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Characterization of organo-mineral cement materials

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Key words : 3D Microstructural characterization, FIB, MET

CONTEXT - MATERIAL

Whatever the applications biomedical, transportation, energy or construction; there is a lack of knowledge on the true organization at the nano scale of mineral and organic phase (qualitative image, together with quantitative analysis). This study was focused on the characterization of cementitious materials, and mainly on organo-mineral composite. Many innovative applications in the field of high-performance construction materials and building insulation systems are under development. The addition of an organic phase in the form of a latex suspension may improve mortar functional properties (rheology, substrate adhesion, hygro-thermal). The mechanical properties are also upgraded, and in particular the crack resistance. The latex addition effect should be characterized at different scales and therefore require the use of different electron microscope techniques.

Material

Several model formulations (Table 1) were elaborated to provide a better understanding (i) of polymer filmification modified by unhydrated cement, (ii) of hydrates formation modified by polymer.

	f_v latex	F_w latex	Latex (dry g)	Super-plasticizer (g)	Cement (g)	Water (g)	Water /Cement
Latex-cement	0.22	10% /cement weight	3.00	0.1 – 1.5 % to ensure gravity casting	30.00	8.03	0.30

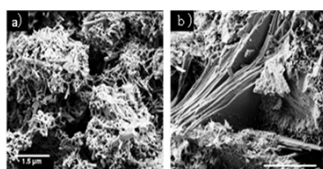
The three raw latex materials differed either by their size or their surfactant :

- a 210 nm styrene butyl acrylate SBA (BASF model dispersion 4) including a charged stabilizer (3%wt PMMA)
- a 1500nm vinyl ethyl acetate VEA (Wacker 2434) including a charged stabilizer (7+1% PVOH-PAA)
- a 1500nm vinyl ethyl acetate VEA (Wacker 2435) including a steric stabilizer (7% PVOH)

The cement was a white cement (Lafarge, CEM I 52.5R Le tel)

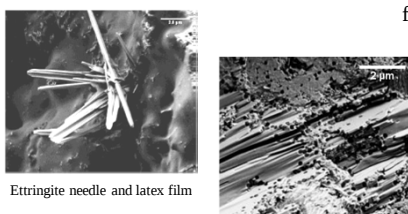
SEM and ESEM characterizations

SEM: spatial organization of hydrates



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

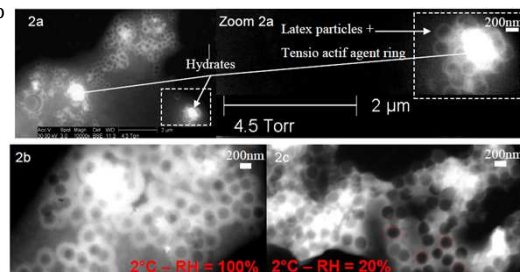
Characterization of latex film and particles



Ettringite needle and latex film

Portlandite crystals and latex particles

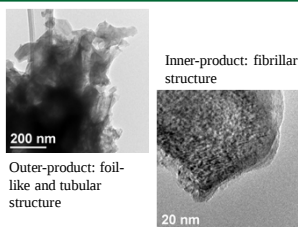
ESEM: follow up of the latex filmification



latex particles were nucleated around nanometric hydrates (bright zones)

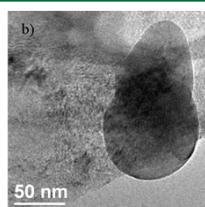
TEM, latex and hydrate nanostructures – compound identification

TEM: characterization of hydrates morphologies

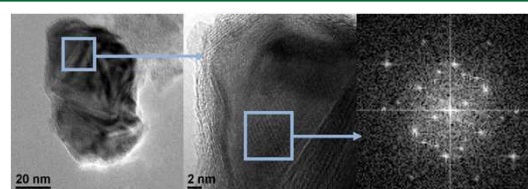


Outer-product: foil-like and tubular structure

characterization of polymer: morphology and latex/cement interactions



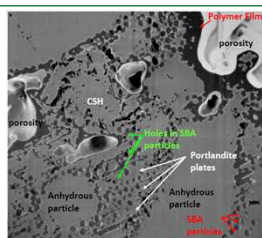
Latex nanoparticle on inner product



Hydrate contained in a composite cement/latex material (VEA_PAA+PVOH); cubic hydrate katoite is observed

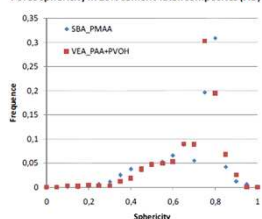
3D arrangement of Hydrates Void and Latex

FIB: characterization of hydrates and nanoporosities

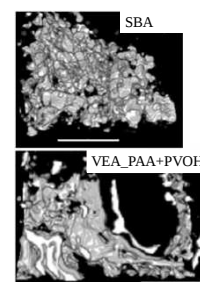


- Polymer on the surface of the anhydrous grains
- Latex filmified or as particles
- Two types of nano-porosities : due to C-S-H hydrates or due to the latex
- Oriented Portlandite plates

Pores sphericity in 10% cement-latex composites (FIB)



Frequency distribution of the nanoporosity 3D representation due to the latex (red) and the CSH (blue)



Conclusion: A multi-scale characterization of building material is essential to optimize their properties and was successfully performed by coupling different electron microscopy techniques. Hydrates morphologies, effect of the addition of latex on the cement microstructure were characterized. Full scale analysis is still under completion

Acknowledgment

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