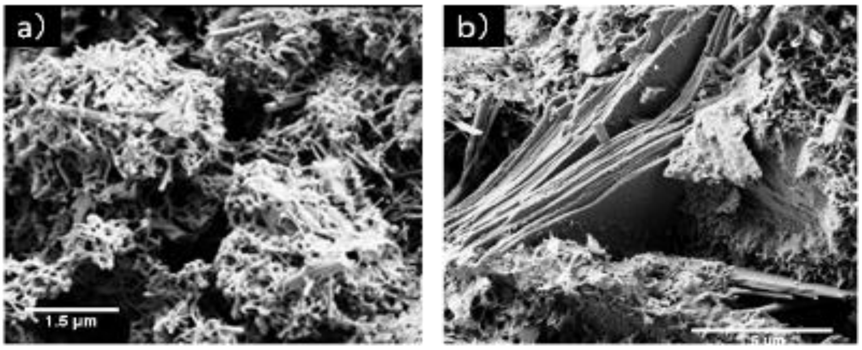

Figure1. SEM images showing two hydrates structures depending on the latex used: a) beads of hydrates, b) hydrates distributed around portlandite crystals (scale 1.5µm)

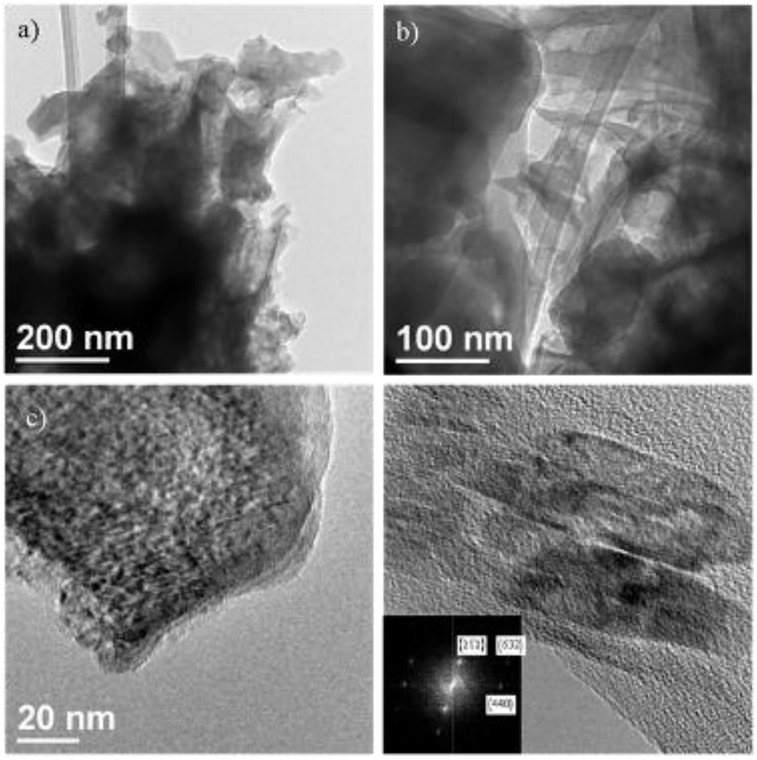

Figure 2. TEM characterization of the different morphologies of C-S-H :a): foil-like, b) tubular structure, c) fibrillar structure and d) thin sheets

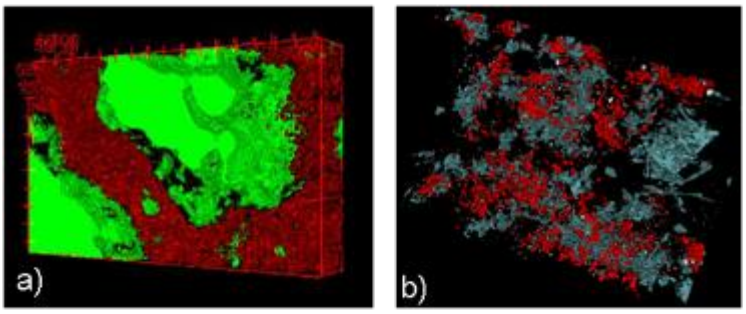

Figure 3. a) distribution of the latex particles around the mineral grains ; **b)** distribution of porosities due to the presence of latex (red) and hydrates (b)