Quantification and analysis of amorphous components (in cements) by synchrotron ptychographic X-ray tomography

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Outline

1. **The relevant problem.**
2. **Concrete:** length scales (our everyday problem)
3. **Background:** Previous PXCT studies of Portland cement binders
4. **Case-1:** Study of a new binder with much lower CO_2 footprint
5. **Case-2:** Study of Portland including Supplementary Cementitious Materials
6. **“Christmas Wishes”**

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1. Problem #1: CO_2 emissions

Portland cement (PC) world production is ~4 Gt/yr, 4.6 Gt in 2015.
PC production is expected ~4–8 Gt/yr by 2100

On average, for every ton of type-I PC, ~0.95 CO_2 t are released, from (i) limestone decomposition, (ii) burning fuel, and (iii) electricity consumption for grinding.

This translates into ~7% of the total anthropogenic CO_2 emissions, 3.5 Gt/yr.

1 Gt = 10^{15} g = 1 Pg

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1. Problem #2: Construction & Demolition Wastes

- The concrete use is today **~18 Gt/yr**.
- The estimated world concrete stock is **315 Gt** which results in 0.3 Gt/yr of CDW.
- The newest model predicts a skyrocket increase of CDW to **20–40 Gt/yr by 2100**,

This could not be processed as aggregates, within concrete, as it will be more than two times the predicted need.

→ **Cements with lower CO_2 footprint and more durable/sustainable.**

Todays expected service live of buildings and infrastructures: <100 years!

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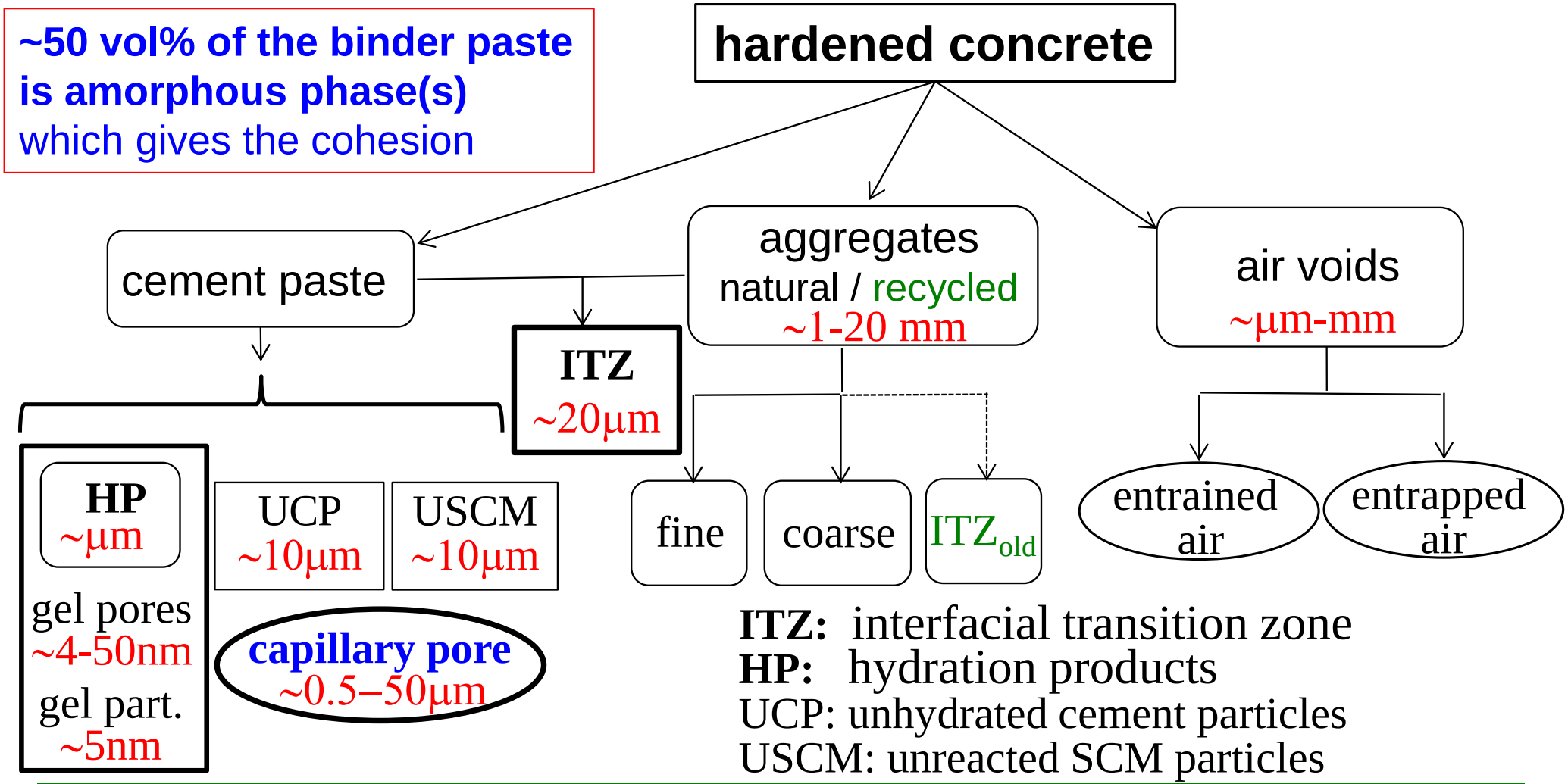
2. Concrete : length scales - heterogeneity

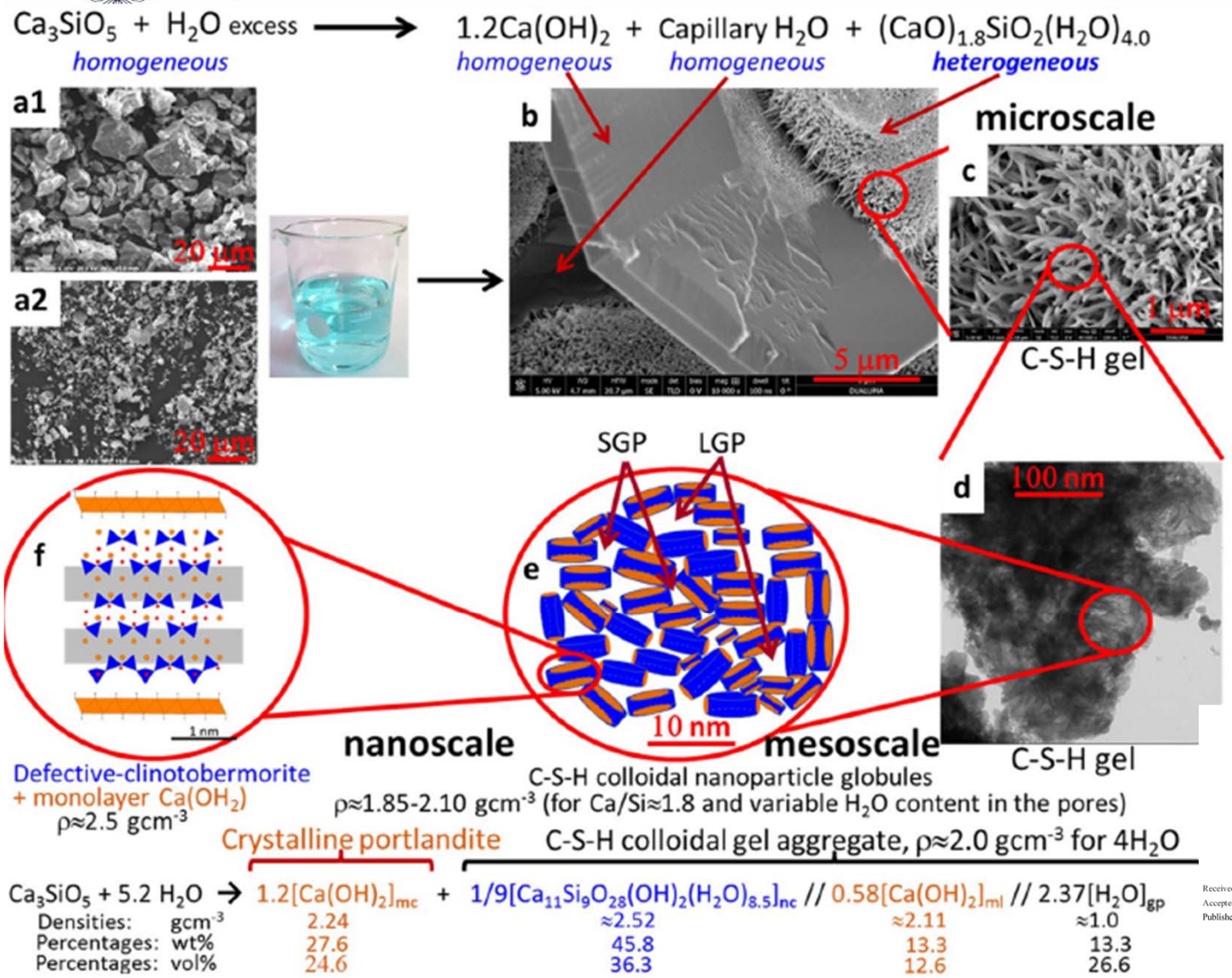
Concrete is made from Portland cement, water, aggregates, additions & admixtures

~50 vol% of the binder paste is amorphous phase(s) which gives the cohesion



ITZ: interfacial transition zone
HP: hydration products
UCP: unhydrated cement particles
USCM: unreacted SCM particles

2. Concrete : length scales – alite hydration



SCIENTIFIC REPORTS

OPEN

Multiscale understanding of tricalcium silicate hydration reactions

Ana Cuesta[1,2], Jesus D. Zea-Garcia[2], Diana Londono-Zuluaga[2], Angeles G. De la Torre[2], Isabel Santacruz[2], Oriol Vallcorba[1], Monica Dapiaggi[3], Susana G. Sanfélix[2,4] & Miguel A. G. Aranda[1]

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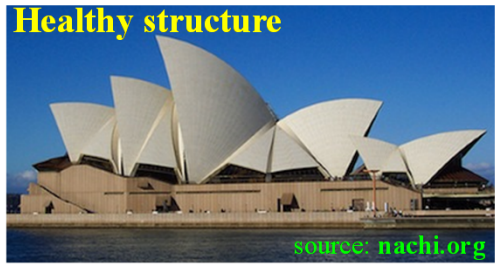
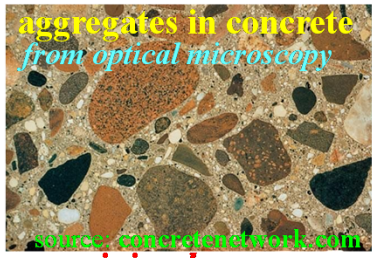
Published online: 04 June 2018

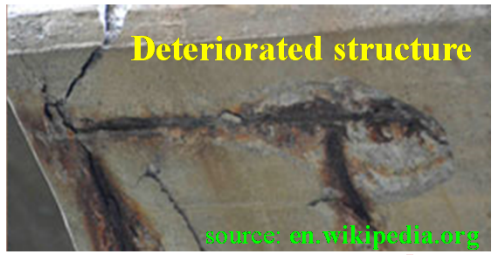
Sci. Rep. **2018**, 8, 8544

2. Concrete : length scales – multiscale / multimodal





miniscale: ~mm



macroscale: ~m

fibers

minicracks

air porosity

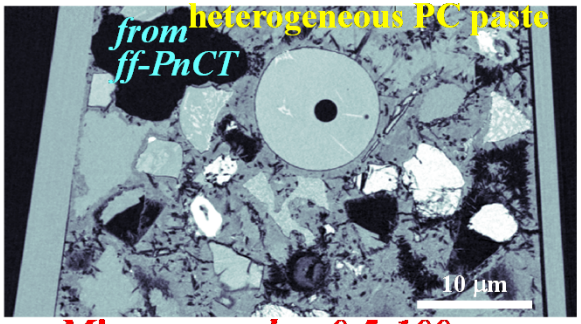
ITZ porosity

microcracks

Solid past component 3D arrangement

water capillary porosity

gel porosity: SGP & LGP



Microcroscale: 0.5-100 μm

Crystalline phases

Amorphous component C-S-H gel

ettringite

portlandite

1.1 nm

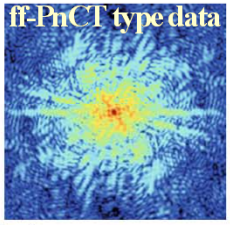
0.7 nm

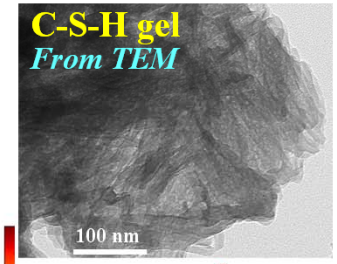
1 nm

Defective-tobermorite & monolayers of $Ca(OH)_2$

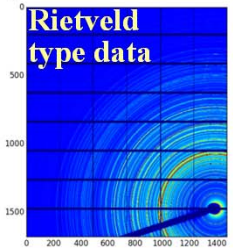
from PDF

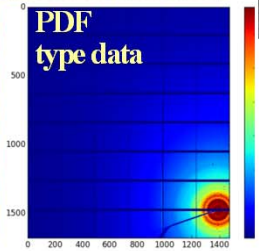
nanoscale: 0.5-10 nm



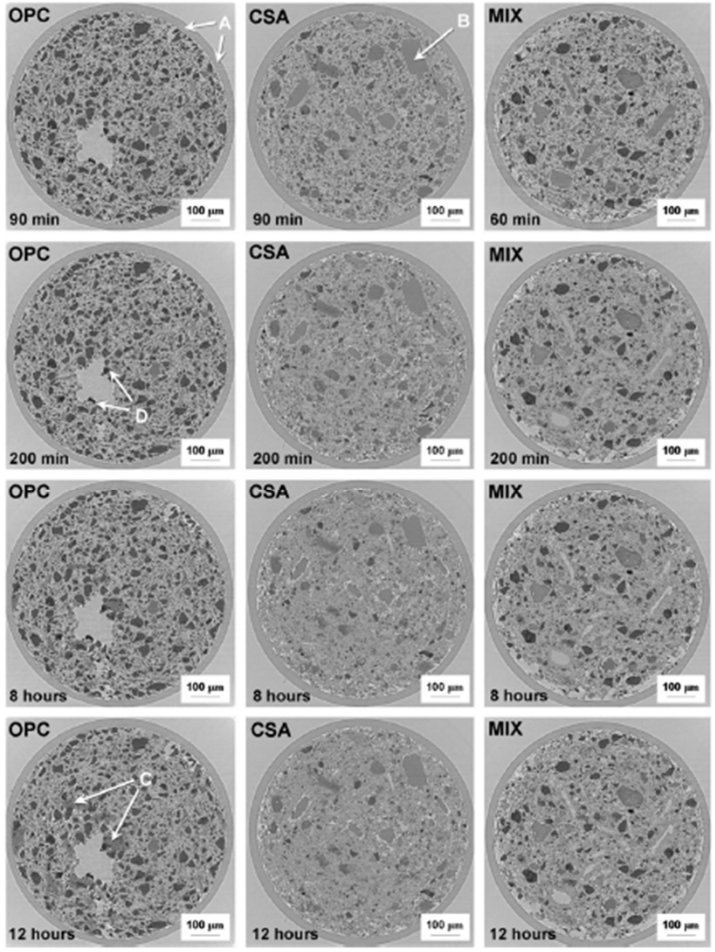


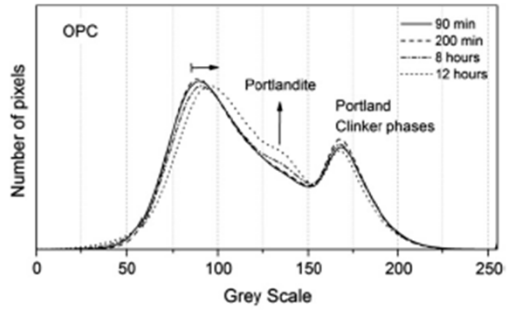
mesoscale: 10-500 nm

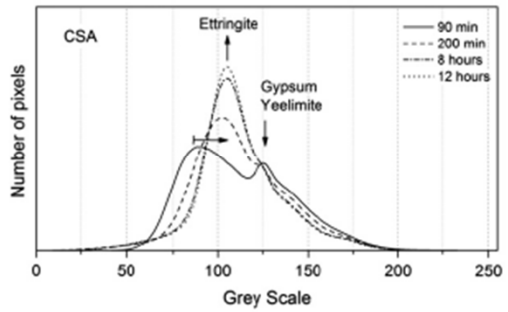


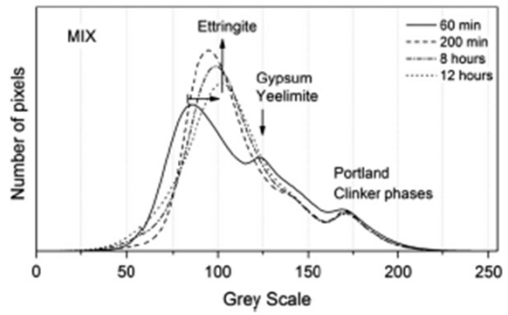


2. Concrete : length scales - microtomography









Gastaldi D, et al. “In situ tomographic investigation on the early hydration behaviors of cementing systems.” *Const Build Mater.* **2012**, 29, 284–290.

Standard μ-tomo (TOMCAT): Larger field of view (ϕ=0.7mm) and fast tomo

BUT **Poor contrast/information & poor spatial resolution**

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3. Background: Previous PXCT studies of PC binders

Density mapping of hardened cement paste using ptychographic X-ray computed tomography

Pavel Trtik [a,*], Ana Diaz [b], Manuel Guizar-Sicairos [b], Andreas Menzel [b], Oliver Bunk [b]

Cement & Concrete Composites 36 (2013) 71–77

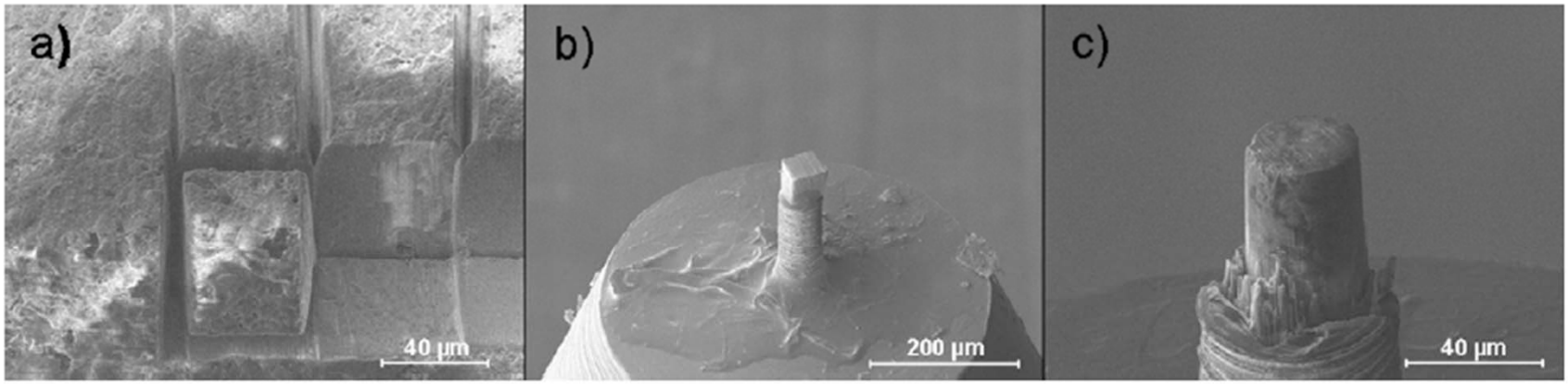
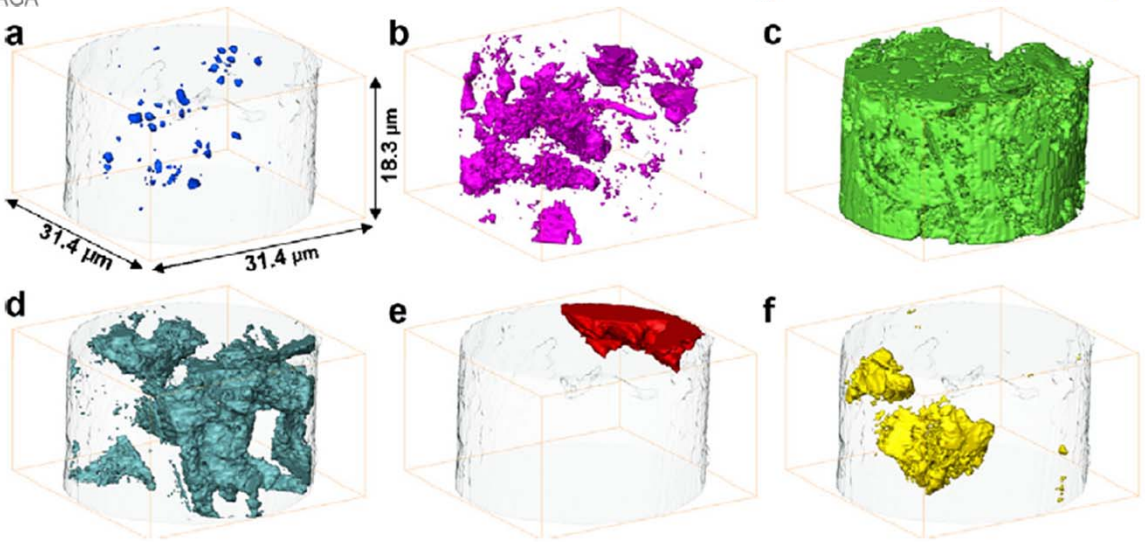


Fig. 2. (a) A view of the 90° sharp edge of epoxy-impregnated hardened cement paste from which a cube-like object was machined using focussed ion beam (FIB) milling. One of the objects is visible in about the centre of the image with the recesses remaining on the right after other similar objects were extracted. (b) The cube-like object positioned on the top of a stainless steel sample holder and (c) the final specimen produced by micromachining of the object in Fig. 2b into cylindrical shape using FIB milling.

3. Background: Previous PXCT studies of PC binders

Cement & Concrete Composites 36 (2013) 71–77



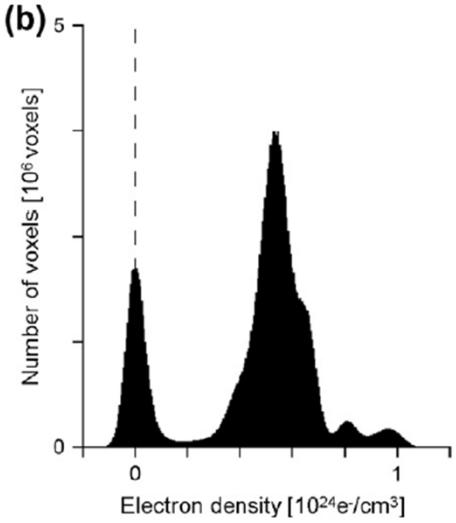


Fig. 5. Surface renderings showing the identified individual material phases. (a) Unimpregnated/'partially impregnated' porosity, (b) epoxy-impregnated porosity, (c) other hydrates (predominantly epoxy-impregnated calcium–silicate–hydrates), (d) calcium hydroxide, (e) calcium carbonate, (f) unhydrated/'partially hydrated' clinker residues.

Table 1

Measured electron densities, estimated chemical composition, and estimated mass density of the identified material phases. The standard deviation of the electron density measurement is based on the binned dataset for the entire regions of the identified phases (as shown in Fig. 5). The value *X* represents the unknown and likely spatially variable ratio between epoxy resin and nanoscale C–S–H particles that may theoretically assume values between 0 and 1, though a value based on a realistic particle packing (see Ref. [27]) is expected.

Material phase	Measured electron density ($10^{23}e^-/cm^3$)	Estimated chemical composition	Estimated mass density (g/cm^3)
Unimpregnated/partially impregnated porosity	0.67 ± 0.22	N/A	N/A
Epoxy-resin impregnated porosity	3.70 ± 0.32	$C_{86}H_{126}O_{18}Cl_3N_2$	1.14 ± 0.10
Other hydrates (predominantly epoxy-resin impregnated calcium–silicate–hydrates)	5.27 ± 0.36	$(1-X)(C_{86}H_{126}O_{18}Cl_3N_2)+X(CaO)_{1.7}(SiO_2)(H_2O)_{1.8}$	(1.63 + 0.09 X) + (0.11 + 0.01 X)
Calcium hydroxide	6.56 ± 0.18	$Ca(OH)_2$	2.12 ± 0.06
Calcium carbonate	8.04 ± 0.20	$CaCO_3$	2.67 ± 0.07
Unhydrated/partially hydrated clinker residues	9.65 ± 0.31	Ca_2SiO_4	3.21 ± 0.11

3. Background: Previous PXCT studies of PC binders

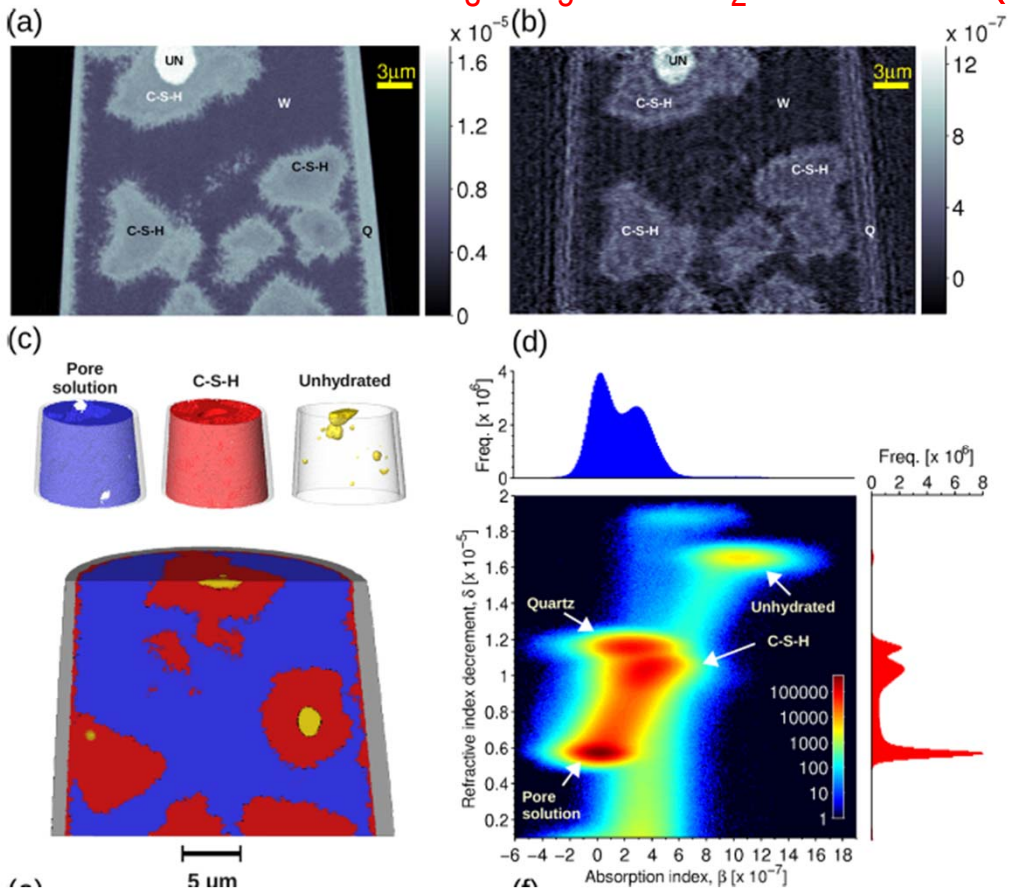
Langmuir Letter
pubs.acs.org/Langmuir

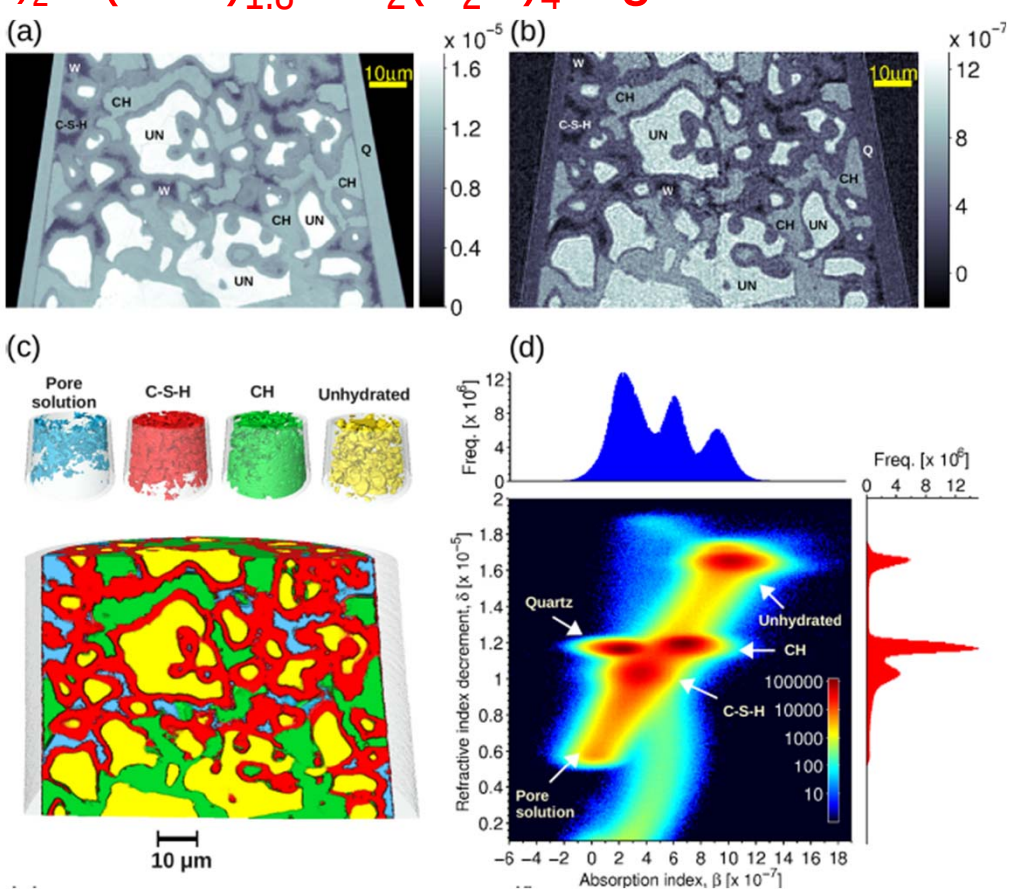
Mass Density and Water Content of Saturated Never-Dried Calcium Silicate Hydrates

Julio C. da Silva,*[,†,∥] Pavel Trtik,[†,‡,§] Ana Diaz,[†] Mirko Holler,[†] Manuel Guizar-Sicairos,[†] Jörg Raabe,[†] Oliver Bunk,[†] and Andreas Menzel*[,†]

Langmuir 2015, 31, 3779−3783

$$Ca_3SiO_5 + 5.2\ H_2O \rightarrow 1.2\ Ca(OH)_2 + (CaO)_{1.8}SiO_2(H_2O)_4 - gel$$





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3. Background: Previous PXCT studies of PC binders

Langmuir 2015, 31, 3779−3783

Table 1. Average Mass Density Values and Composition of the C−S−H in the Fully and Partially Hydrated Particles, Where y Represents the Estimated Number of Moles of Water in the Stoichiometry of C−S−H, Which Is Written As $C_{1.75}SH_y$

C−S−H	mass density [g·cm^{-3}]	relative mass of C	relative mass of S	relative mass of H	number of moles of water (y)
fully hydrated particles in volume A	1.726 ± 0.006[a]	0.334 ± 0.009[a]	0.204 ± 0.006[a]	0.462 ± 0.014[a]	7.5 ± 0.4[a]
	1.909 ± 0.005[b]	0.420 ± 0.009[b]	0.257 ± 0.006[b]	0.324 ± 0.015[b]	4.2 ± 0.3[b]
partially hydrated particles in volume A	1.860 ± 0.004[a]	0.383 ± 0.007[a]	0.235 ± 0.004[a]	0.382 ± 0.011[a]	5.4 ± 0.3[a]
	1.96 ± 0.04[b]	0.387 ± 0.011[b]	0.237 ± 0.007[b]	0.376 ± 0.018[b]	5.3 ± 0.4[b]
partially hydrated particles in volume B	1.828 ± 0.005	0.389 ± 0.010	0.238 ± 0.006	0.373 ± 0.015	5.2 ± 0.4

[a]Apparent inner-product C−S−H. [b]Apparent outer-product C−S−H.

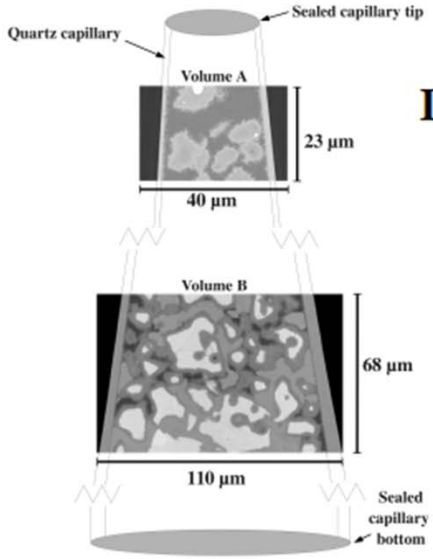

Figure S1. Scheme showing two volumes of the hydrated cement paste in a quartz capillary measured by PXCT are shown. The top and bottom ends were sealed and dimensions of each region are indicated.

Determination of the average water content and composition of the C-S-H

1. For stoichiometric (crystalline) compounds,
from the electron densities is possible to (accurately) determine the mass density

2. For amorphous compounds,
from the electron densities and the absorption values it is possible to determine the mass density with some knowledge of the stoichiometries

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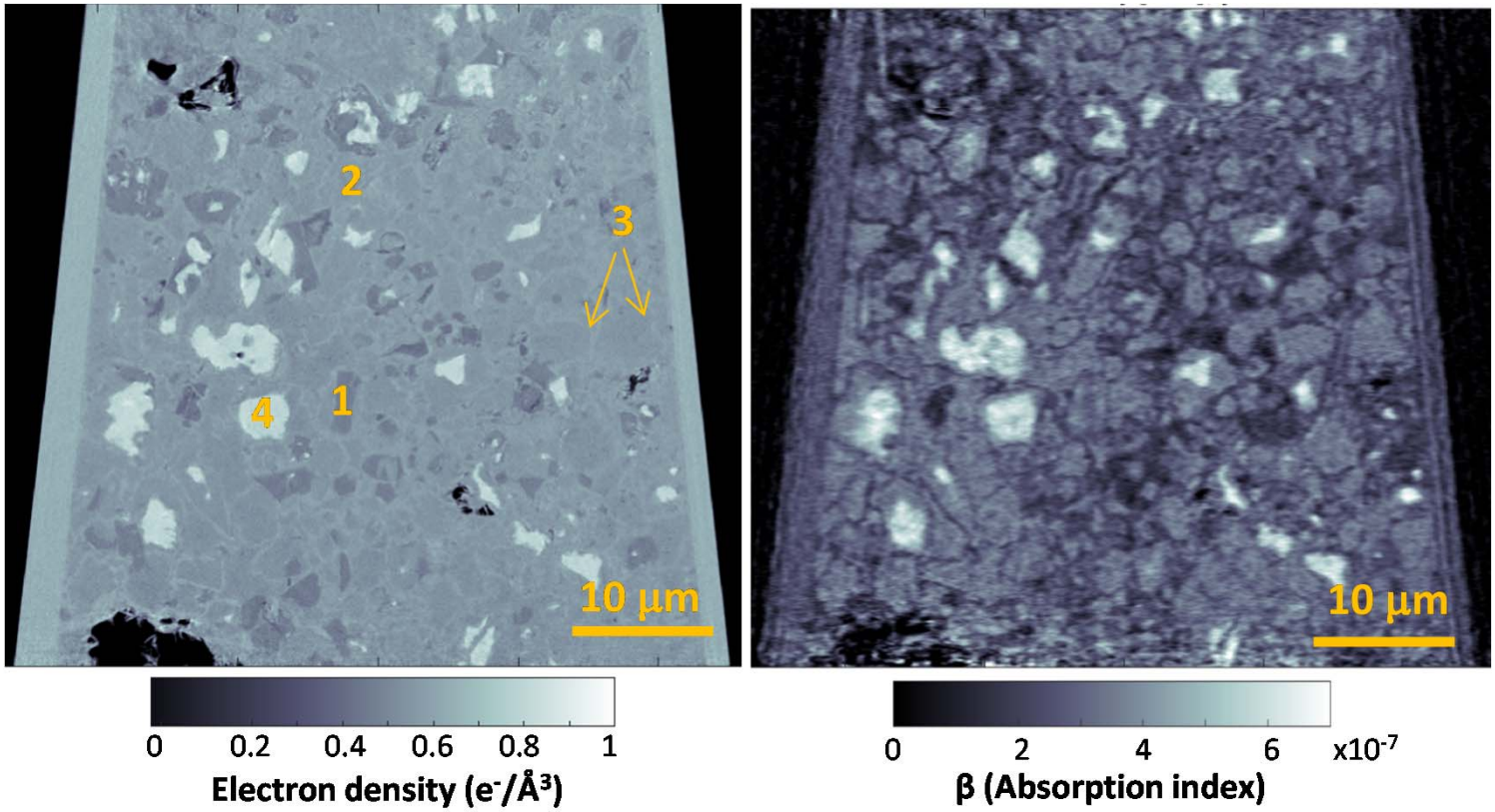
4. Case-1: Study of a new eco-cements, under development



Ye'elimite
$Ca_4Al_6SO_{16}$

Ye'elimite paste at 6 d of hydration, no additional sulfate source.
Air porosity: 0.3 vol%; AFt: 20.0 vol %(**1**); AFm: 36.9 vol% (**2**); **A-H gel: 38.6 vol% (3);**
ye'elimite: 4.1 vol% (**4**); w/c mass ratio ≈0.45

Cuesta A, et al. ""Chemistry and Mass Density of Aluminum Hydroxide Gel in Eco-Cements PXCT"
Journal Physical Chemistry C, **2017**, 121, 3044–3054. Data taken @ cSAXS with OMNY

4. Case-1: Study of a new eco-cements, under development

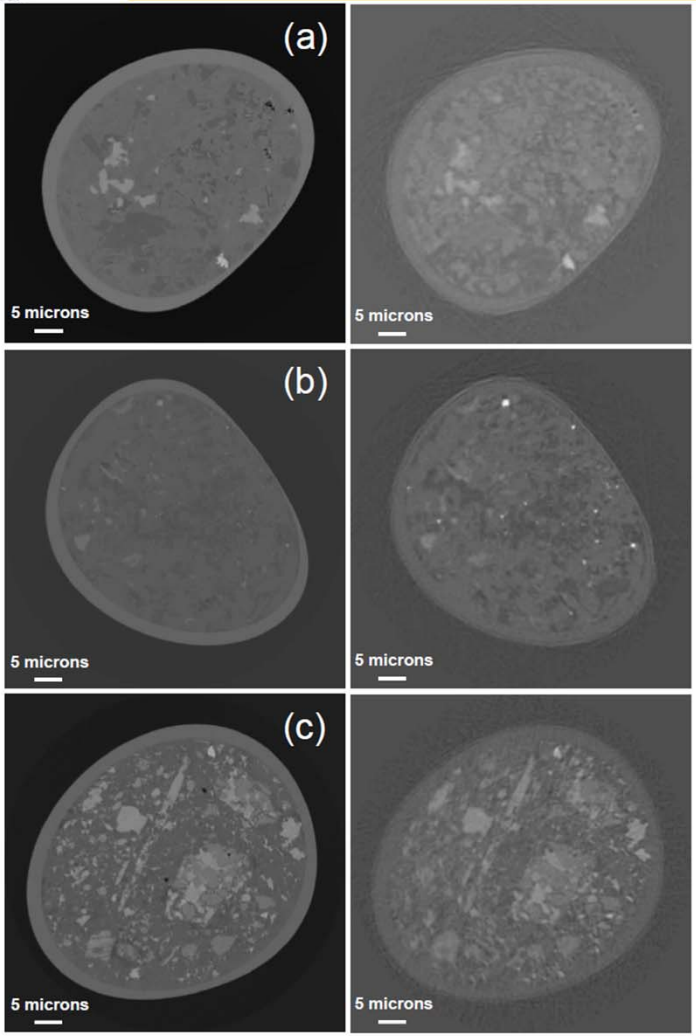


Figure S3. Selected horizontal slices of (left) $n_e(\boldsymbol{r})$ [electron density] and (right) $\beta(\boldsymbol{r})$ [absorption inde: tomograms for (a) ye'elimite paste at 8 days of hydration; (b) ye'elimite-gypsum paste at 18 days hydration; (c) CSA paste at 22 days of hydration.

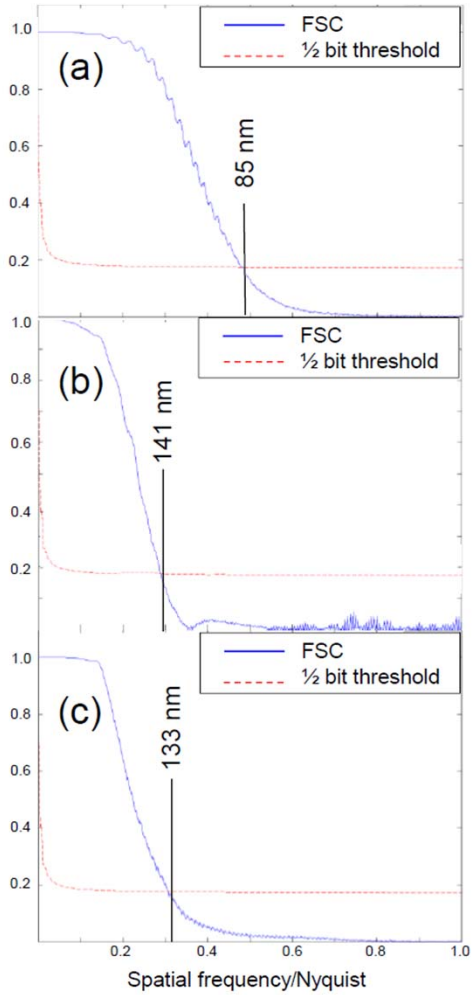
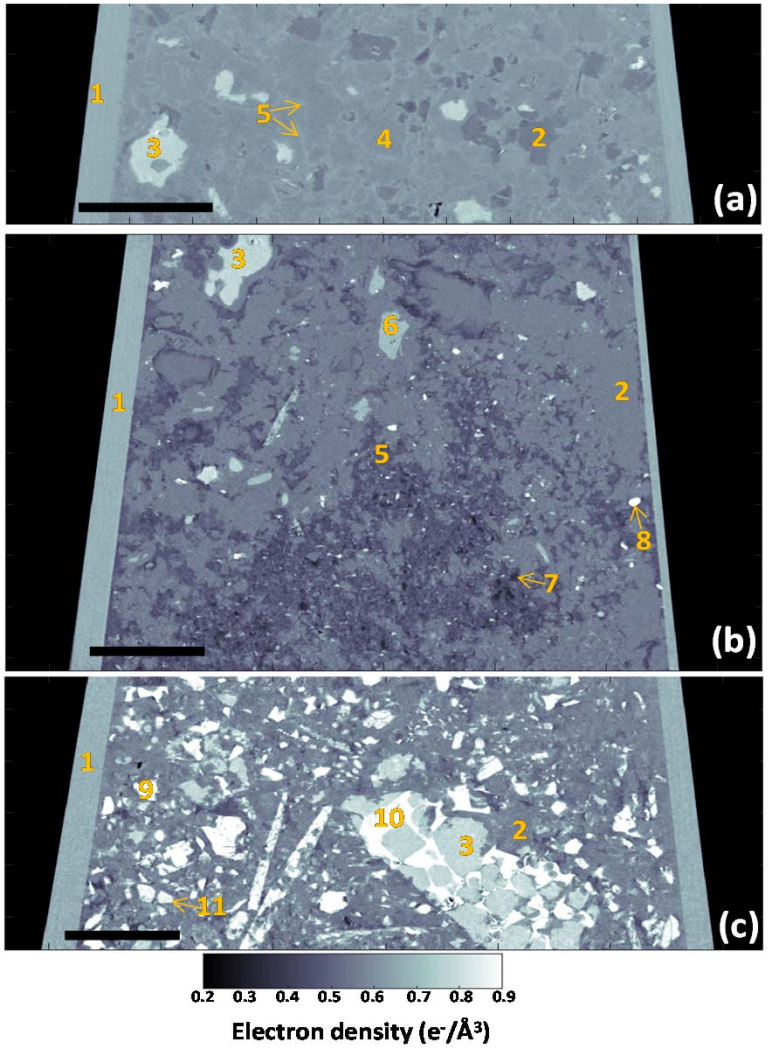
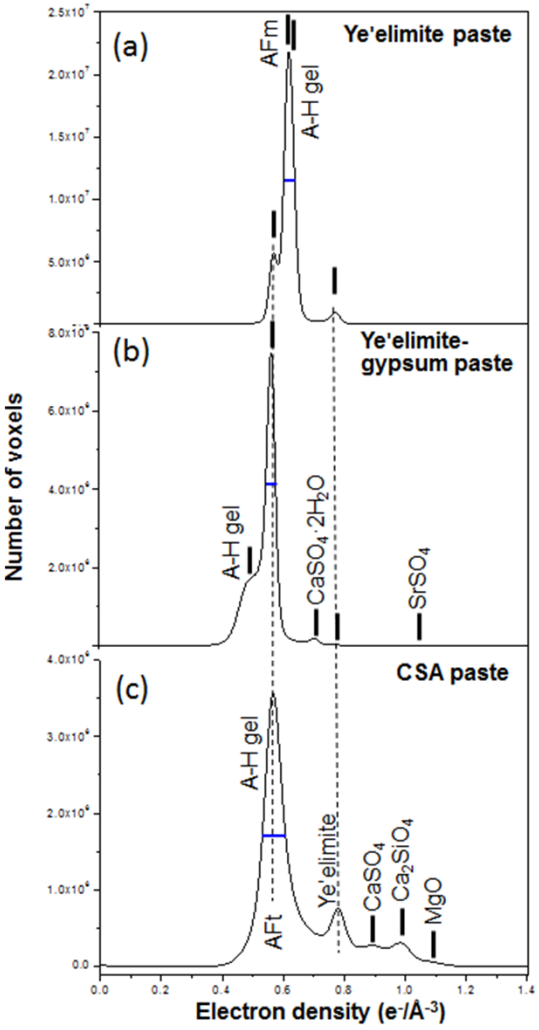
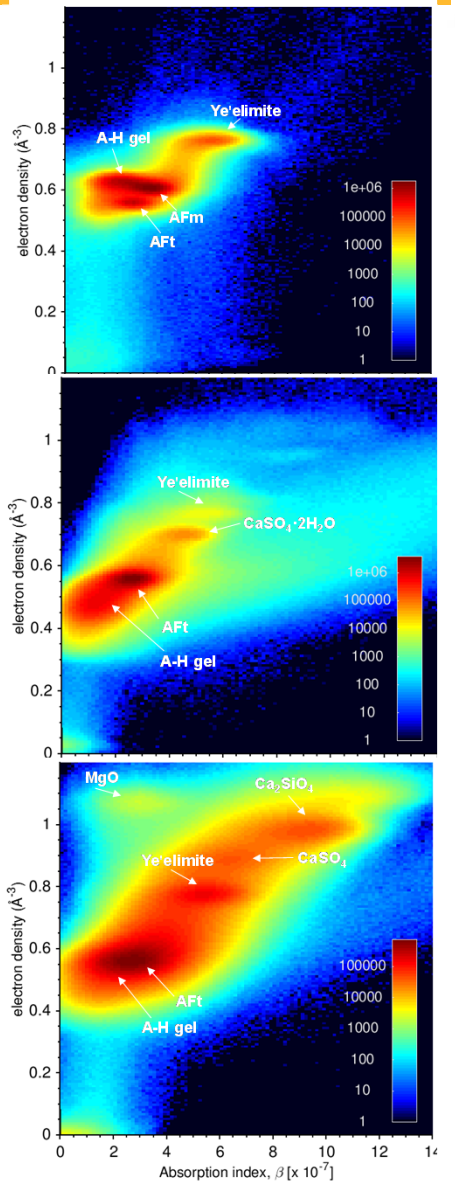


Figure S4. Fourier Shell Correlation plots from $n_e(\boldsymbol{r})$ tomograms for (a) ye'elimite paste at 8 days of hydration; (b) ye'elimite-gypsum paste at 18 days of hydration; (c) CSA paste at 22 days of hydration.

4. Case-1: Study of a new eco-cements







PXCT vertical slices of the electron density tomograms for (a) ye'elimite paste at 8d of hydration; (b) ye'elimite-gypsum paste at 18 d; (c) CSA paste at 21 d.

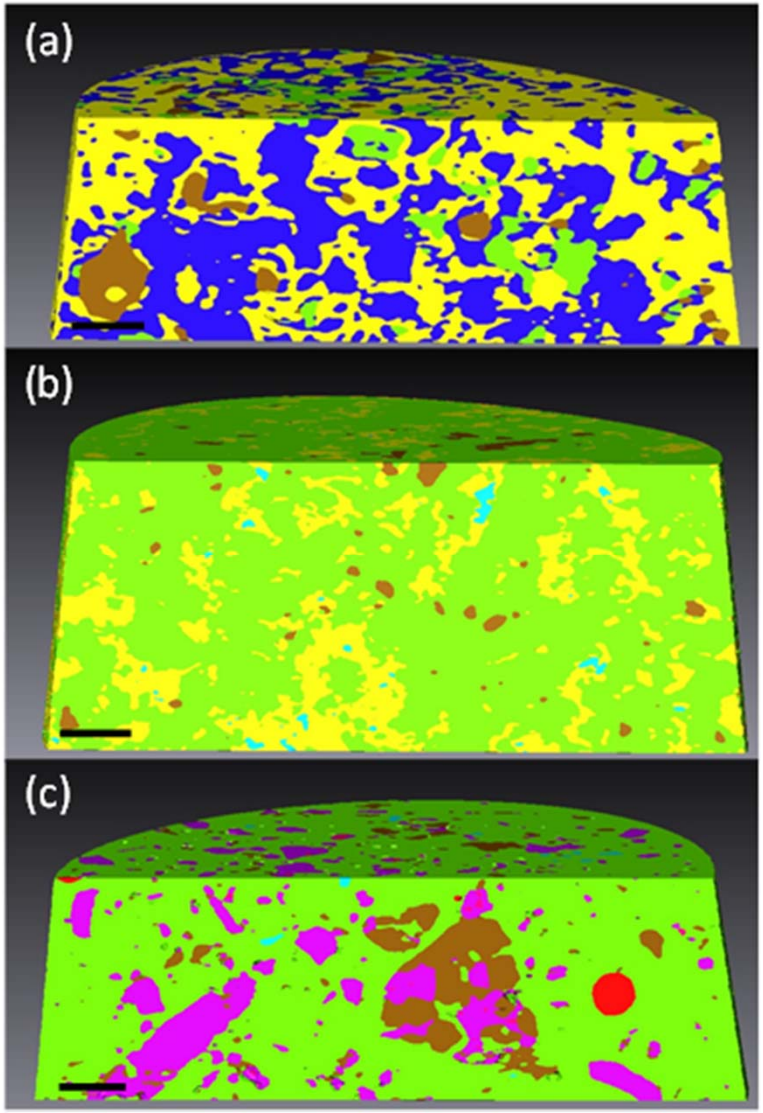
4. Case-1: Study of a new eco-cements

The composition and mass density of two aluminum hydroxide gel agglomerates have been determined:

For **ye'elimite paste** with w/c mass ratio of ≈0.45:
$(CaO)_{0.12}Al(OH)_3$
ρ=2.05(3) g·cm^{-3}
Ye'elimite, AFm, AFt, A-H gel

For **ye'elimite-gypsum paste** with w/c mass ratio of ≈0.61:
$(CaO)_{0.04}Al(OH)_3 \cdot 2.3H_2O$
ρ= 1.48(3) g·cm^{-3}
Ye'elimite, AFt, A-H gel

For **CSA paste** with w/c mass ratio of ~0.35:
~$Al(OH)_3$ - **ρ~1.8(1) g·cm^{-3}**
Ye'elimite, C_2S, AFt, A-H gel



Density of gibbsite, crystalline $Al(OH)_3$ is **2.42 gcm^{-3}**.

The density of 'amorphous' gibbsite is important to determine the volume stabilities of the resulting binders.

The densities of nanocrystalline materials (and amorphous) is very difficult to determine

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4. Case-1: Study of a new eco-cements

Table 1. Electron, Mass Densities, and Mass Attenuation Coefficients (μ) by PXCT; Expected Mass Densities Taken from the CIF Files and Expected[51] Mass Attenuation Coefficients (μ)

	n_e[a]/e$^-$·Å^{-3}			mass density[b]/g·cm^{-3}				μ[d]/cm^{-1}			
phase	Y paste	Y-G paste	CSA paste	Y paste	Y-G paste	CSA paste	expected mass density[c]/g·cm^{-3}	Y paste	Y-G paste	CSA paste	Expected μ[e]/cm^{-1}
ye'elimite	0.770(4)	0.770(5)	0.784(4)	2.58(1)	2.58(2)	2.64(1)	2.60	340	376	342	361.1
gypsum		0.704(3)			2.28(1)		2.30		274		300.0
AFt	0.563(5)	0.558(7)	0.569(7)	1.79(2)	1.77(2)	1.80(2)	1.78	152	172	159	181.0
AFm	0.605(5)			1.94(2)			2.02	231			247.7
A-H gel	0.632(8)	0.47(1)		2.05(3)	1.48(3)		2.40[f]	133	63		120.0[f]
capillary	0.670(3)	0.677(5)	0.672(3)	2.22(1)	2.25(2)	2.24(1)	2.20	160	157	156	161.8

[a]Y-G paste: 0.97(3) and 0.38(3) e$^-$·Å^{-3} for $SrSO_4$ and H_2O. CSA paste: 0.887(8), 0.978(8), and 1.08(3) e$^-$·Å^{-3} for $CaSO_4$, Ca_2SiO_4, and MgO. [b]Y-G paste: 3.4(1) and 1.13(9) g·cm^{-3} for $SrSO_4$ and H_2O. CSA paste: 2.94(3), 3.25(3), and 3.6(1) g·cm^{-3} for $CaSO_4$, Ca_2SiO_4, and MgO. [c]Theoretical mass densities are 3.96, 1.00, 2.95, 3.30, and 3.58 g·cm^{-3} for $SrSO_4$, H_2O, $CaSO_4$, Ca_2SiO_4, and MgO. [d]Measured absorption coefficients in CSA paste are 603 and 213 cm^{-1} for Ca_2SiO_4 and MgO. [e]Theoretical absorption coefficients are 582.0, 20.9, 459.8, 621.4, and 210.8 cm^{-1} for $SrSO_4$, H_2O, $CaSO_4$, Ca_2SiO_4, and MgO. [f]Values for crystalline gibbsite, $Al(OH)_3$.

4. Case-1: Study of a new eco-cements

Chemical shrinkage of ye'elimite with and without gypsum addition

Frank Bullerjahn [a,b,*], Jan Skocek [a], Mohsen Ben Haha [a], Karen Scrivener [b]

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Construction and Building Materials 200 (2019) 770–780

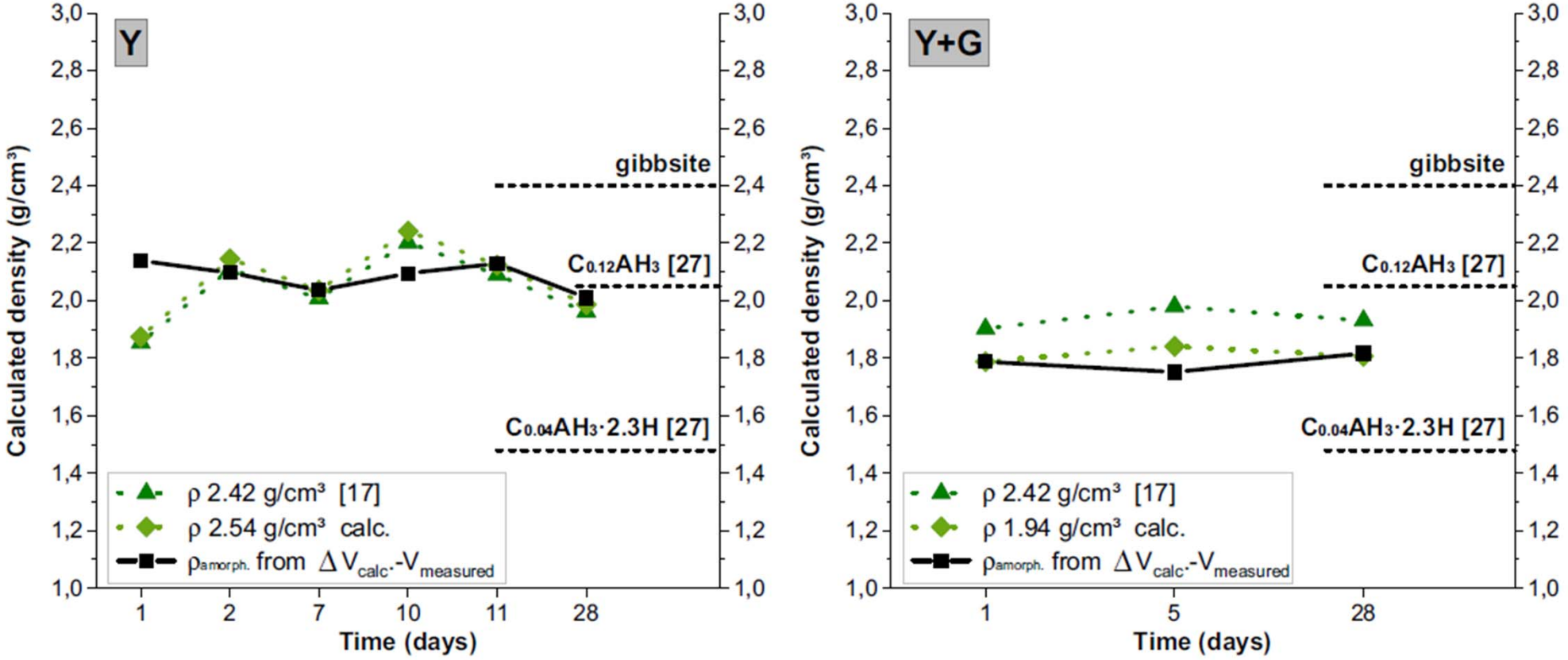


Fig. 11. Calculated amorphous phase densities.

[27] A. Cuesta, A. De la Torre, I. Santacruz, P.D.S. Trtik, D.A. Julio, M. Holler, M. Aranda, Chemistry and mass density of aluminum hydroxide gel in eco-cements by ptychographic X-ray computed tomography, J. Phys. Chem. 121 (2017) 3044–3054.

4. Case-1.2: Kinetics of new eco-cement hydration

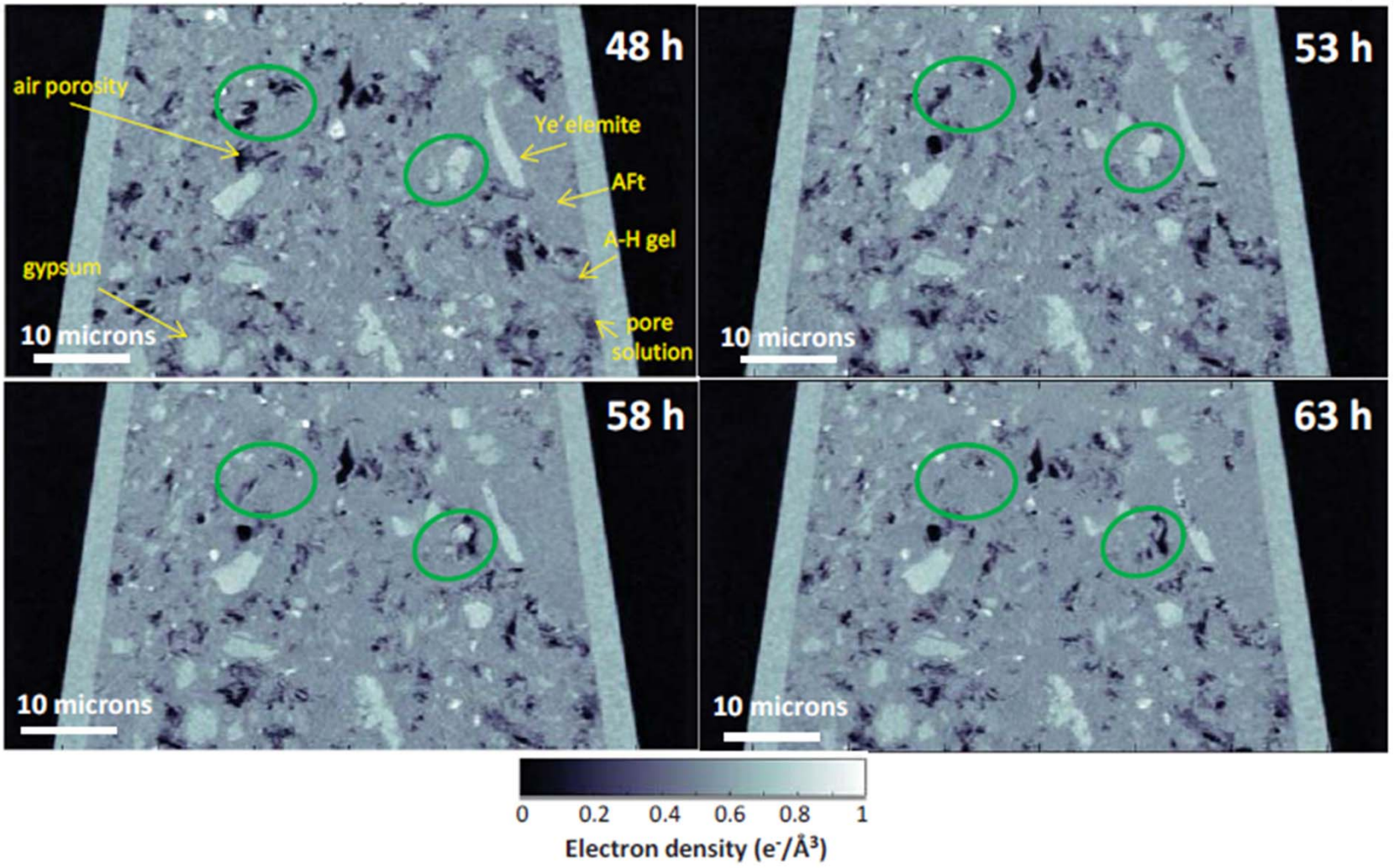
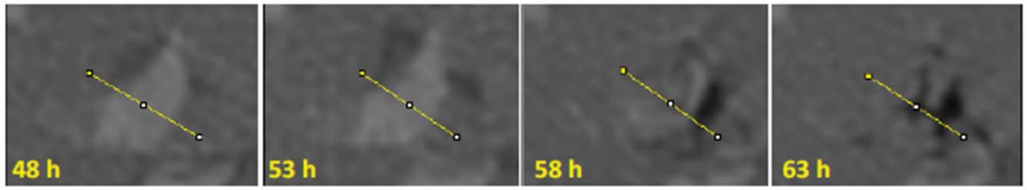
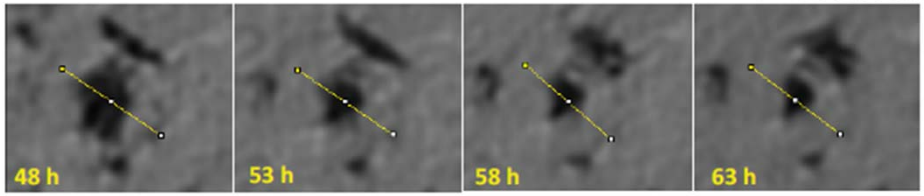
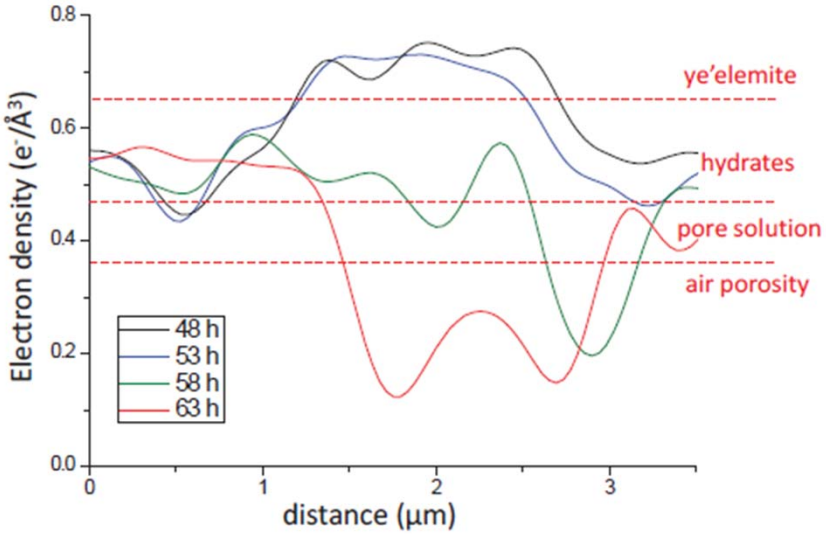


Figure 1. Vertical slices of the electron density tomograms from PXCT for ye'elimite with gypsum paste at the different ages.

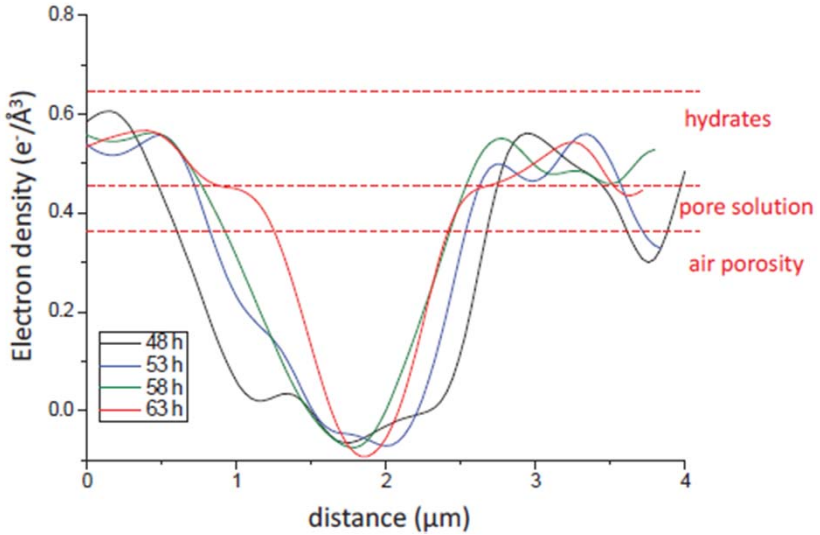
4. Case-1.2: Kinetics of new eco-cement hydration







Ye'elimite dissolution process



Hydration product precipitation

The dissolution rate was determined to be **~2 nm·min^{-1}** for some ye'elimite particles, while the precipitation rate was determined as **~1 nm·min^{-1}** for the formation of ettringite and aluminum hydroxide gel within the pores

Cuesta, A.; De la Torre, A.G.; Santacruz, I.; Trtik, P.; da Silva, J.C.; Diaz, A.; Holler, M.; Aranda, M.A.G.
"**In situ hydration imaging study of a ye'elimite paste by Ptychographic X-ray Computed Tomography**"
Proceedings of the Thirty Ninth International Conference on Cement Microscopy, International Cement Microscopy Association, Toronto, Canada, **2017**, pp 17-32.

Outline

1. **The relevant problem.**
2. **Concrete:** length scales (our everyday problem)
3. **Background:** Previous PXCT studies of Portland cement binders
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5. **Case-2:** Study of Portland including Supplementary Cementitious Materials
6. **“Christmas Wishes”**

UNIVERSIDAD DE MÁLAGA

5. Case-2: Study of Portland with SCM

$$n(\mathbf{r}) = 1 - \delta(\mathbf{r}) + i\beta(\mathbf{r})$$

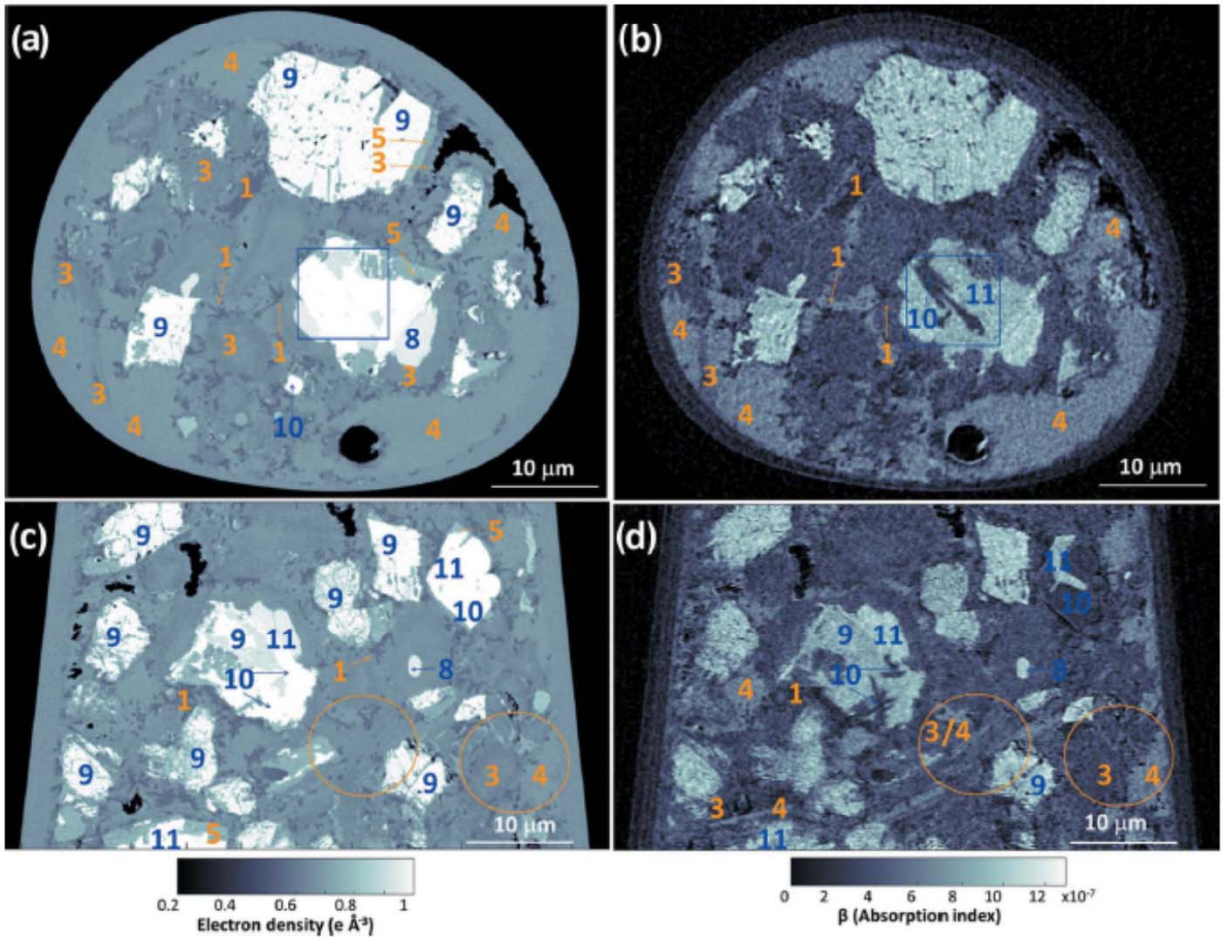
Quantitative disentanglement of nanocrystalline phases in cement pastes by synchrotron ptychographic X-ray tomography

IUCrJ (2019). 6, 473–491

Ana Cuesta,[a] Ángeles G. De la Torre,[a] Isabel Santacruz,[a] Ana Diaz,[b] Pavel Trtik,[b,c] Mirko Holler,[b] Barbara Lothenbach[d] and Miguel A. G. Aranda[e,a]*

Table 1
Chemical stoichiometries of the component phases with the abbreviation and numbering system used in the text and in the figures, respectively.

Numerical labels used in the figures	Chemical formula	Text abbreviation
1	$Ca_6Al_2(SO_4)_3(OH)_{12}\cdot 26H_2O$	AFt
2	$\sim(CaO)_{1.8}\ (SiO_2)(H_2O)_6$	LD_C-S-H
3	$\sim(CaO)_{1.8}(SiO_2)(H_2O)_4$	HD_C-S-H
4	$Ca(OH)_2$	CH or Portlandite
5	$\sim Ca_3FeAl(SiO_4)_{0.84}(OH)_{8.64}$	Fe–Al–Si–**Hg**
6	$\sim SiO_2$	FA
7	$CaCO_3$	CC or Calcite
8	Ca_3SiO_5	C_3S
9	Ca_2SiO_4	C_2S
10	MgO	MgO
11	Ca_2AlFeO_5	C_4AF



Portland cement paste (neat)
Data taken @ cSAXS with OMNY

5. Case-2: Study of Portland with SCM

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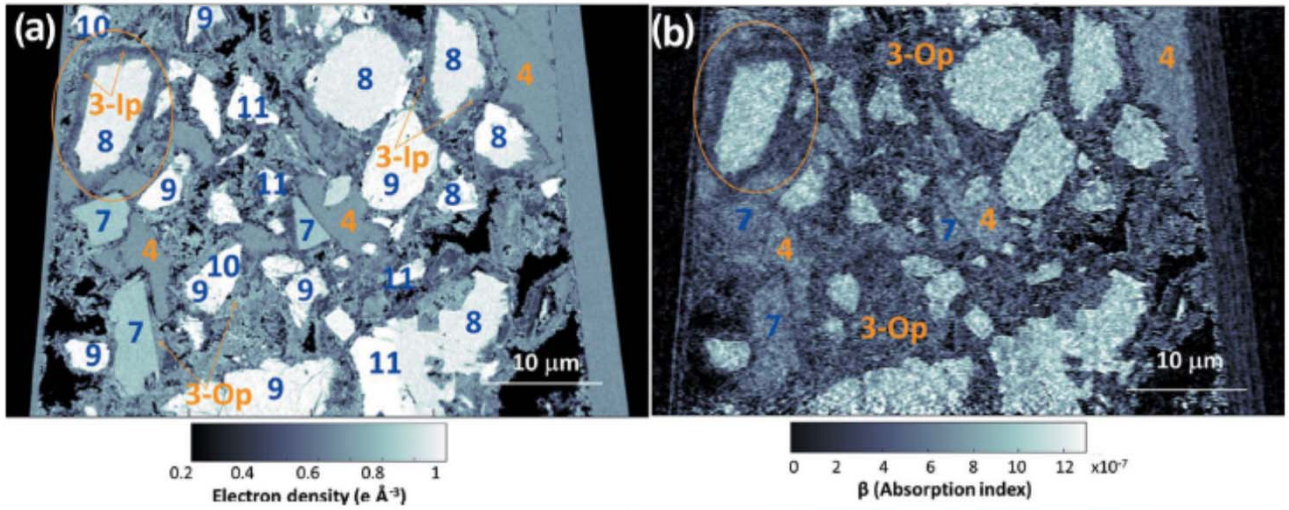
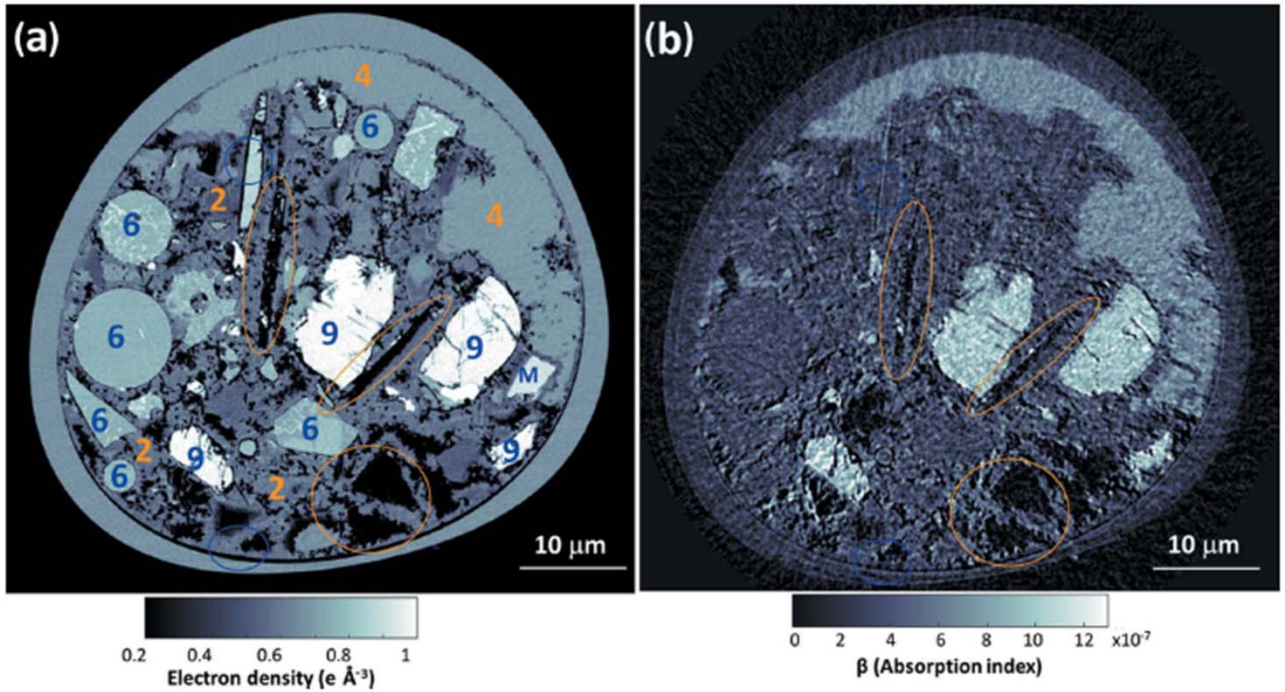


Portland cement + calcite blend



Portland cement + fly ash blend

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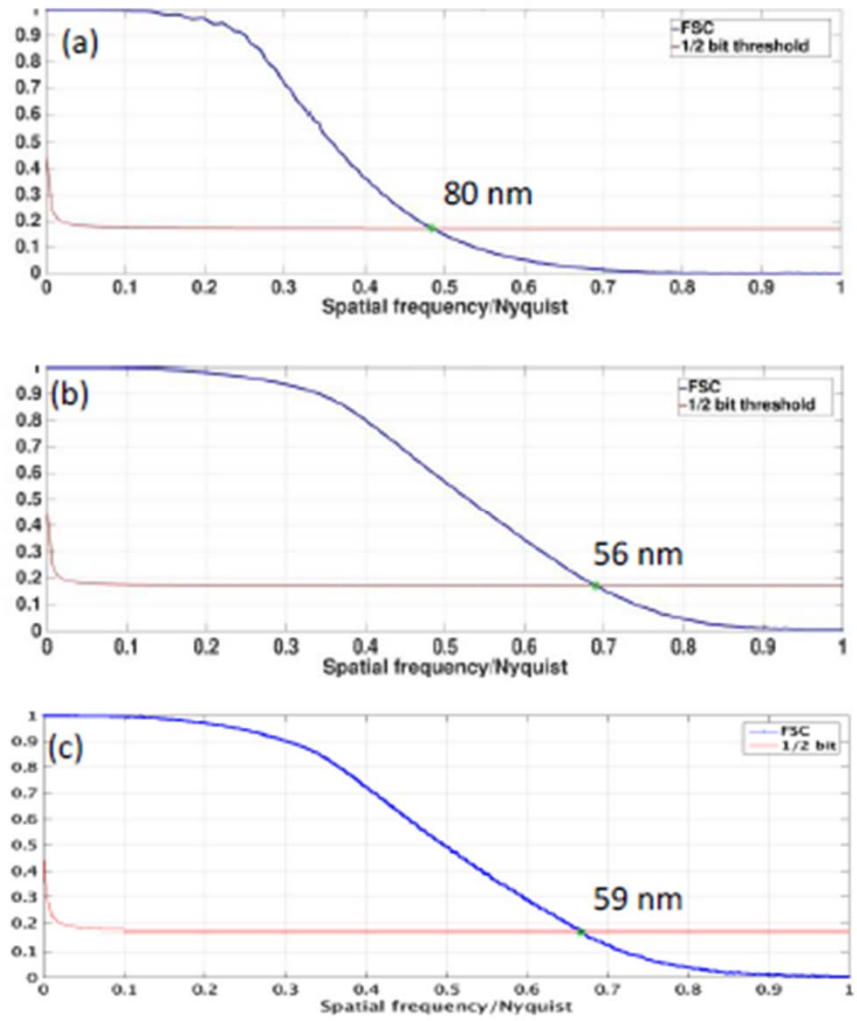

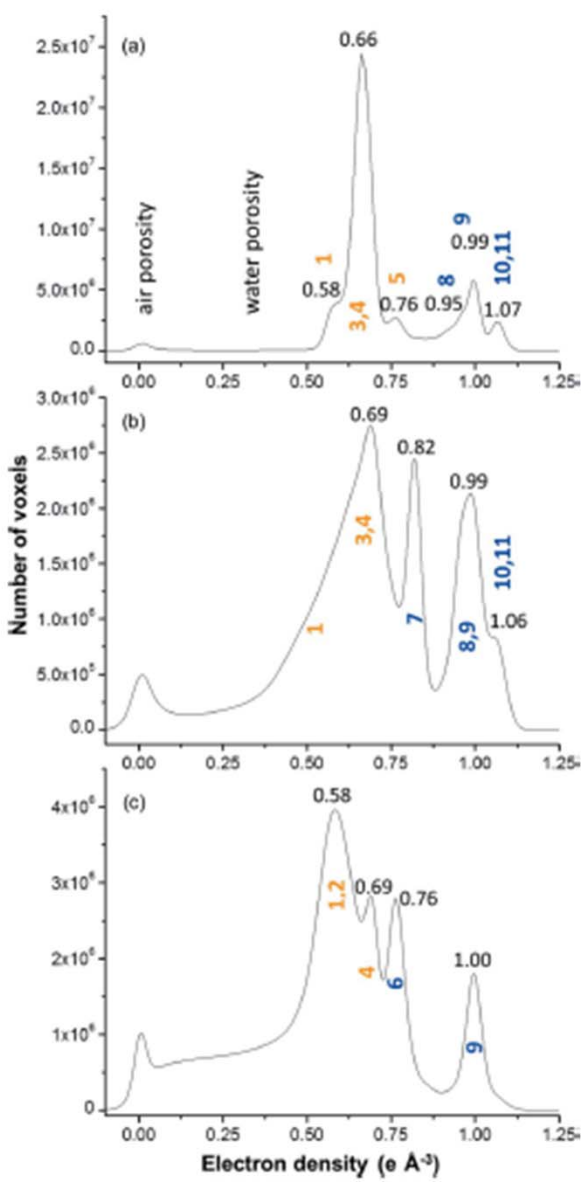

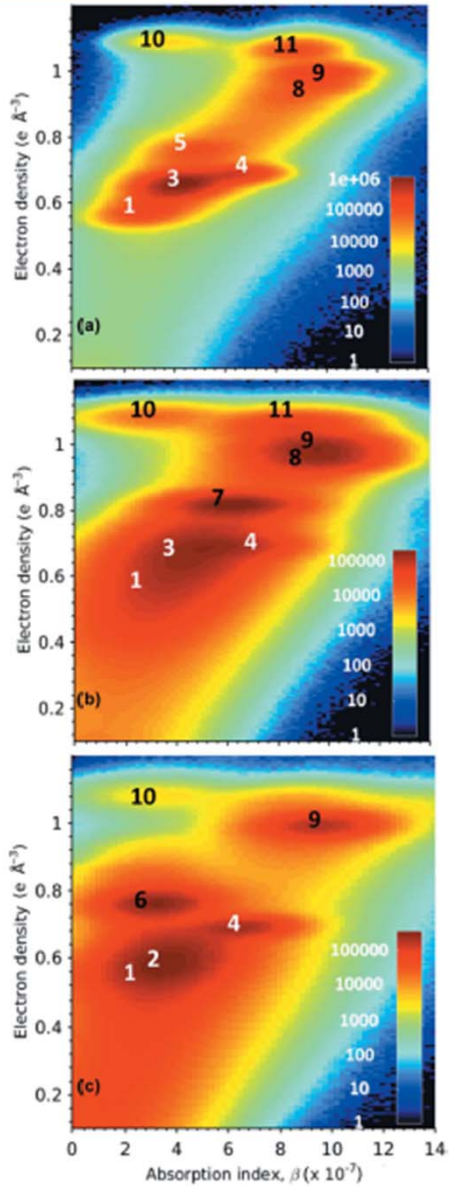

FSC plots for (a) PC, (b) PC-limestone, (c) Pc-fly ash

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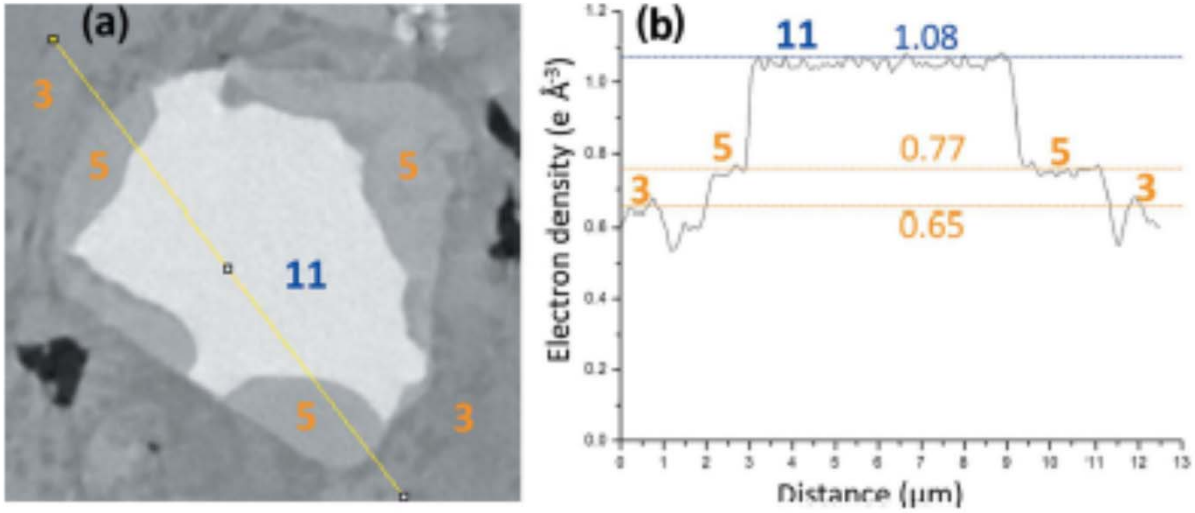
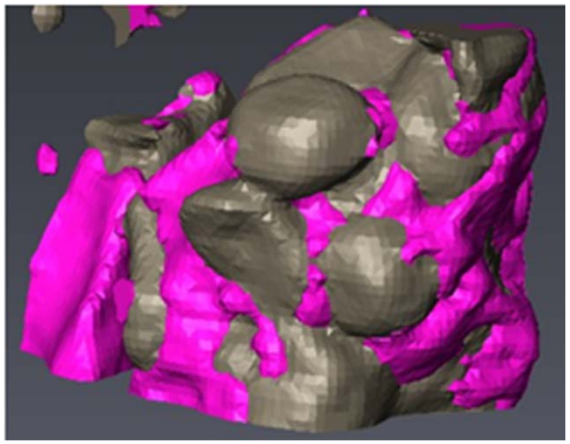


Figure 6
(*a*) A partially reacted C_4AF particle surrounded by hydrated component phases from the electron-density tomogram of the neat PC paste. The different phases and a line to show the electron-density values are also shown. (*b*) Electron-density values corresponding to the yellow line in (*a*), with horizontal lines showing the average values of the electron densities obtained for the component phases using ten different particles, data from Table 3. It clearly shows, as an example, how phase 5 encloses the unreacted fraction of the C_4AF particle. From the electron-density value and its spatial arrangement, phase 5 is concluded to be Fe–Al siliceous hydrogarnet.



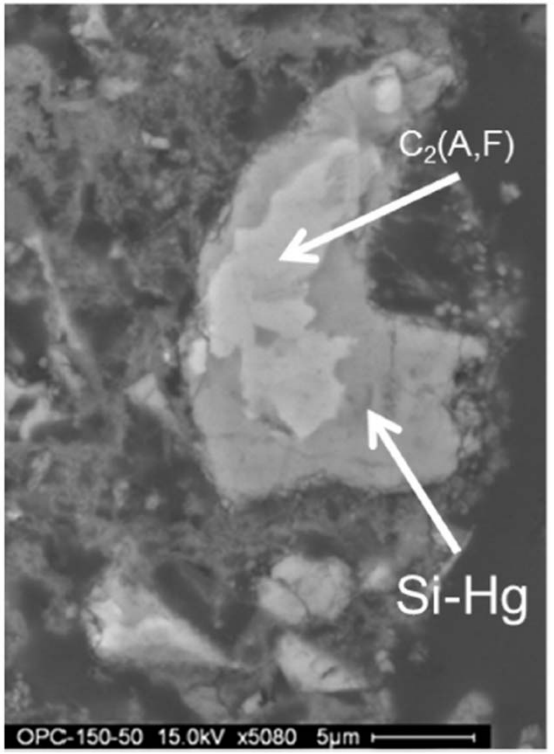


Fig. 8. SEM of ferrite clinker surrounded by hydration products after selective extraction.
Cement and Concrete Research 58 (2014) 45–55

For the neat PC paste, PXCT study gave densities of **2.11** and **2.52** g cm^{-3} and a content of **41.1** and **6.4 vol%** for amorphous C-S-H [$(CaO)_{1.8}SiO_2(H_2O)_4$] & iron siliceous hydrogarnet [$Ca_3FeAl(SiO_4)_{0.84}(OH)_{8.64}$] gels, respectively

5. Case-2: Study of Portland with SCM

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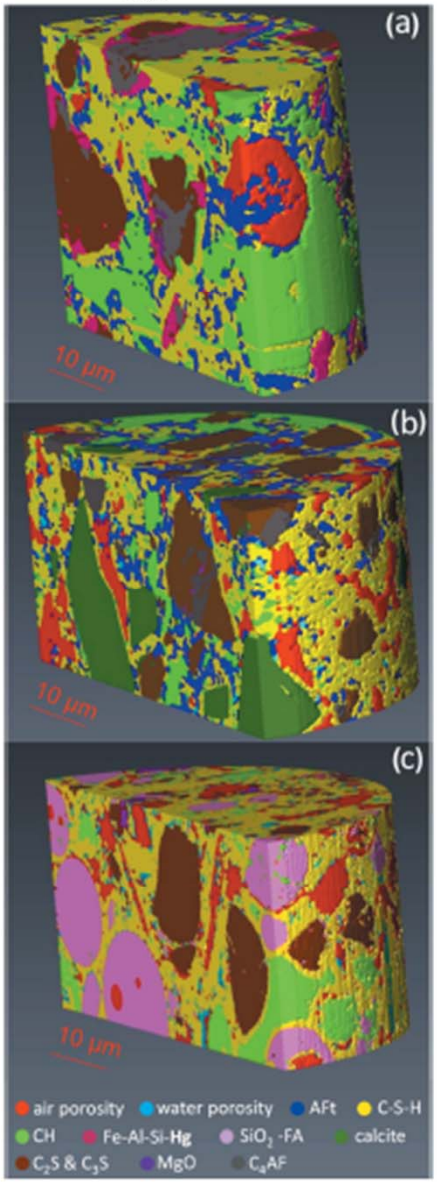
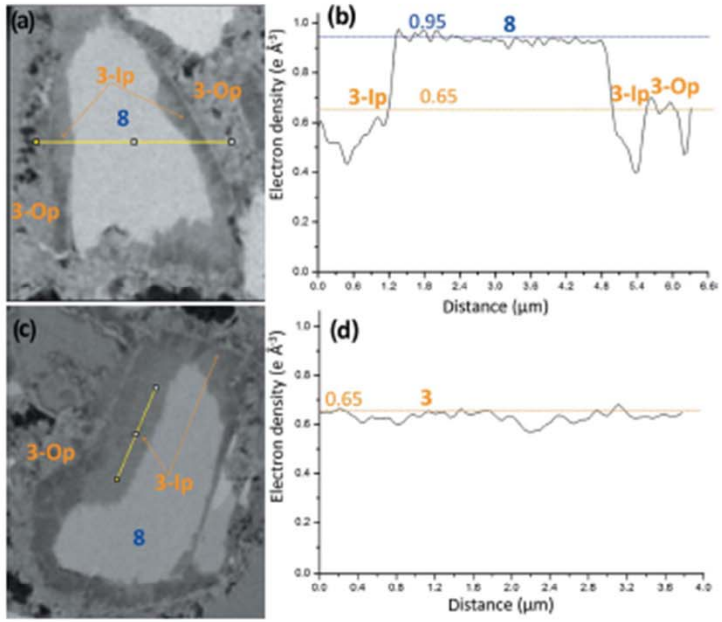
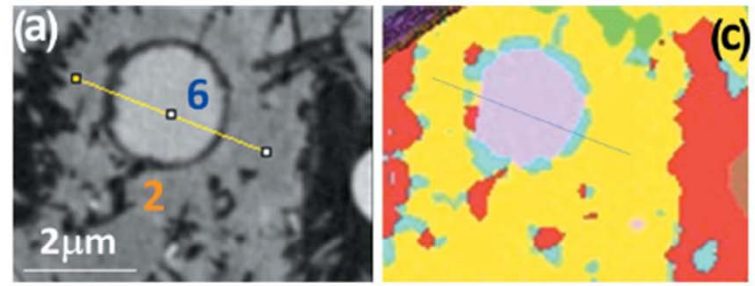
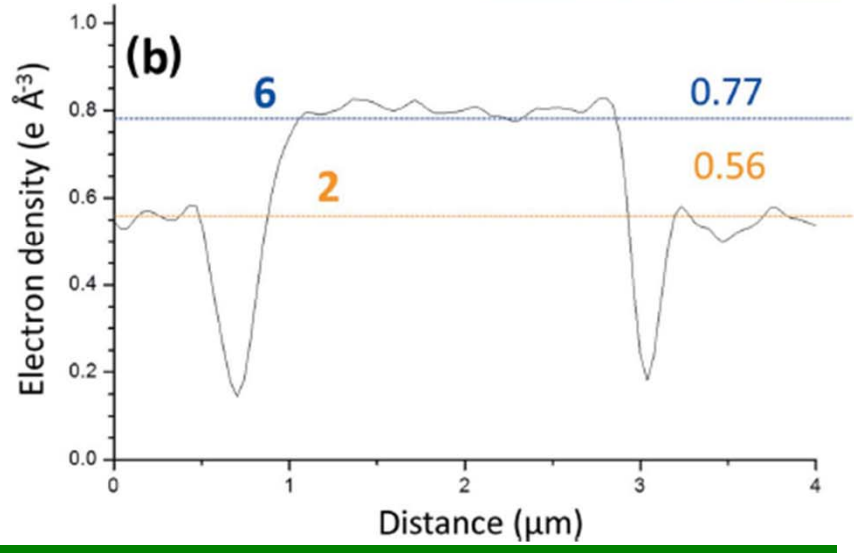




Spatial resolution
after segmentation ?





5. Case-2: Study of Portland with SCM

$$n(\mathbf{r}) = 1 - \delta(\mathbf{r}) + i\beta(\mathbf{r})$$

Table 3
Electron, mass densities and mass attenuation coefficients (μ) obtained by PXCT for the neat PC paste at five months of hydration; expected mass densities taken from the CIF files (Aranda, 2016) and expected μ (Henke *et al.*, 1993) are also given.

Phase	Electron density (e Å^{-3}) average of ten values	Electron density (e Å^{-3}) full volume†	Expected electron density (e Å^{-3})	Calculated density (g cm^{-3})	Expected density (g cm^{-3})‡	Calculated μ (cm^{-1}) (corrected)†	Expected μ (cm^{-1})
1, AFt	0.568 (4)	0.55	0.56	1.80 (1)	1.78	187	181.0
3, HD_C-S-H	0.657 (7)	0.64	—	2.11 (2)	—	279	—
4, CH	0.690 (6)	0.67	0.69	2.23 (2)	2.23	440	446.1
5, Fe-Al-Si-**Hg**	0.766 (8)	0.76	—	2.52 (3)	3.09	350	—
8, C_3S	0.957 (11)	0.92	0.95	3.18 (4)	3.15	614	657.8
9, C_2S	0.999 (4)	0.98	0.99	3.32 (1)	3.30	646	637.3
10, MgO	1.080 (10)	1.05	1.07	3.58 (3)	3.58	228	217.4
11, C_4AF	1.080 (10)	1.05	1.10	3.66 (3)	3.73	591	566.4
Capillary	0.675 (4)	—	0.66	2.24 (1)	2.20	—	—

All reported attenuation coefficients (for the three pastes) are determined from the complex part or the refraction index datasets multiplied by the correction factor 1.05.

Table 4
Electron, mass densities and μ obtained by PXCT for the PC–CC blend paste after five months of hydration; expected mass densities taken from the CIF files (Aranda, 2016) and expected μ (Henke *et al.*, 1993) are also given.

Phase	Electron density (e Å^{-3}) average of ten values	Electron density (e Å^{-3}) full volume†	Expected electron density (e Å^{-3})	Calculated density (g cm^{-3})	Expected density (g cm^{-3})‡	Calculated μ (cm^{-1}) (corrected)†	Expected μ (cm^{-1})
1, Monocarbo, AFt pore solution	0.45 (3)	0.48	—	1.36 (9)	—	193	—
3, HD_C-S-H	0.64 (1)	0.63	—	~2.05	—	228	—
4, CH	0.698 (7)	0.68	0.69	2.23 (2)	2.23	464	446.1
7, CC	0.826 (4)	0.80	0.82	2.75 (1)	2.71	411	415.2
8, C_3S	0.963 (4)	0.93	0.95	3.20 (1)	3.15	649	657.8
9, C_2S	0.998 (8)	0.99	0.99	3.32 (3)	3.30	639	637.3
10, MgO	1.062 (16)	1.06	1.07	3.52 (5)	3.58	211	217.4
11, C_4AF	1.062 (16)	1.06	1.1	3.60 (5)	3.73	566	566.4
Capillary	0.674 (6)	—	0.66	2.24 (2)	2.20	—	—

† Values obtained from the segmented components by *Avizo* software. ‡ The expected density values are determined from crystallographic data and so they are not no available for nanocrystalline/amorphous components.

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6. “Christmas Wishes”

- **To select one (or two) samples and to carry out a Round Robin in all (at least) European ptycho-tomography BLs**

- To understand why the absorption tomograms have better (or worst) s/n ratio(s) if recorded in very similar conditions. Internal porosity?

- To understand why absorption quantitative values are underestimated by about 5%.

- Will the high flux of EBS render better absorption tomograms before burning/boiling the water-containing samples?

- Any views on the spatial resolution *after segmentation*, which is (one of the) most important information to be derived.

- Relevant studies are done in real samples, the quest for better spatial resolution should not be done sacrifying the field of view (too much).

- 20 hours for tomo is too slow. Every strategy to take it to ~1-2 hours is most needed.

- **Pozzolanic reaction:** $Al_2Si_2O_{7\ amorph} + Ca(OH)_{2\ cryst} + H_2O \rightarrow C\text{-}S\text{-}H_{\ amorph} + C\text{-}A\text{-}S\text{-}H_{\ amorph}$

- **Fast online reconstruction is a must.**

Acknowledgements

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