Microstructure and transport properties of partially saturated concrete

International Workshop on Mechanisms of Concrete Carbonation, ENPC, Champs-sur-Marne

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Scope

I. Transport properties of partially saturated concrete
   - Drying & re-wetting
   - Microcracking
   - Moisture hysteresis

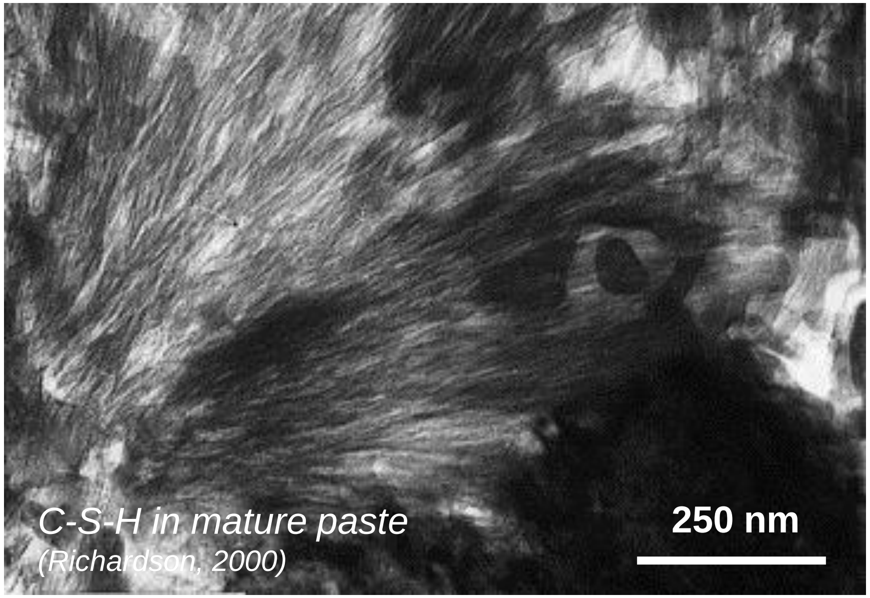
II. Relevance to carbonation
   - Transport properties of carbonated concretes containing SCMs

III. Case study: Composting facility

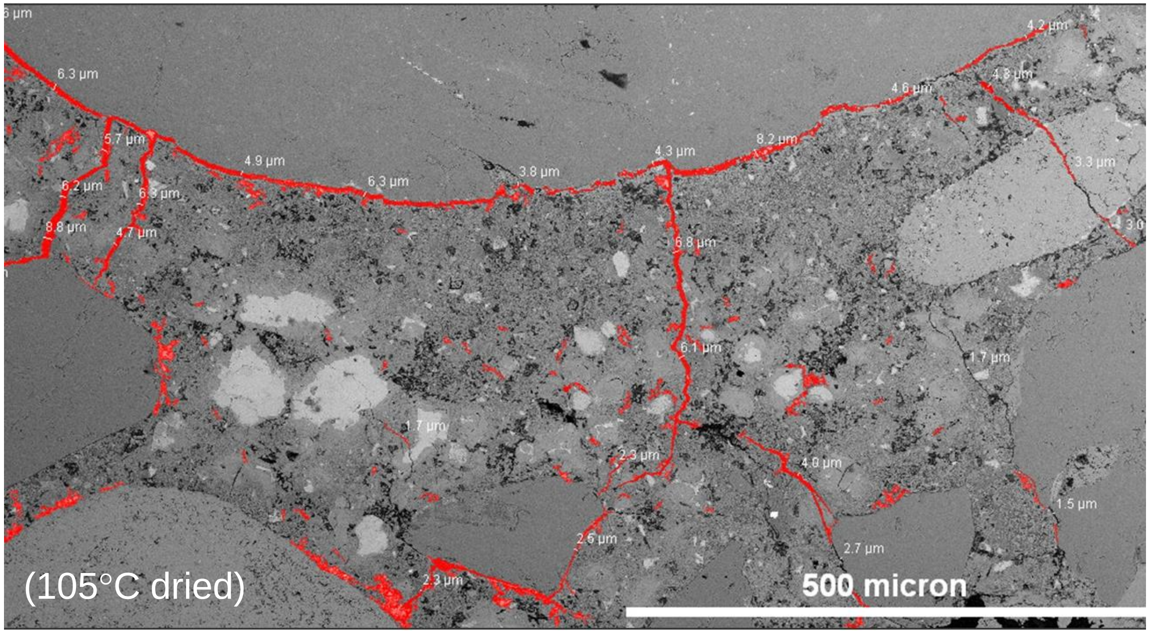
IV. Concluding remarks

Effects of drying on microstructure

RH (%)	Water driven off
100 – 85	Water in large capillary pores (> 10 nm)
85 – 50	Gel water in LD C-S-H
50 – 25	Gel water in HD C-S-H
< 25% or at high temp.	Interlayer water between C-S-H sheets (~ 2 nm)



- **Saturation degree ↓**
- **Accessible porosity ↑**
- **C-S-H densifies**
- **Pore coarsening**
- **Shrinkage → microcracking**
- **Effects on transport properties?**
- **Can we de-couple these?**



Samples (selection)

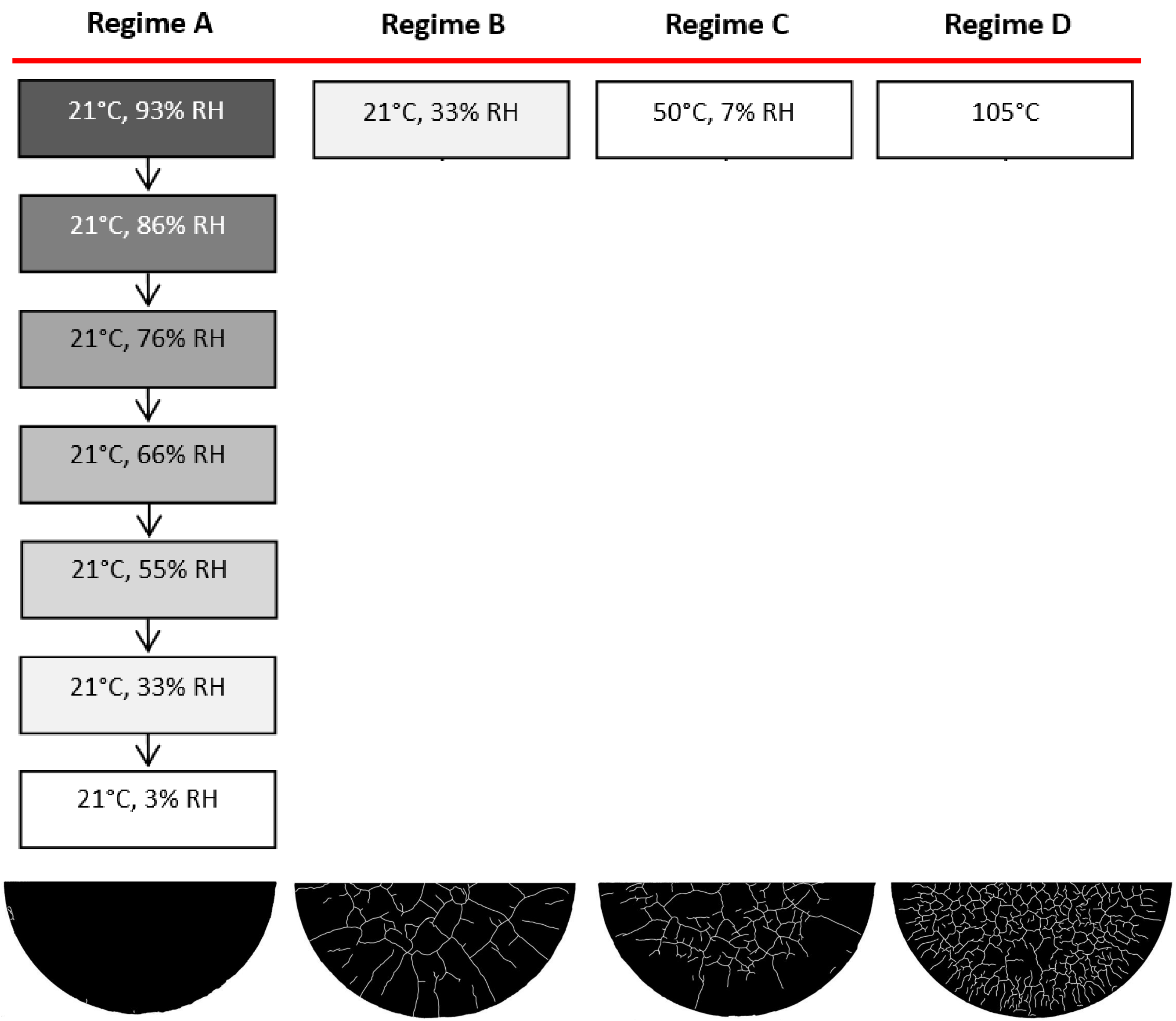
Series	Binder	Sample type	w/b	Aggregate type	Aggregate fraction (%)	Max agg. size (mm)
I	CEM I	Paste	0.35 0.50	----	0	-----
		Mortar	0.35 0.50	Siliceous sand	50 60	5, 2.5
		Concrete	0.35 0.50	Siliceous sand & limestone coarse	68	10, 20
II	CEM I + **10% SF**	Concrete	0.35	Siliceous sand & limestone coarse	68	10, 20
III	CEM I + **70% GGBS**	Concrete	0.35	Siliceous sand & limestone coarse	68	10, 20

100 mm

t = 25 or 50 mm

- *100Ø mm discs, as cast or cut*
- *Three replicates*
- *Sealed cured: 3, 28 & 90d*

Drying regimes to generate microcracking



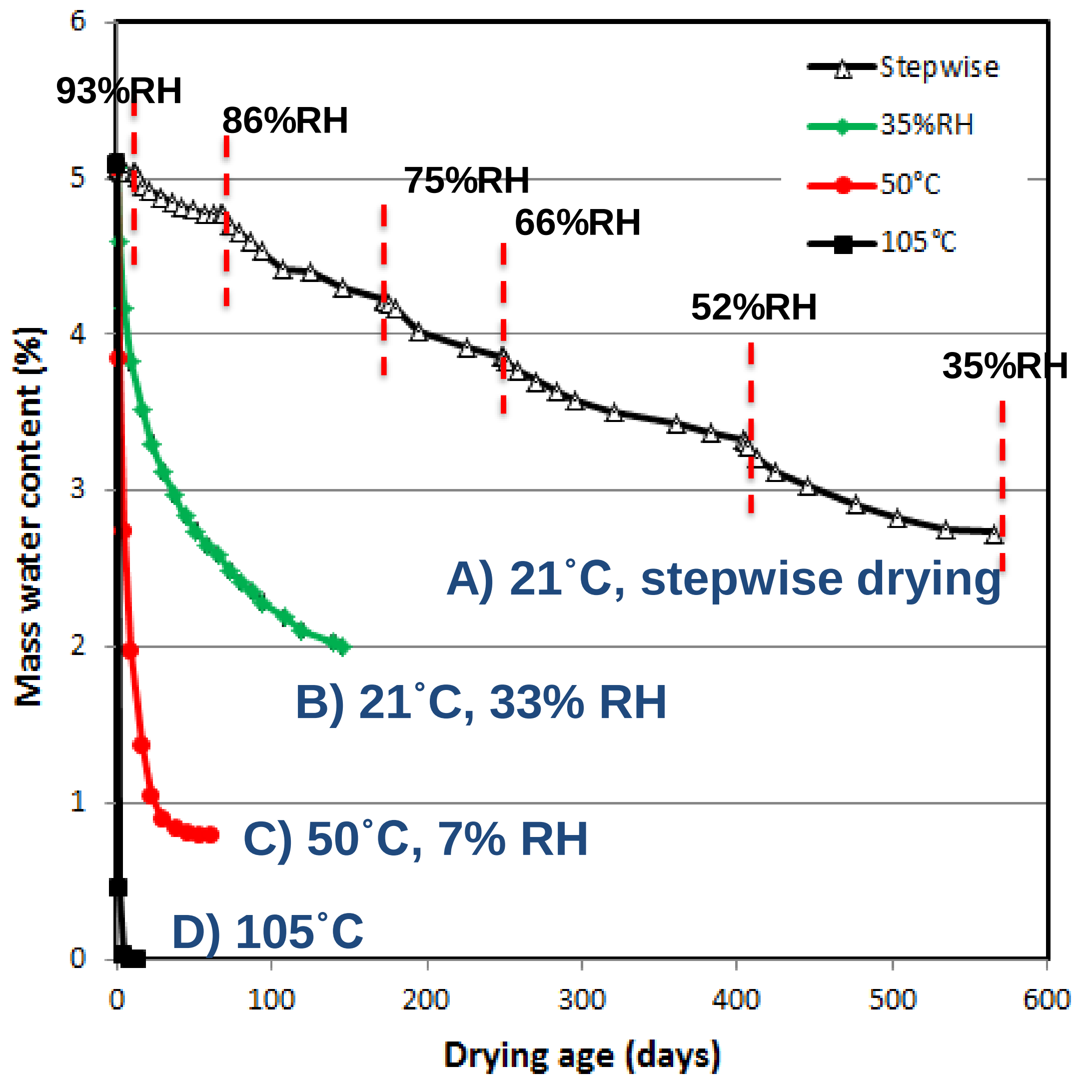
- **Drying to induce varying degrees of microcracking**
- **“Equilibrium” = mass loss < 0.01% per day**
- **Characterise microcracking**
- **Measure transport property at every conditioning step…**

Drying regimes to generate microcracking

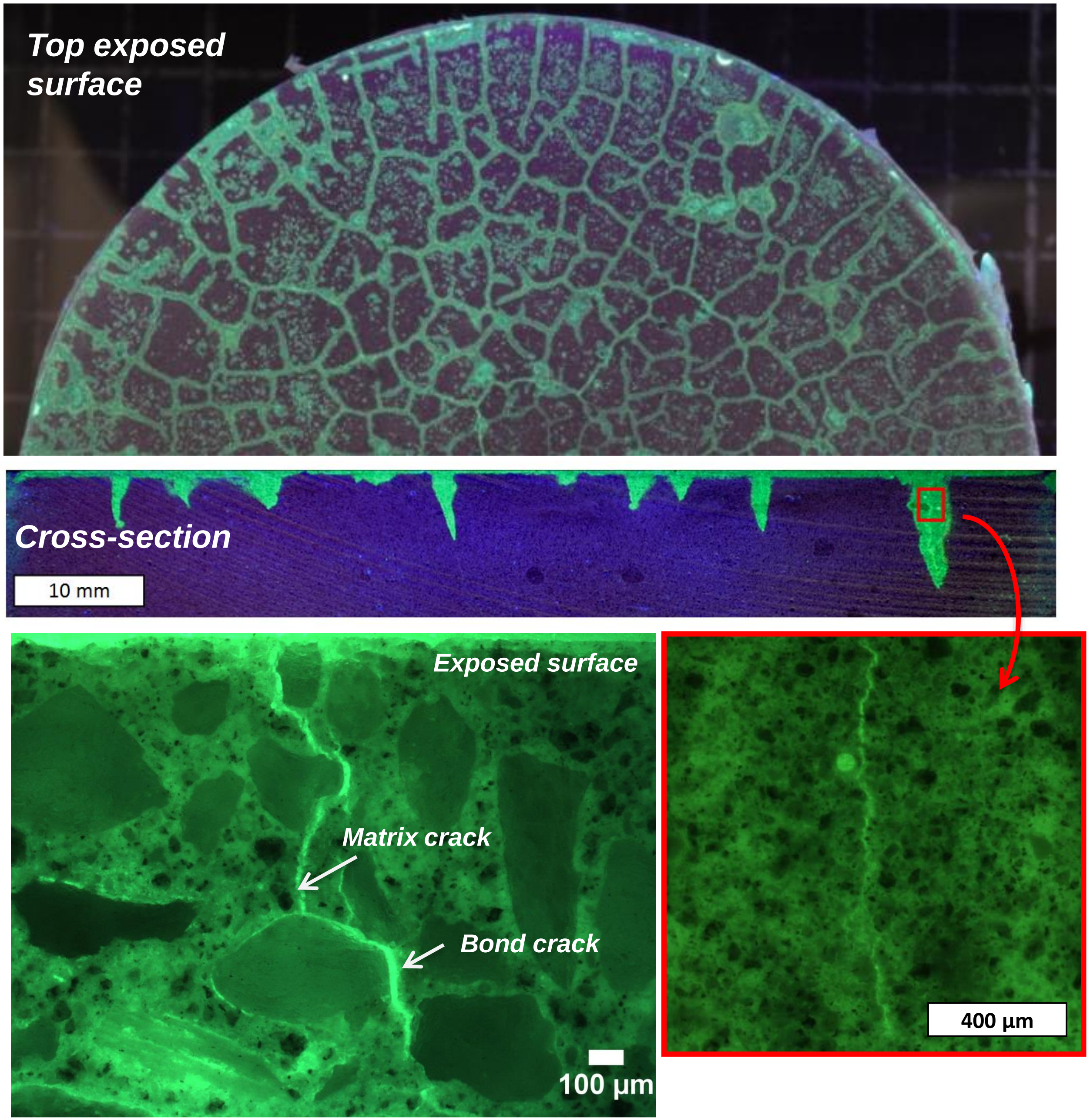
Curing:

- **3-day**
- **28-day**
- **90-day**

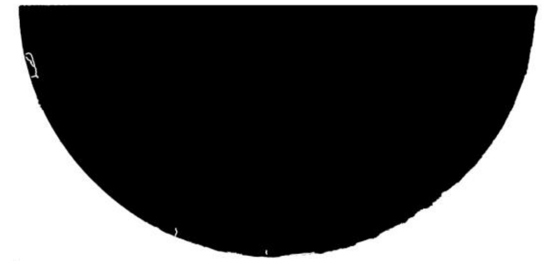
Conditioning: four drying regimes to "constant" mass (< 0.01% per day):



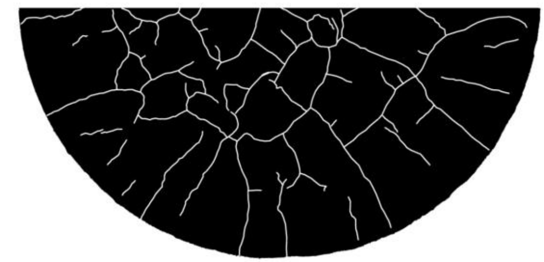
Microcracking (width < 100 μm)



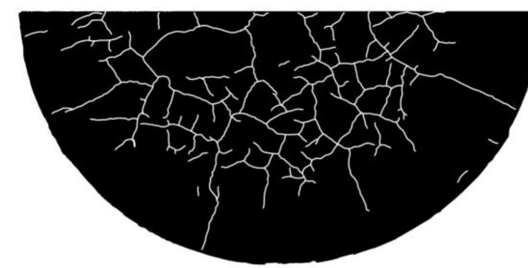
w/c = 0.50, sample ø100mm



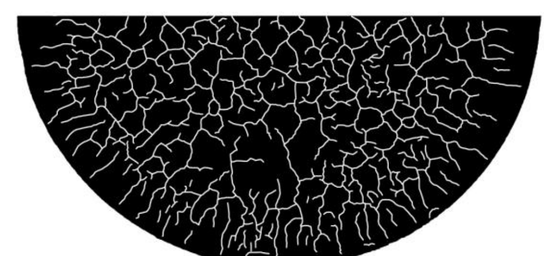
21 ℃, stepwise drying



21 ℃ , 35%RH



50 ˚C, 7% RH



105 ˚C

Characterisation of microcracking – quantitative analysis

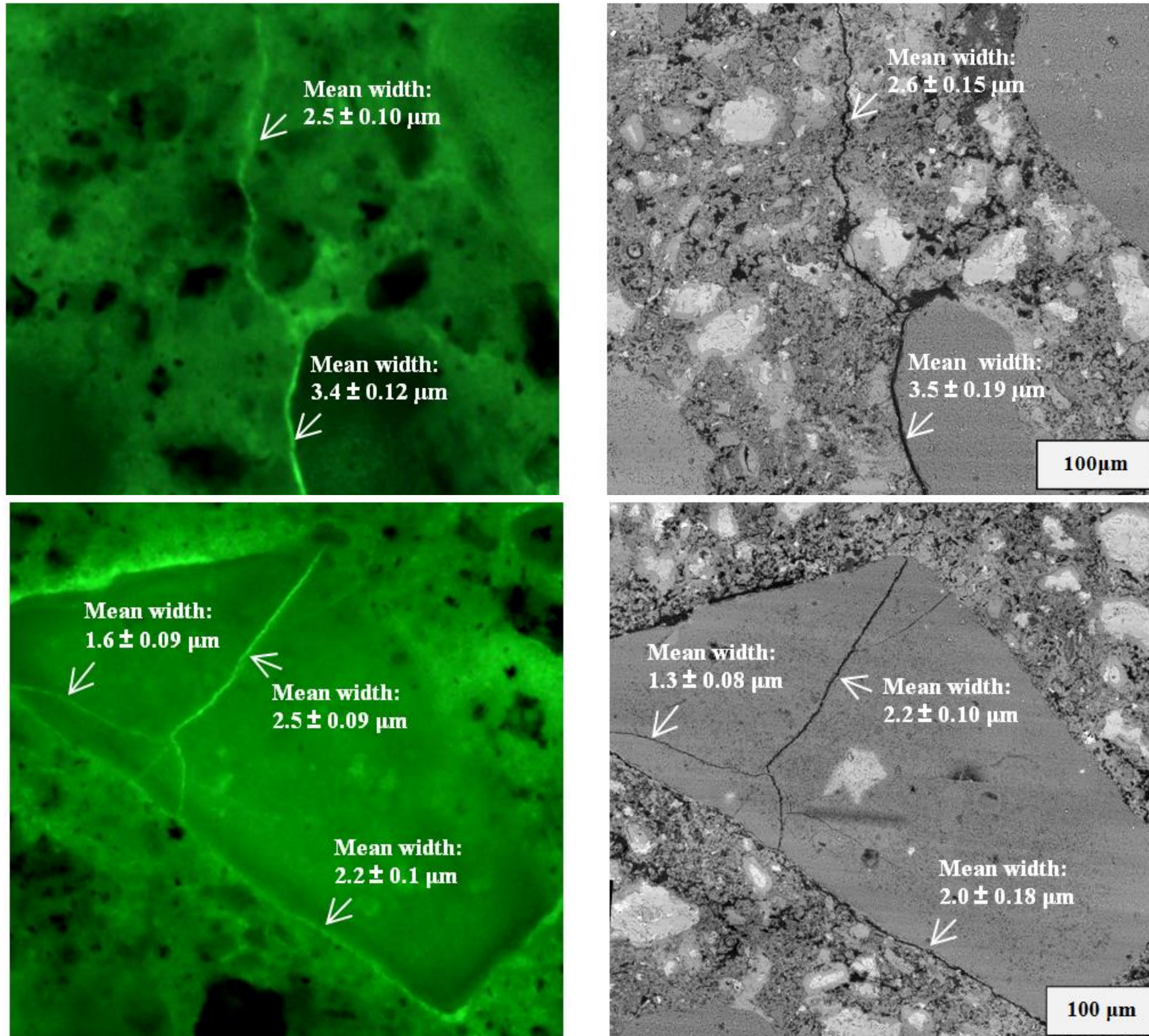
(d) Imaging

Fluorescence microscopy
- *50× magnification*
- *Pixel size 0.89 µm*

SEM-BSE
- *500× magnification*
- *Pixel size 0.09 µm*

3D LSCM + MicroCT

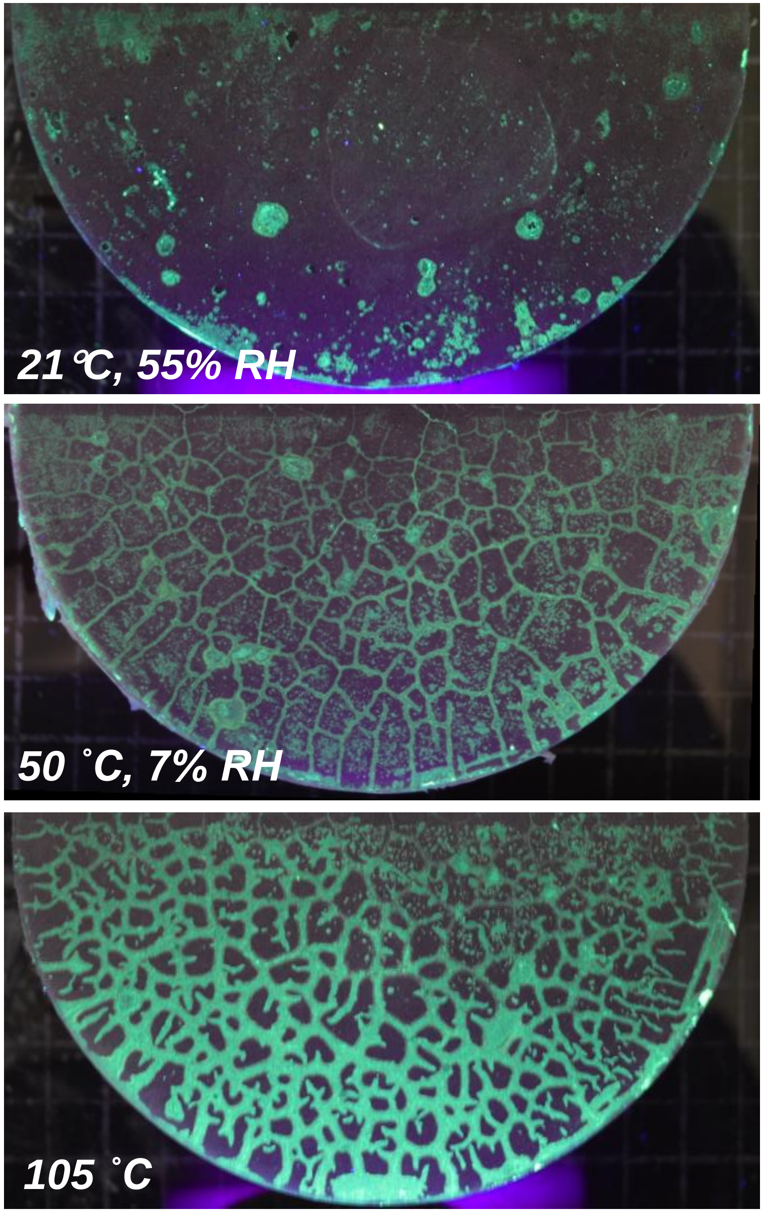
(e) Image analysis:
- *Width, length*
- *Depth*
- *Density*
- *Orientation*

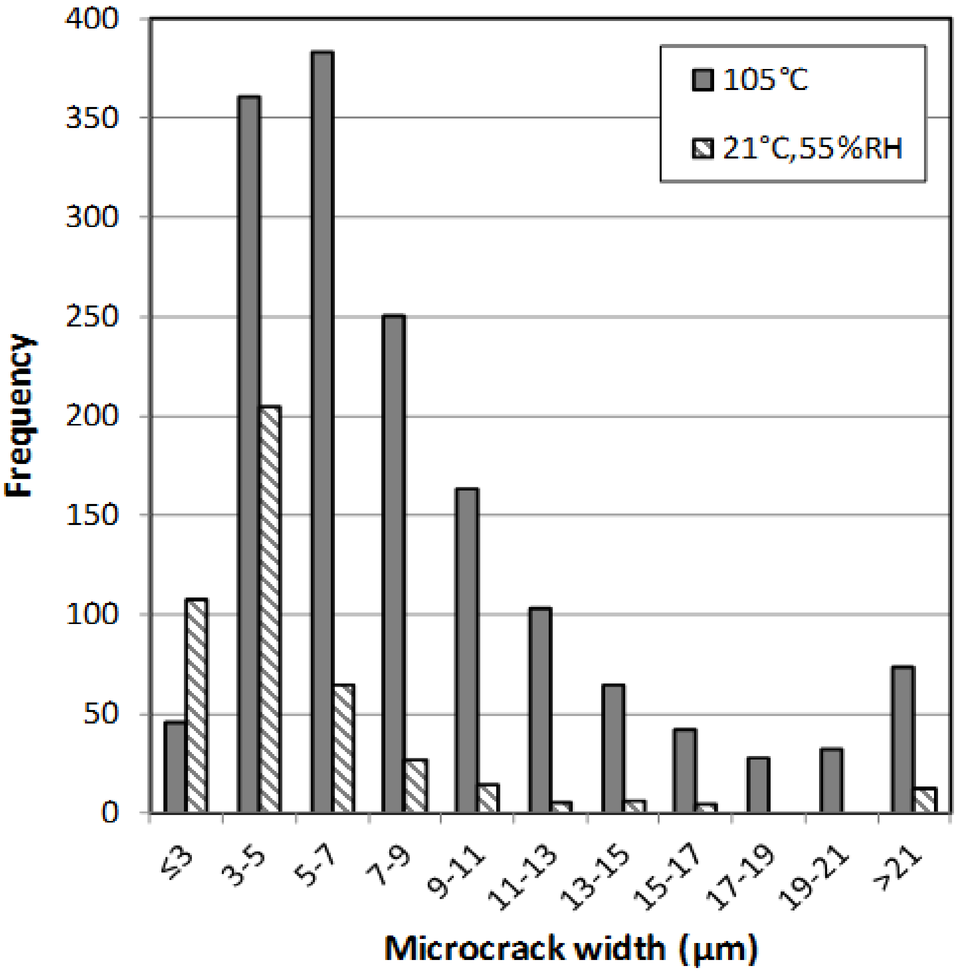


Fluorescence vs. BSE microscopy. Crack widths were measured at 10 µm intervals and averaged values (± standard error) compared

Microcracking – on surface exposed to drying

CEM I paste, 28-d, w/c 0.50

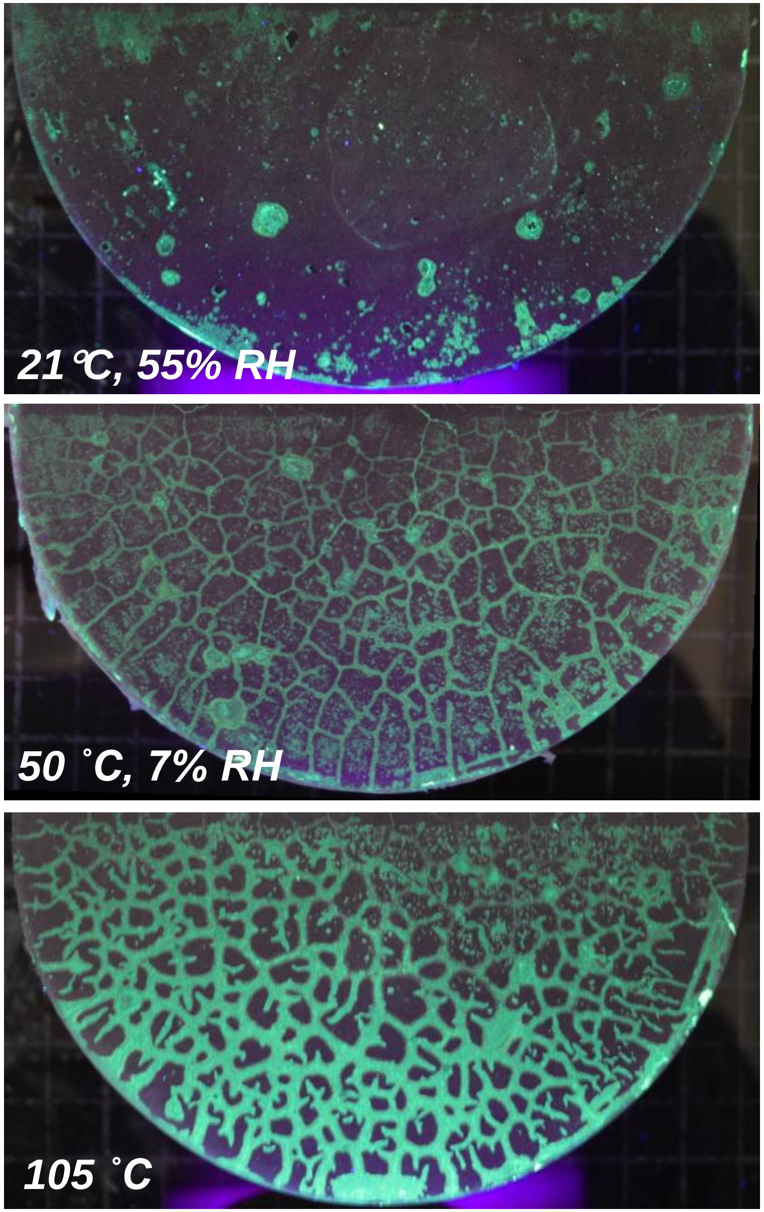


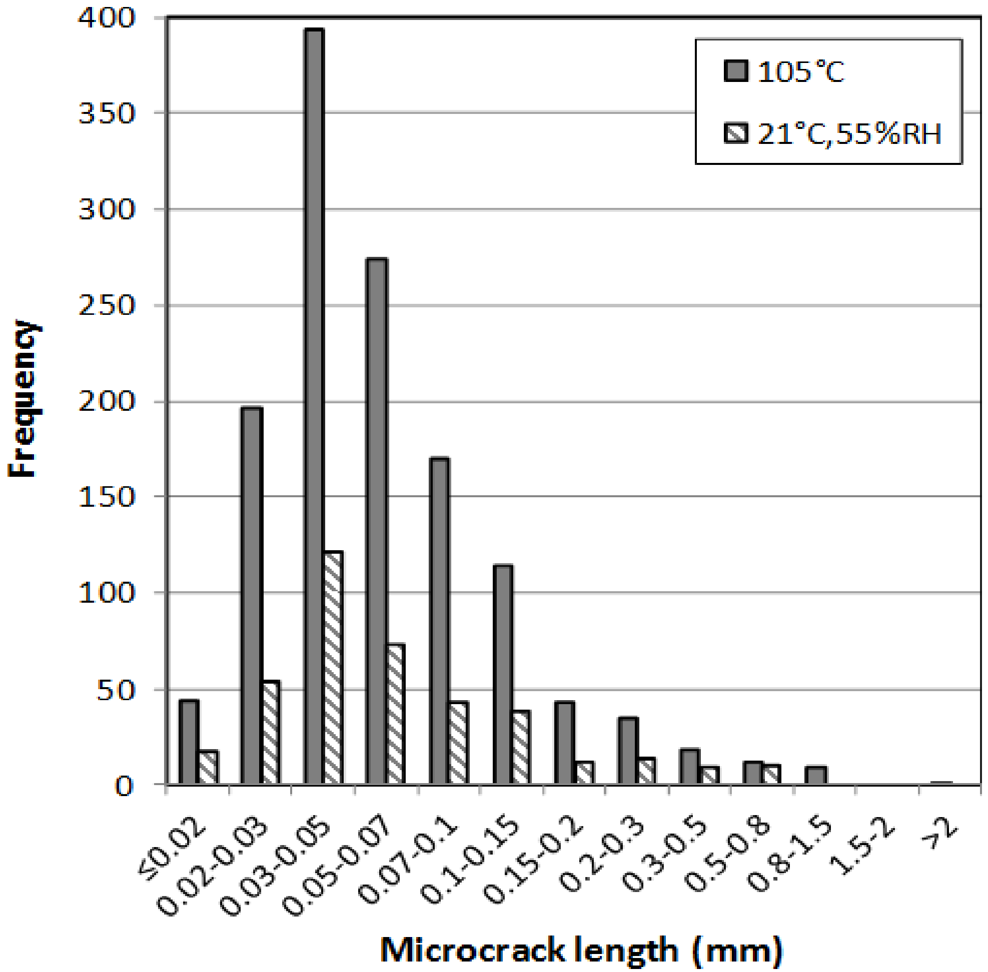


- Typical "map-cracking"
- Crack width range from 1-60 µm
- Crack width increases with drying severity
- > 80% have widths < 10 µm

Microcracking – on surface exposed to drying

CEM I paste, 28-d, w/c 0.50

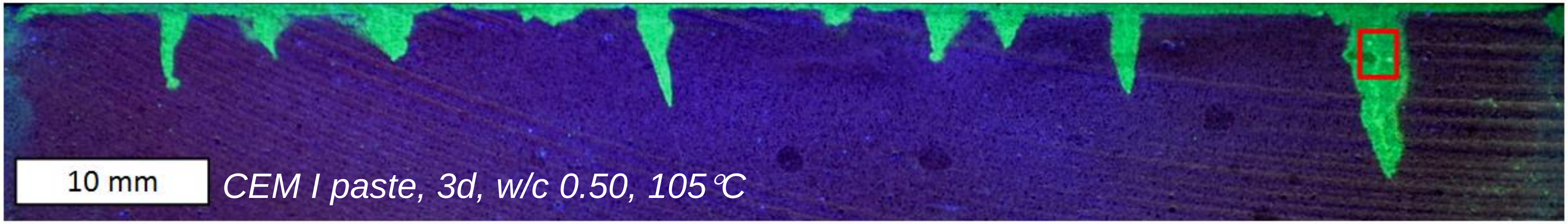


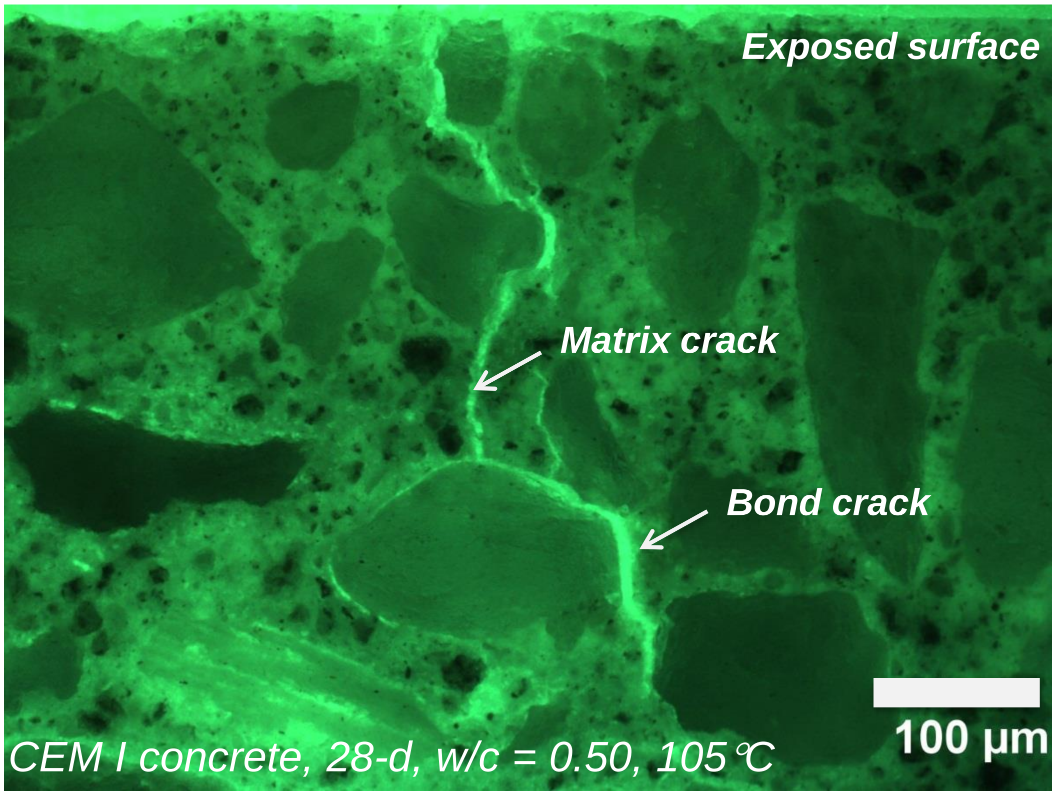


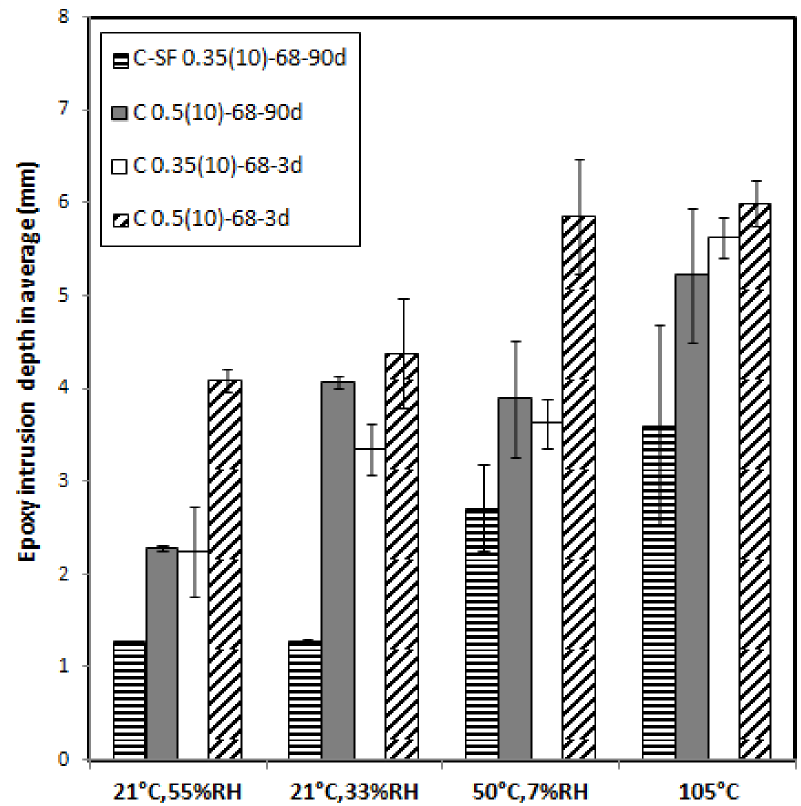
- Microcrack length increases with drying severity
- > 80% have lengths < 100 μm

Microcracking – on cross section

Exposed surface

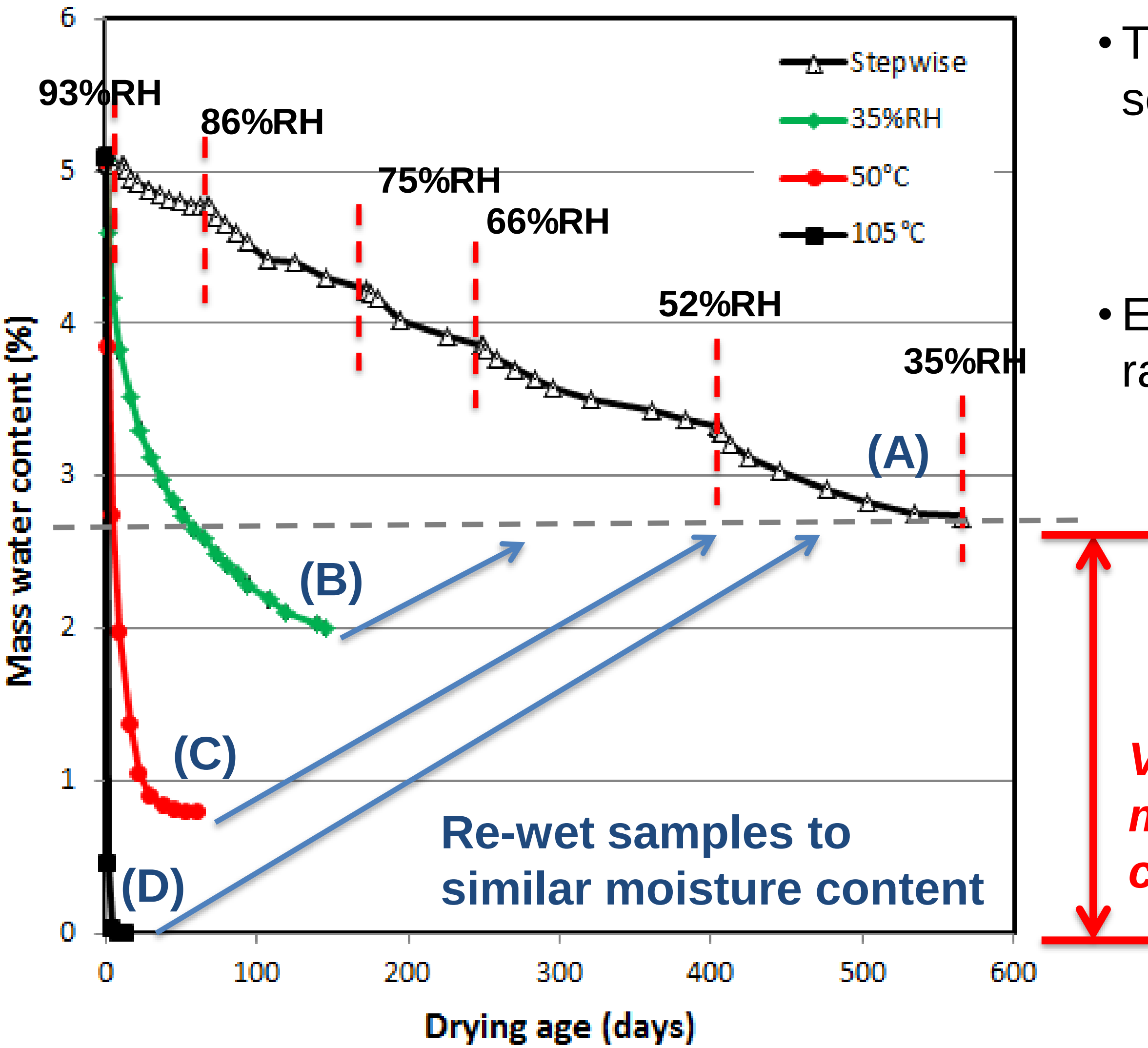






- Microcracks develop approximately perpendicular to exposed surface
- Crack width and depth increases with drying severity
- < 10 mm from exposed surface

Effect of drying on mass transport



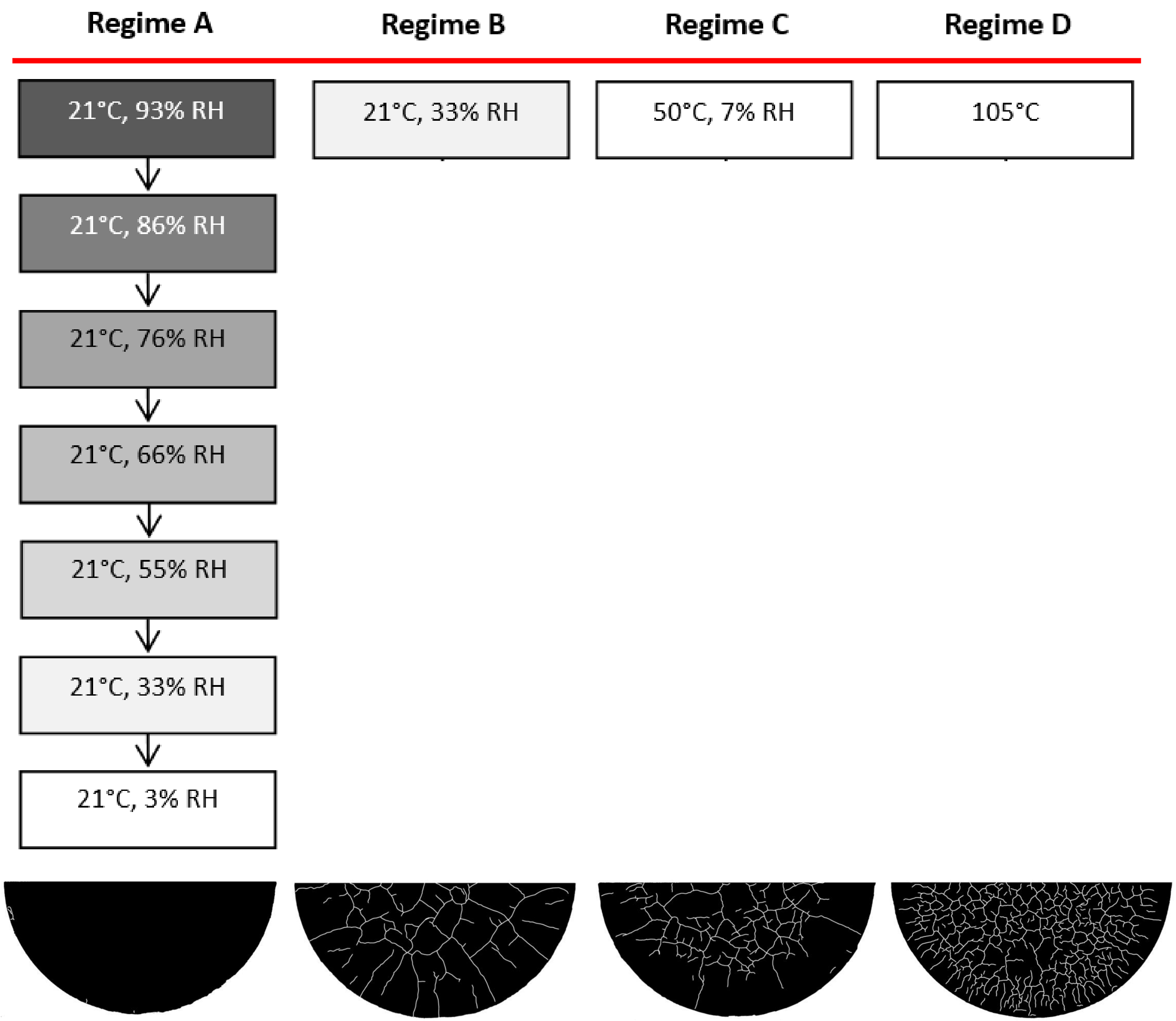
Variable moisture content!

- Transport increases with severity of drying:
  - ***D*: 2 to 18×**
  - ***k* : 3 to 25×**
- Effect is greater than w/b ratio, curing age, binder type

- Possible reasons:
  - Microcracks?
  - Drying induced changes to C-S-H?
  - Pore coarsening?

- Moisture content has huge influence on transport…

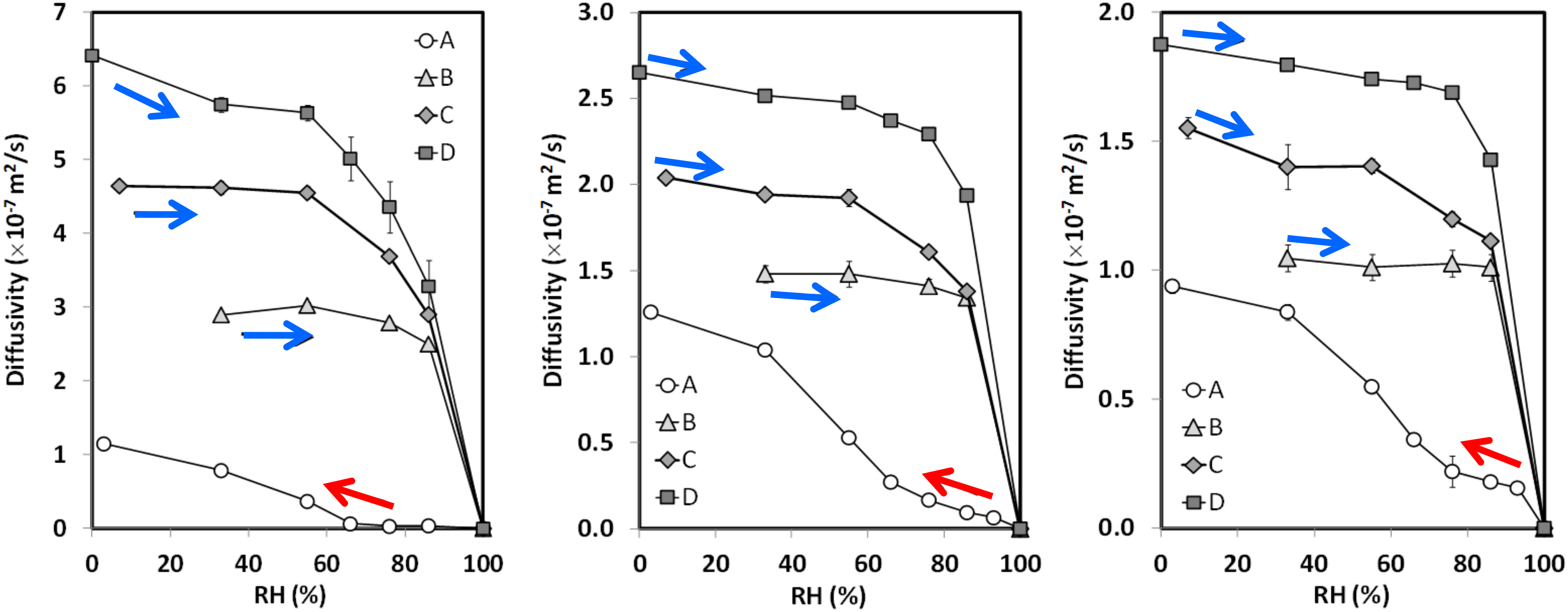
Drying + re-wetting



- Drying to induce varying degrees of microcracking
- Re-wetting via step wise increasing RH at 21ºC
- “Equilibrium” = mass loss < 0.01% per day
- Measure transport property at every conditioning step…

Effect of drying-wetting on transport

Arrows indicate progress of drying / wetting



a) P 0.5 – 3d

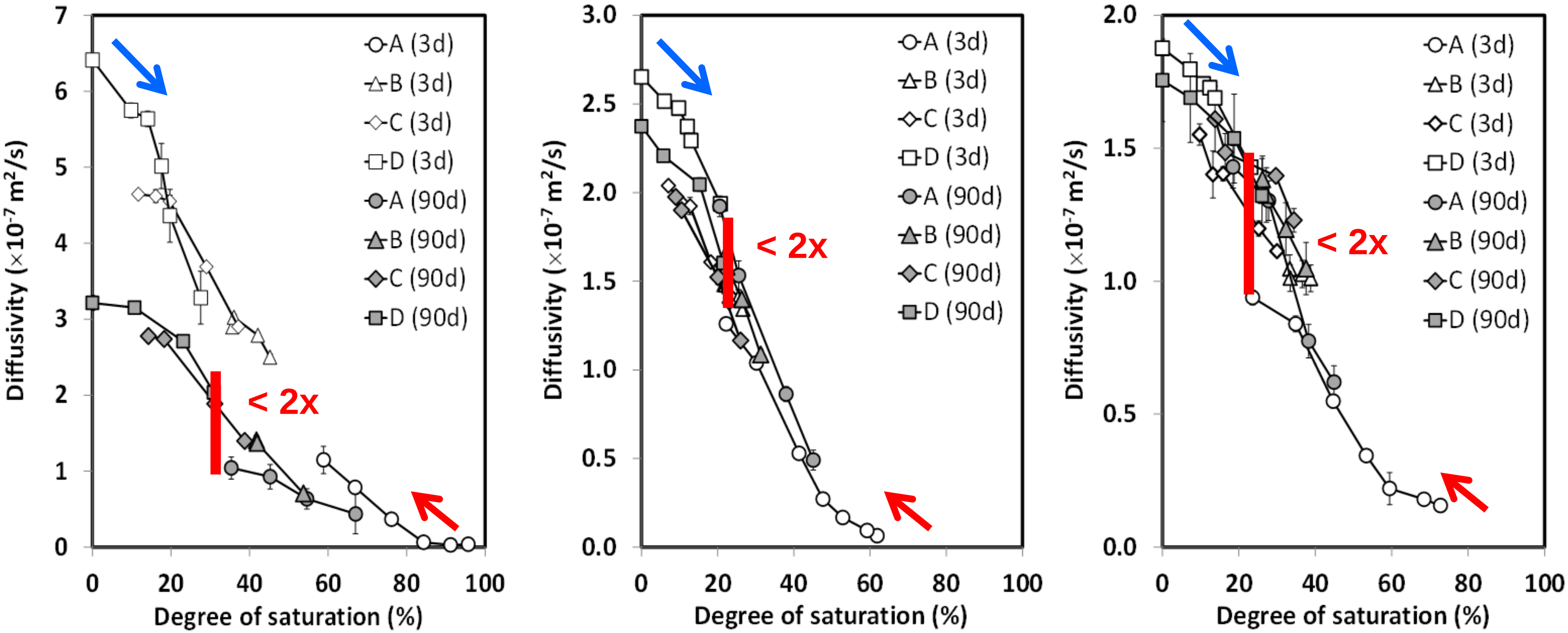
b) M 0.5 – 3d

c) C 0.5 – 3d

- Drying induces a huge change in transport properties.
- Water is removed quicker on drying, gained slower on re-wetting.
- Significant moisture and transport hysteresis over the entire RH range.
- Samples with SCMs show lower transport than CEM I at similar porosities.

Diffusivity vs. degree of saturation

Arrows indicate progress of drying / wetting

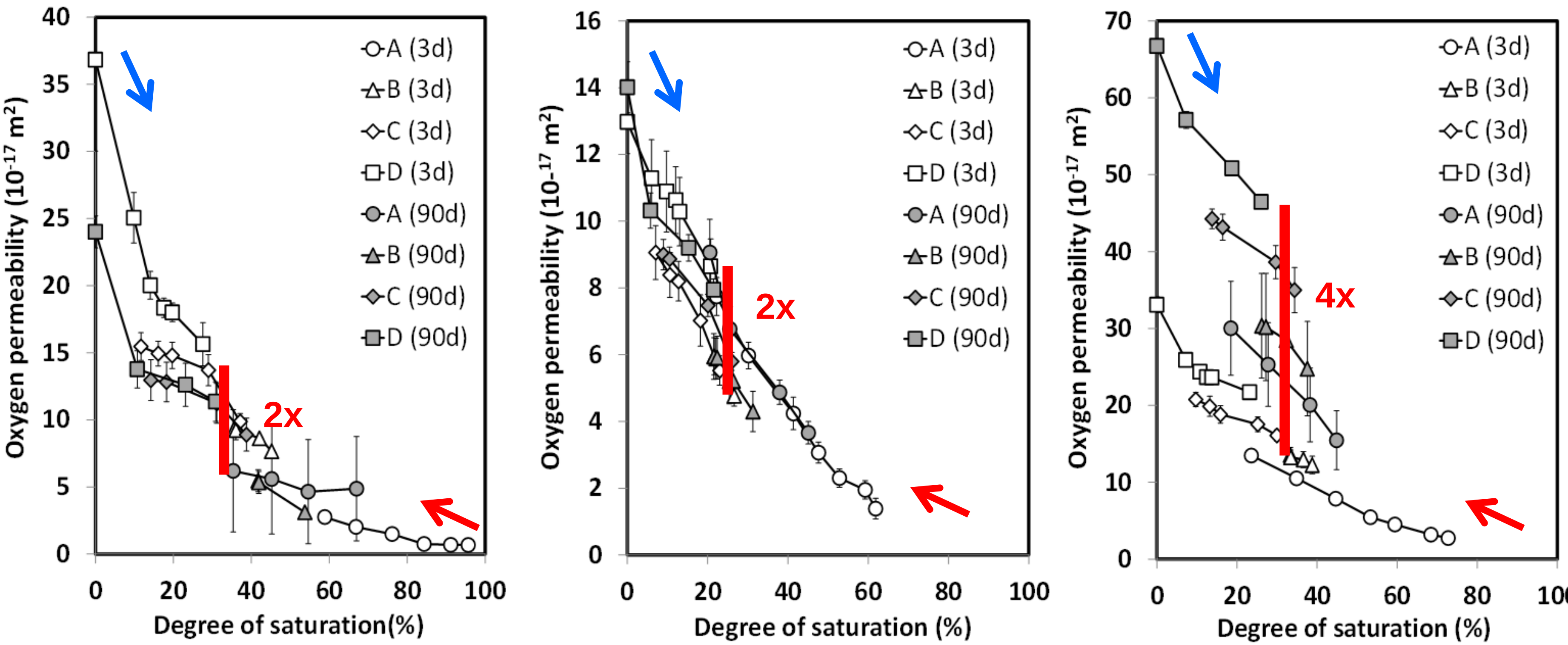


a) P 0.5 (3d & 90d) b) M 0.5 (3d & 90d) c) C 0.5 (3d & 90d)

- Comparison at equal saturation degree isolates the effect of moisture
- Difference between drying and wetting cycles decreased significantly
- "Residual effect" of drying-induced damages (microcracking, pore coarsening, C-S-H collapse etc) on diffusivity is ~ < **2x**

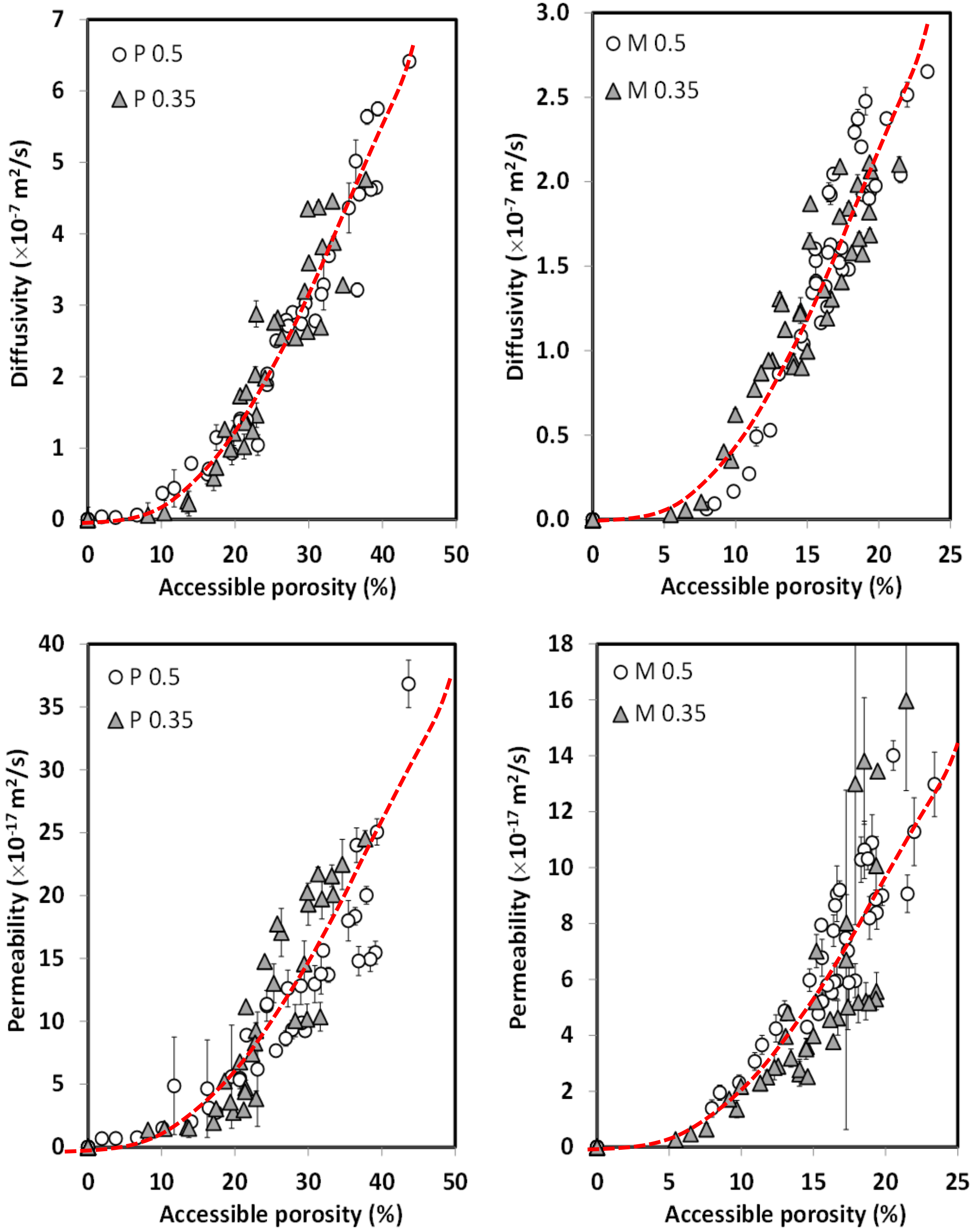
Permeability vs. degree of saturation

Arrows indicate progress of drying / wetting



a) P 0.5 (3d & 90d) b) M 0.5 (3d & 90d) c) C 0.5 (3d & 90d)

- Comparison at equal saturation degree isolates the effect of moisture
- Difference between drying and wetting cycles decreased significantly
- "Residual effect" of drying-induced damages (microcracking, pore coarsening, C-S-H collapse etc) on permeability is ~ **2-4x**



a) Paste   b) Mortar

<u>Samples</u>

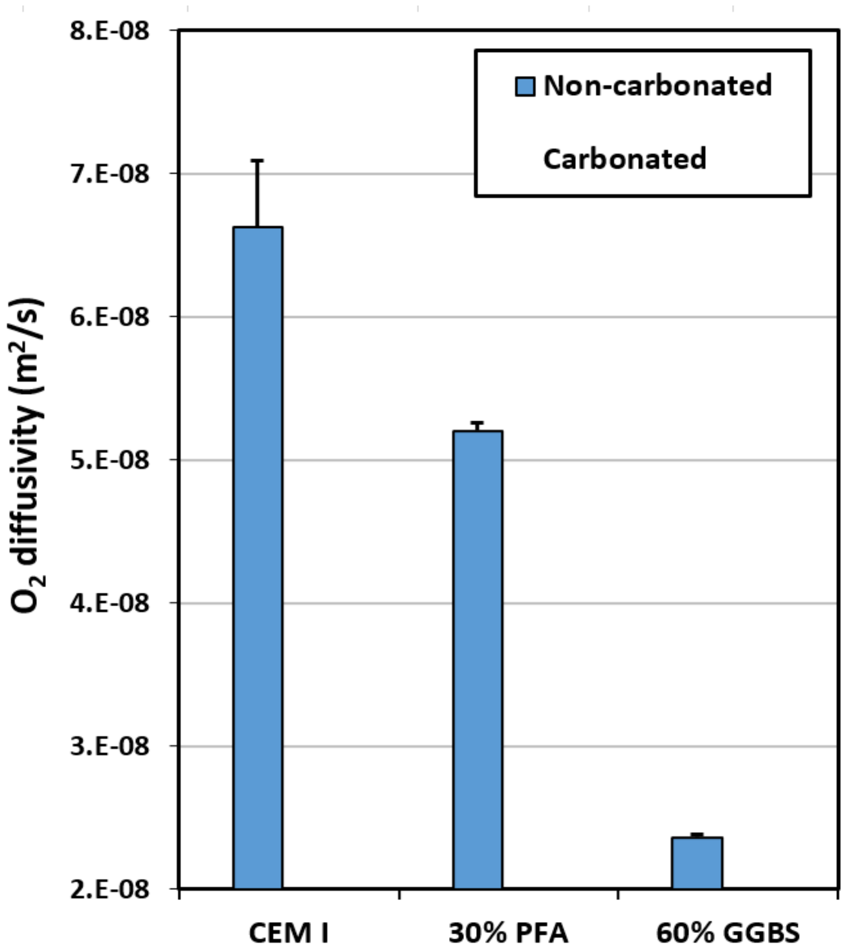
- CEM I
- CEM I + 10%SF
- CEM I + 70%GGBS
- w/b: 0.35 - 0.50
- Paste, mortar & conc.
- Aggregate vol.: 0-68%
- Sealed curing: 3-90d
- Conditioning:
  - 105C
  - 50C, 7%RH
  - 21C, 33%RH
  - 21C, 55%RH
  - 21C, 76%RH
  - 21C, 86% RH

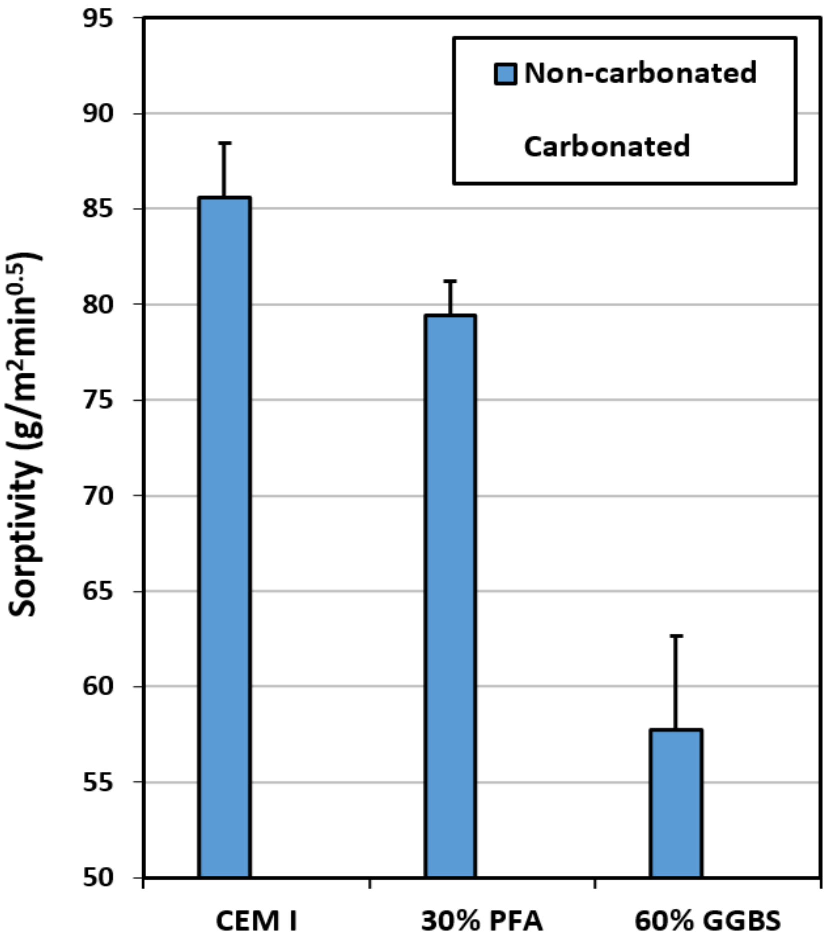
How does carbonation changes transport properties and these correlations?

Impact of carbonation on transport

(M. Bertin, V. Baroghel-Bouny, B. Huet, H. Wong & O. Metalssi)

- Blended concretes (w/b 0.57-0.61), 6 months water cured
- Discs (100∅ × 50 mm), conditioned at 45°C, 63% RH
- 3% CO_2, 20°C, 63% RH for 6 months





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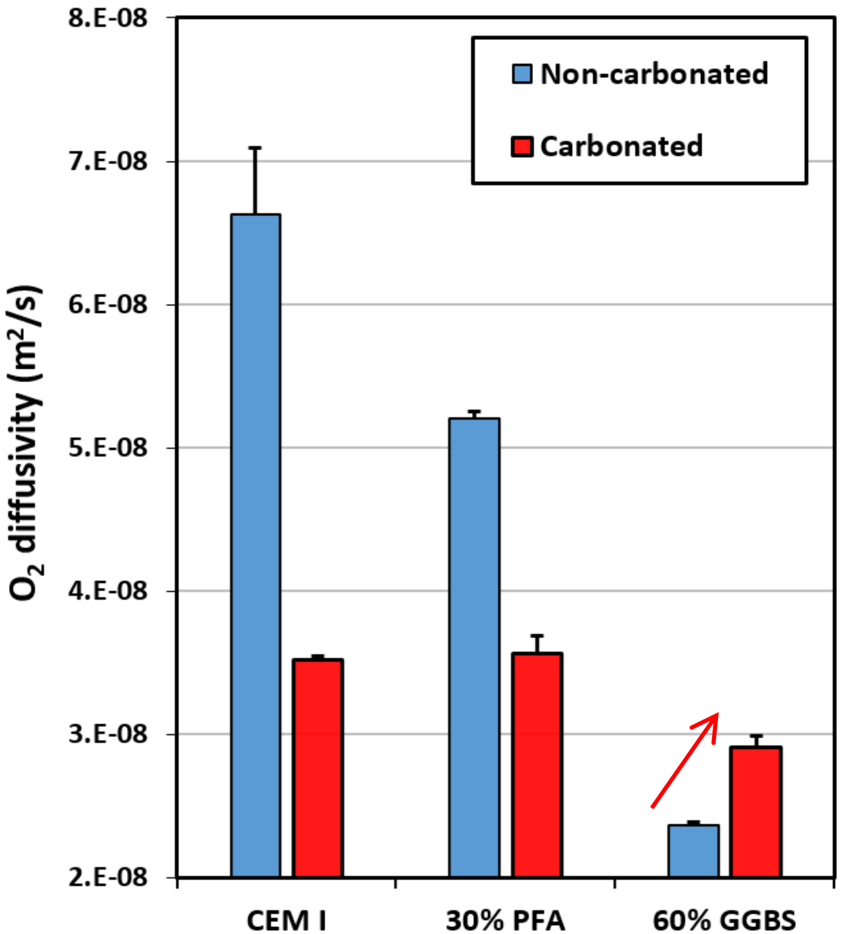
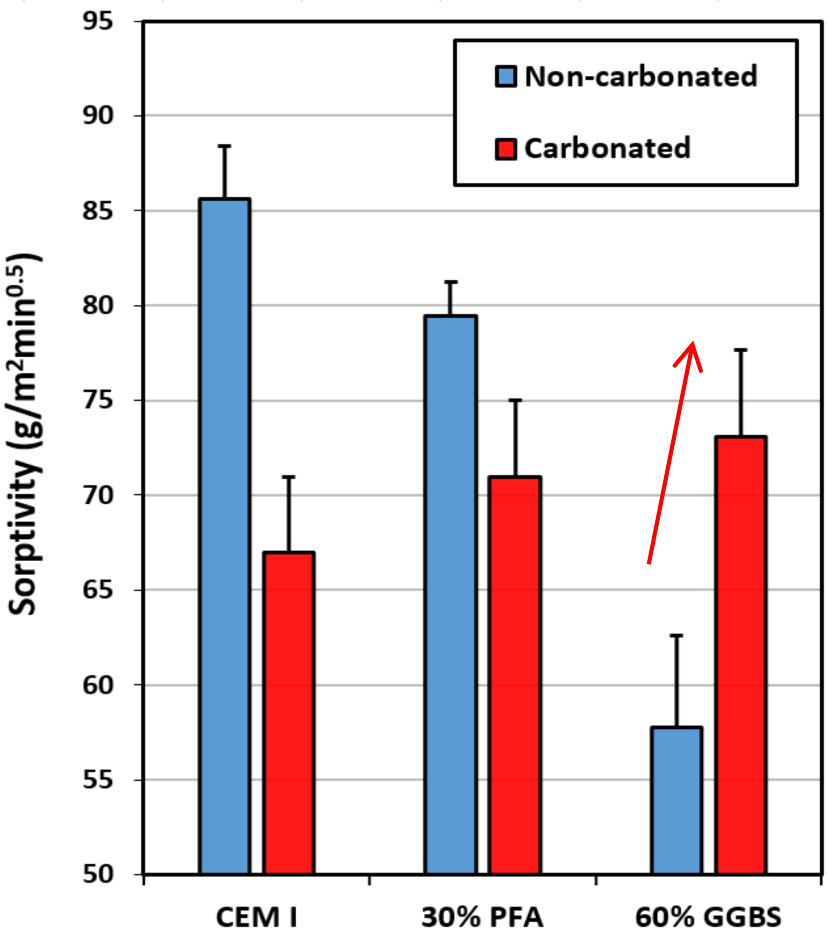
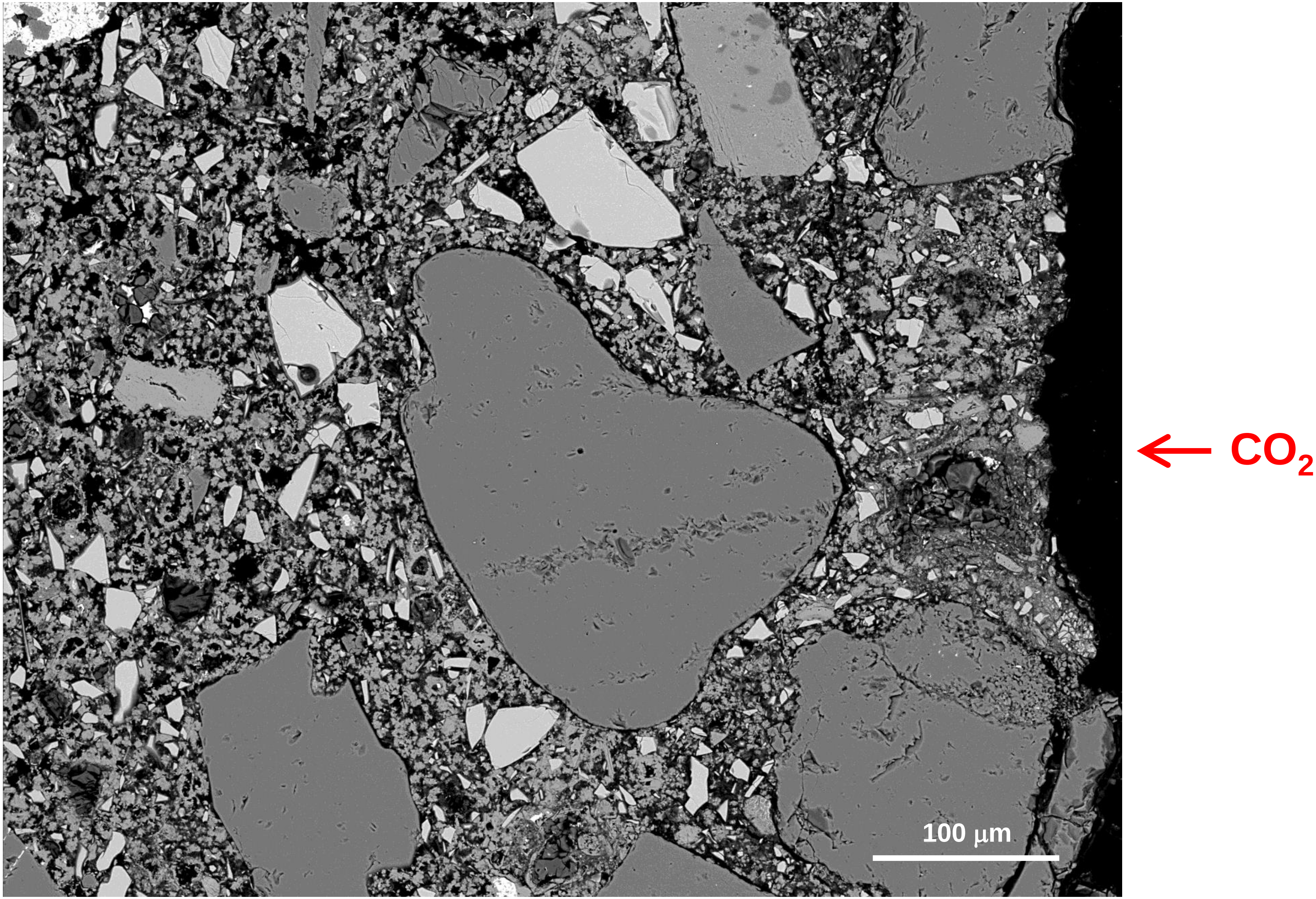
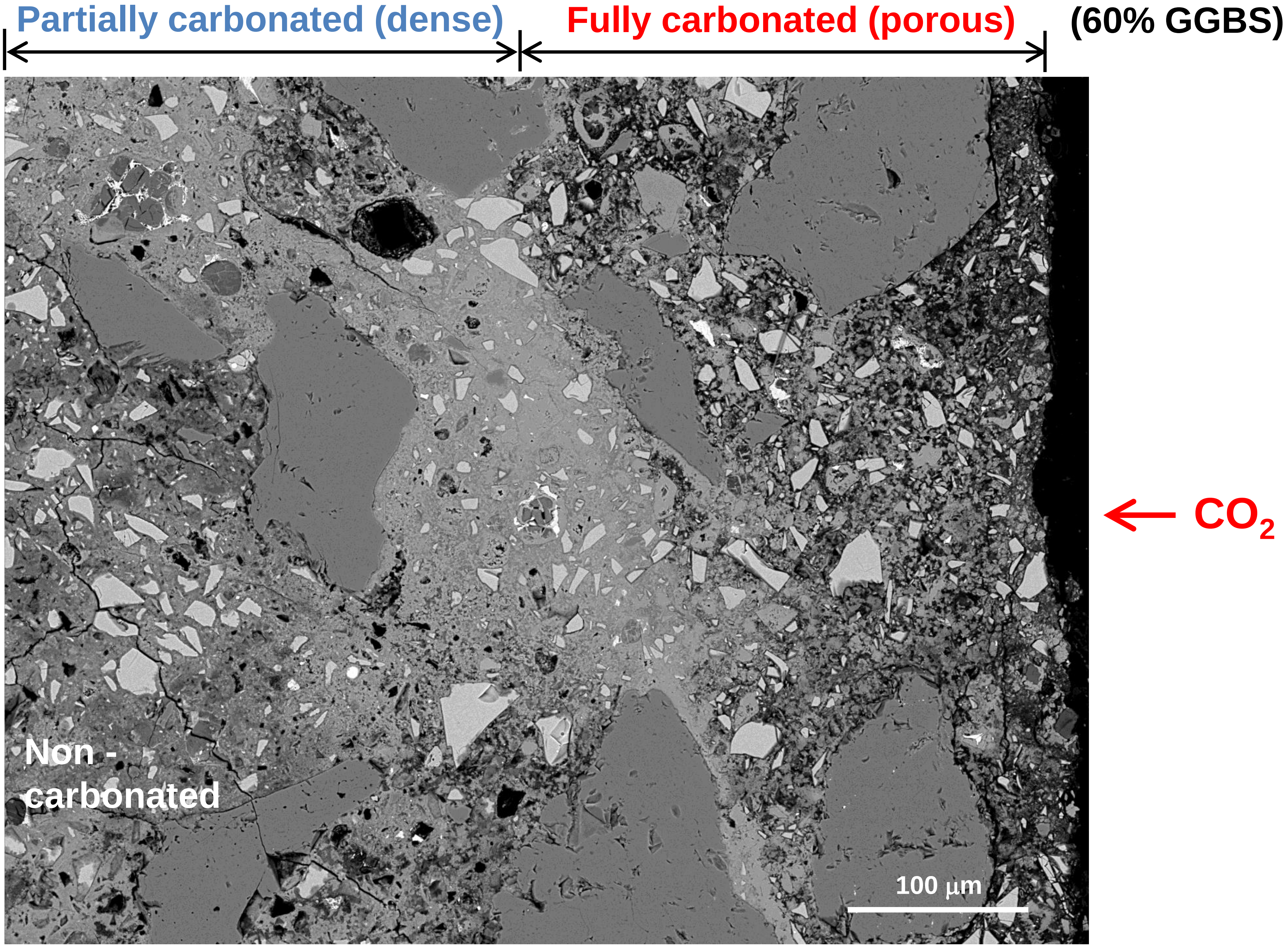




Carbonation depth, mm (%)	**9.5 (38%)**	**14.2 (59%)**	**15.9 (64%)**

Microstructure of fully carbonated concrete (60% GGBS)





Partially carbonated (dense)
Fully carbonated (porous)
(60% GGBS)
CO2
Non -
carbonated
100 μm

Impact of carbonation on permeability

(M. Bertin, V. Baroghel-Bouny, B. Huet, H. Wong & O. Metalssi)

- Concrete (w/b 0.57-0.61), 6 months water cured
- Discs (100∅ × 50 mm), conditioned at 45°C, 63% RH
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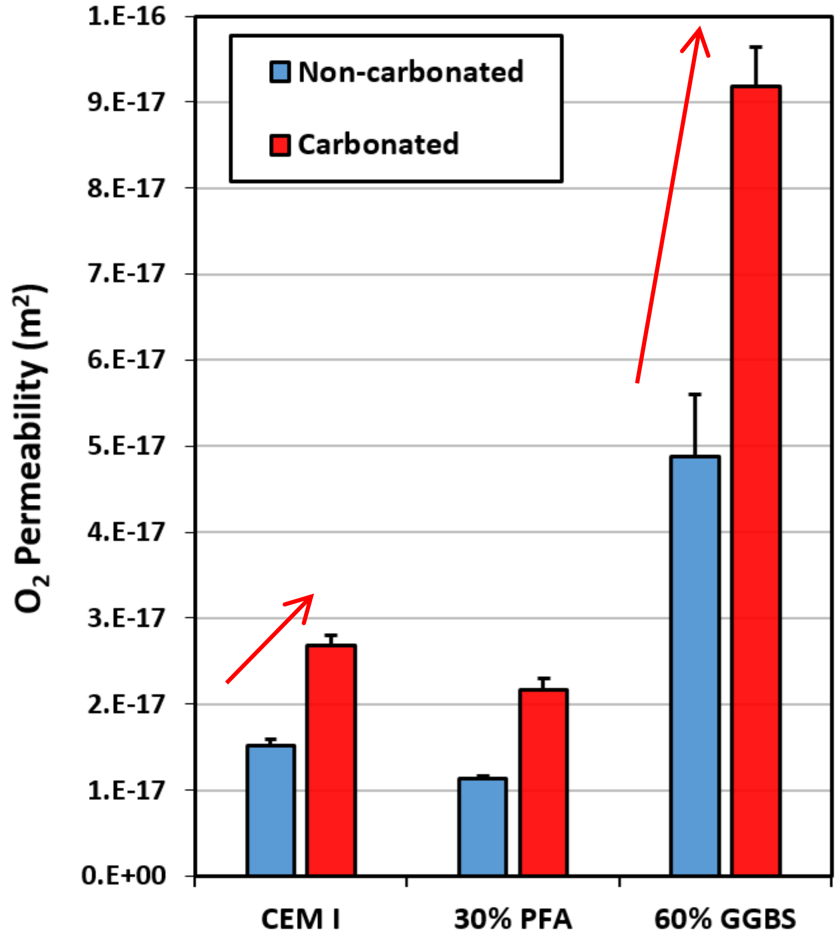
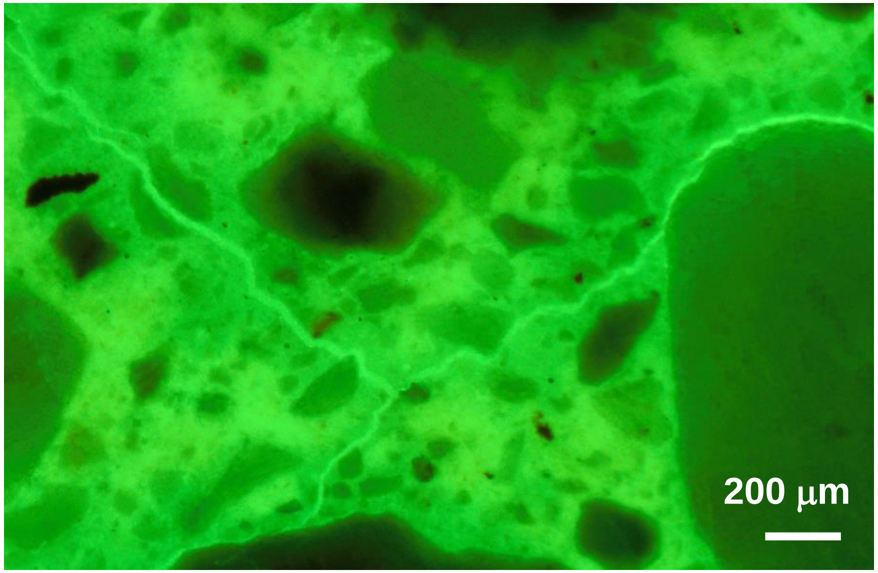


Carbonation depth, mm (%)	9.5 (38%)	14.2 (59%)	15.9 (64%)

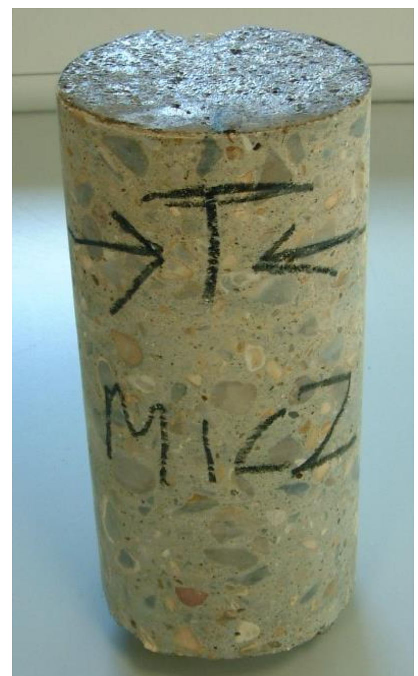


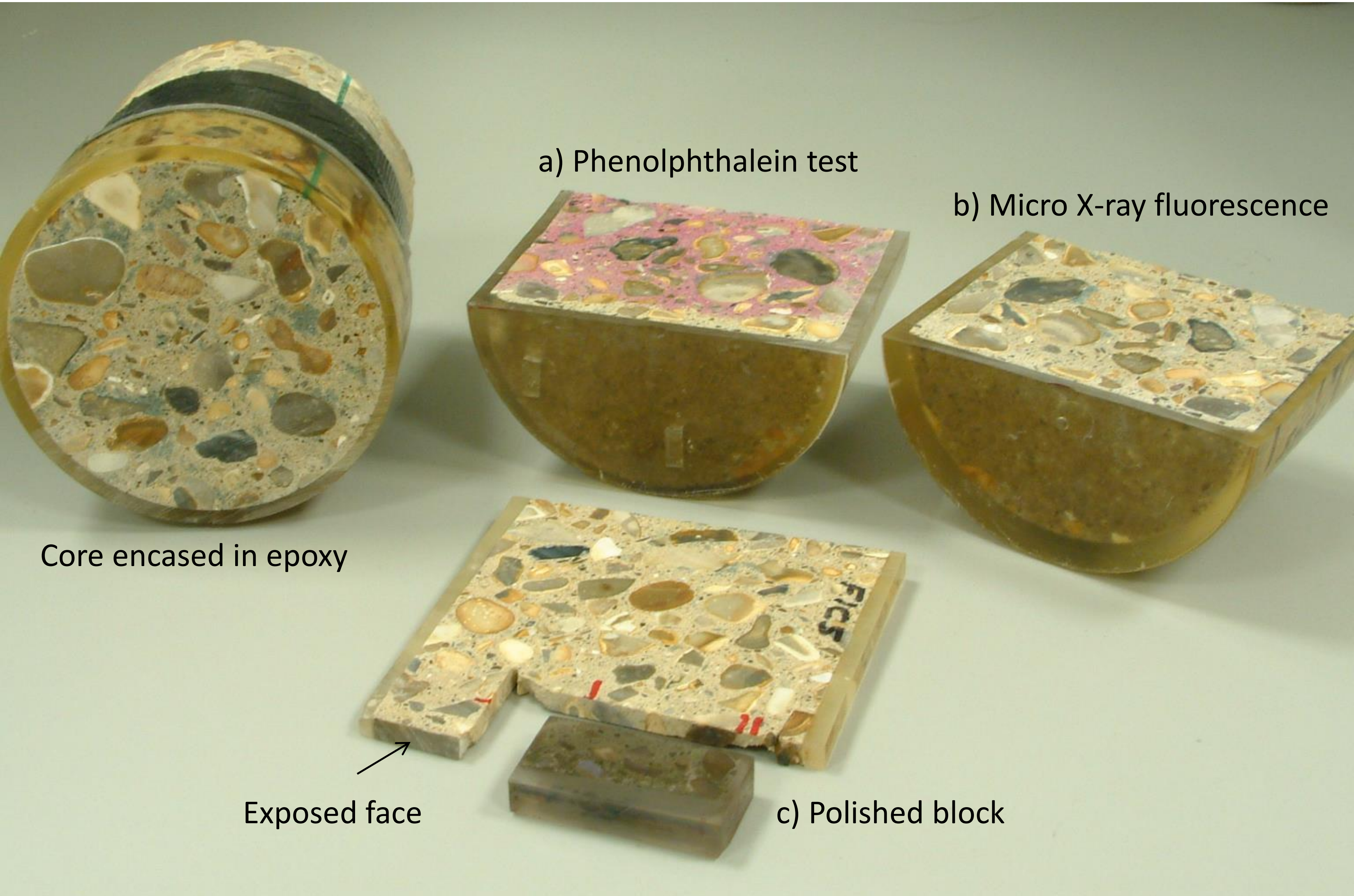
Average width (and total length) of microcracks:

	Non-carbonated	Carbonated
CEM I	6.6 μm (56.6 mm)	18.2 μm (86.3 mm)
30% PFA	5.8 μm (98.2 mm)	9.0 μm (172.5 mm)
60% GGBS	13.8 μm (53.9 mm)	22.3 μm (378.6 mm)

Case study: Composting facility

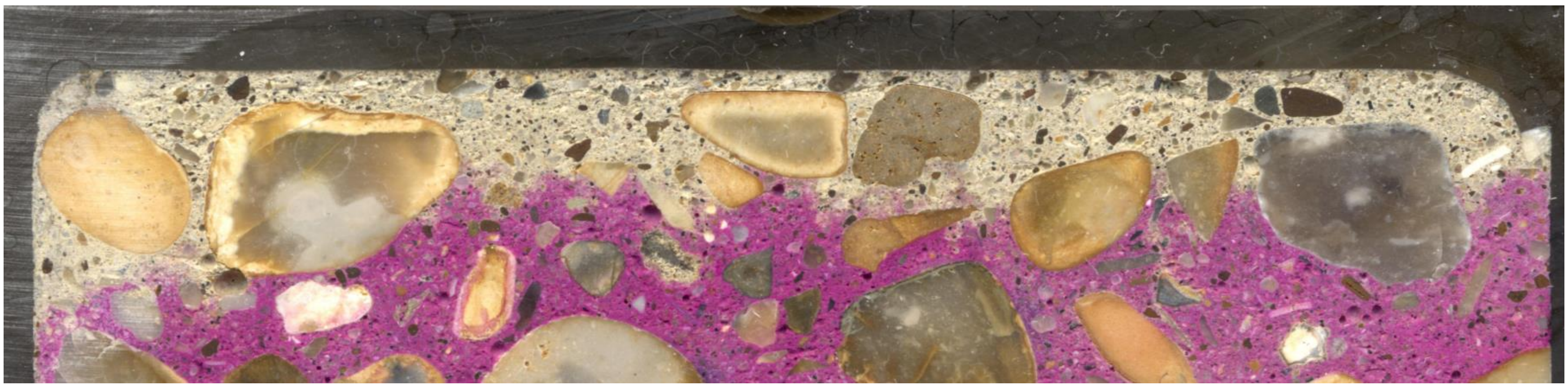
- 25 years design life
- Deteriorated after < 3 years operation (softening & erosion)
- w/b 0.5 concrete
- 60% GGBS
- 15 cores:
  - Internal wall (exposed to compost)
  - External wall (not exposed to compost)

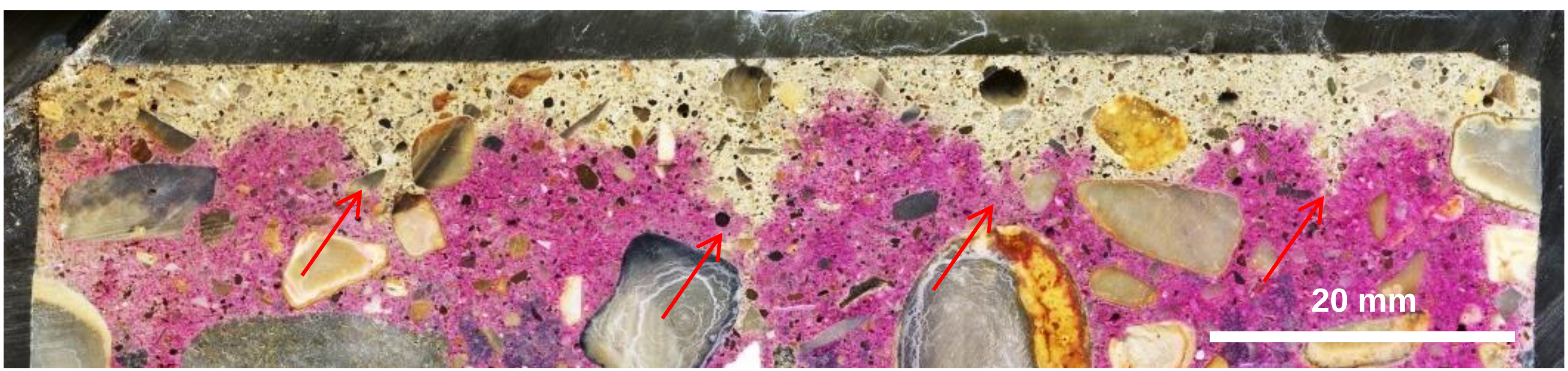




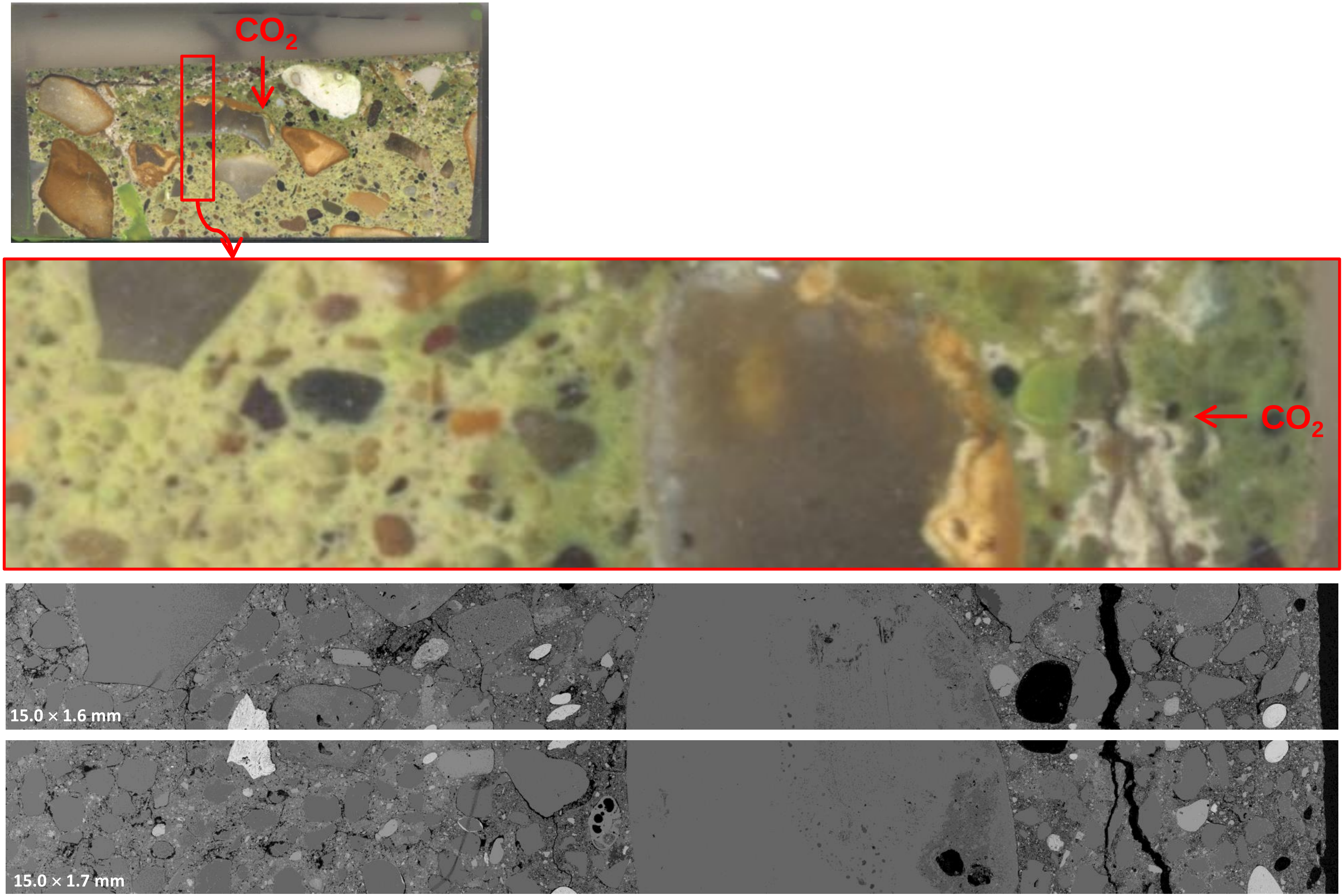
Samples for a) phenolphthalein test, b) micro X-ray fluorescence, and c) polished block for scanning electron microscopy.

Location	Carbonation depths (mm)		
	Average	Minimum	Maximum
External wall	5 - 10	3 - 6	11 – 14
Internal wall (compost)	2 - 9	1 - 3	4 - 16





Typical microstructure of external wall (control).
Paste near the exposed surface is highly porous due to carbonation.



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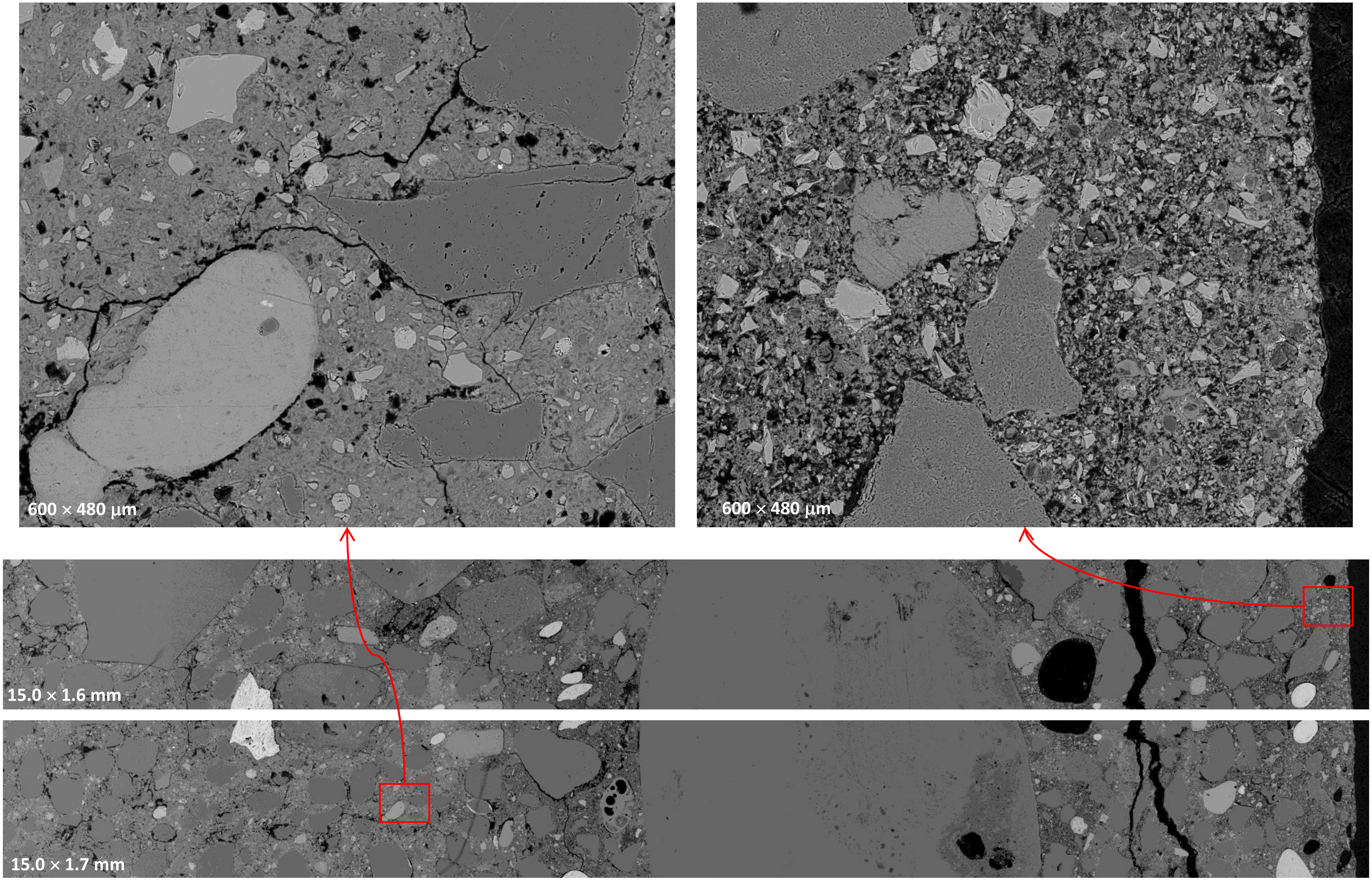
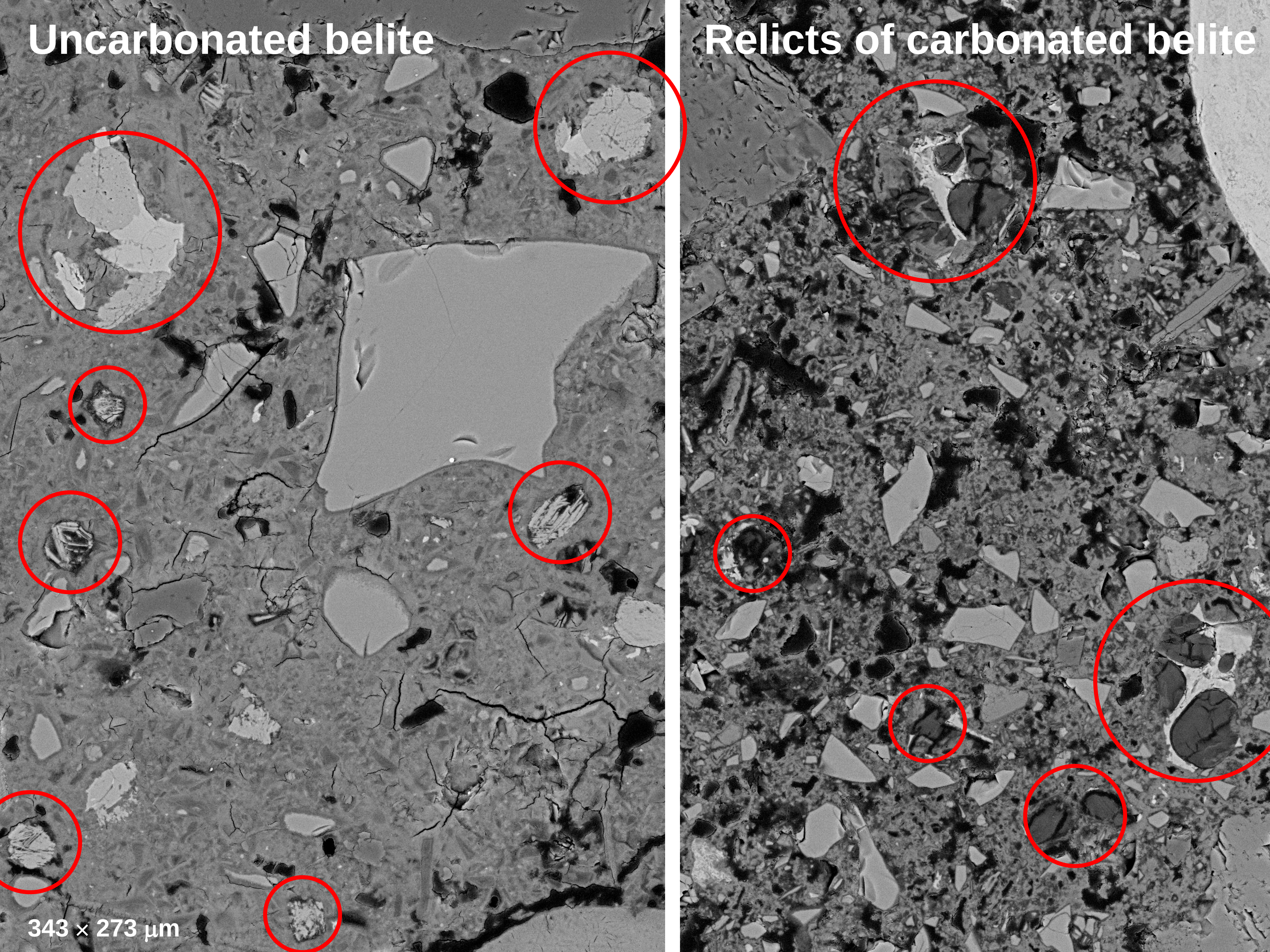
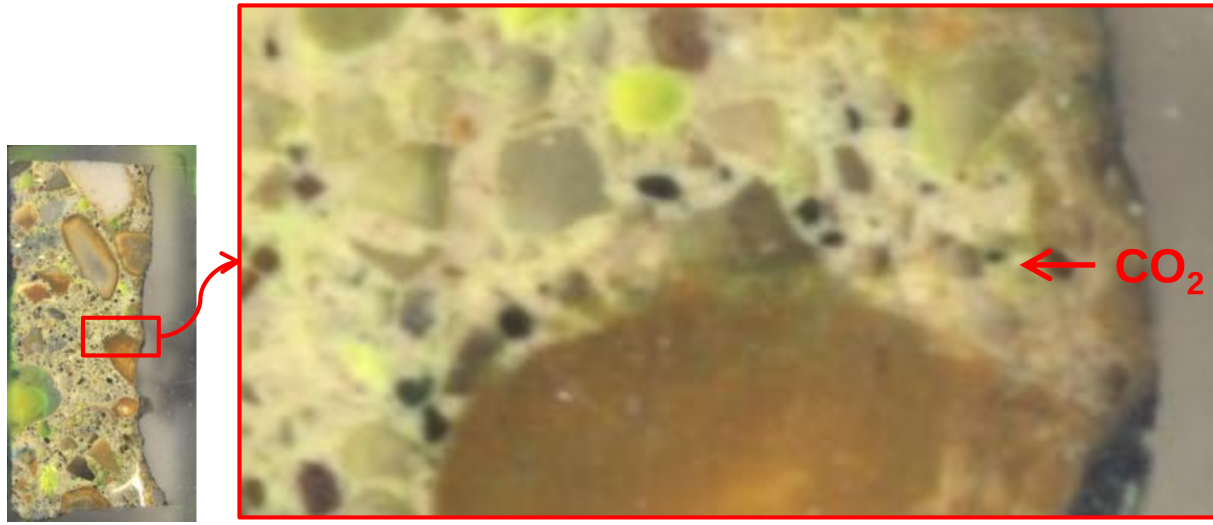
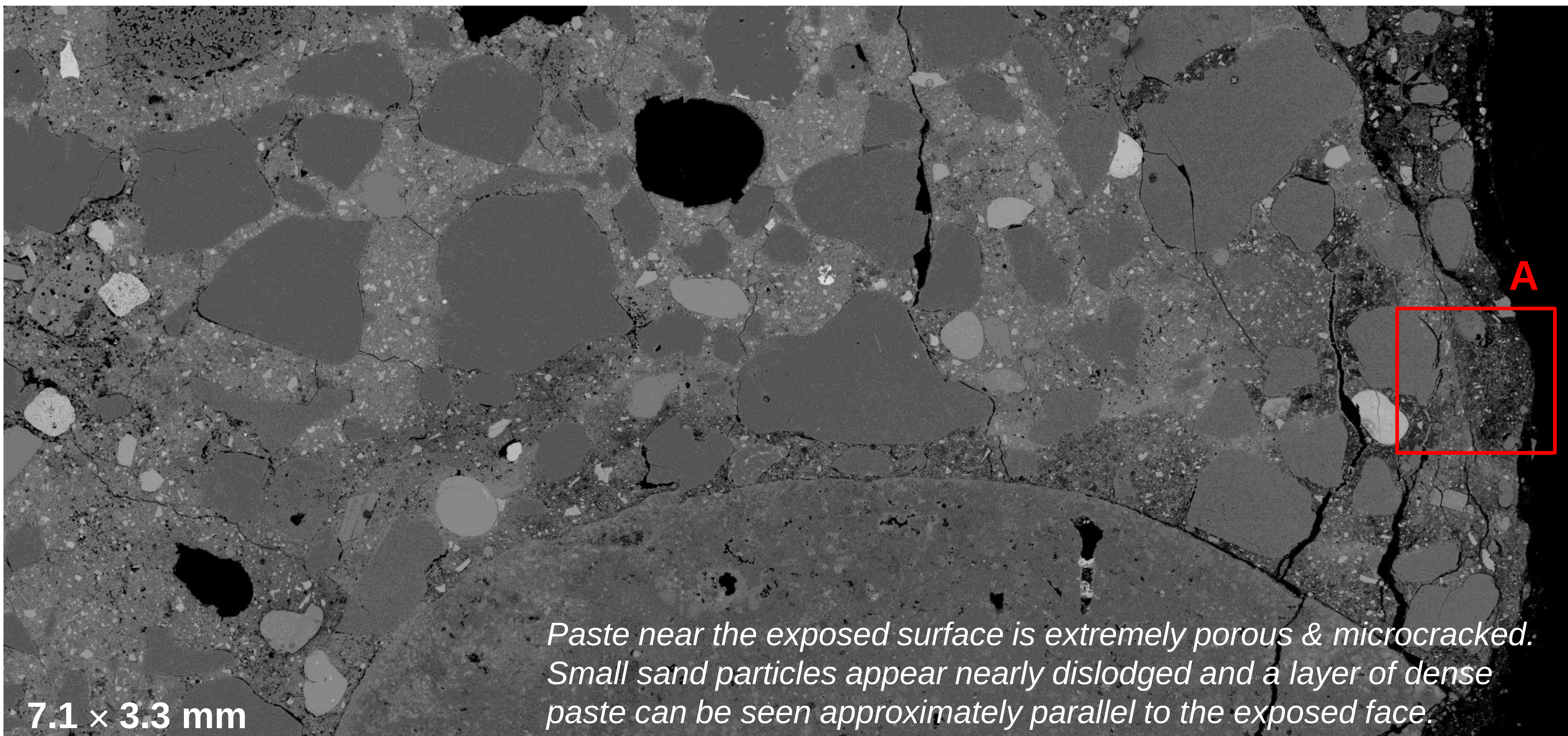


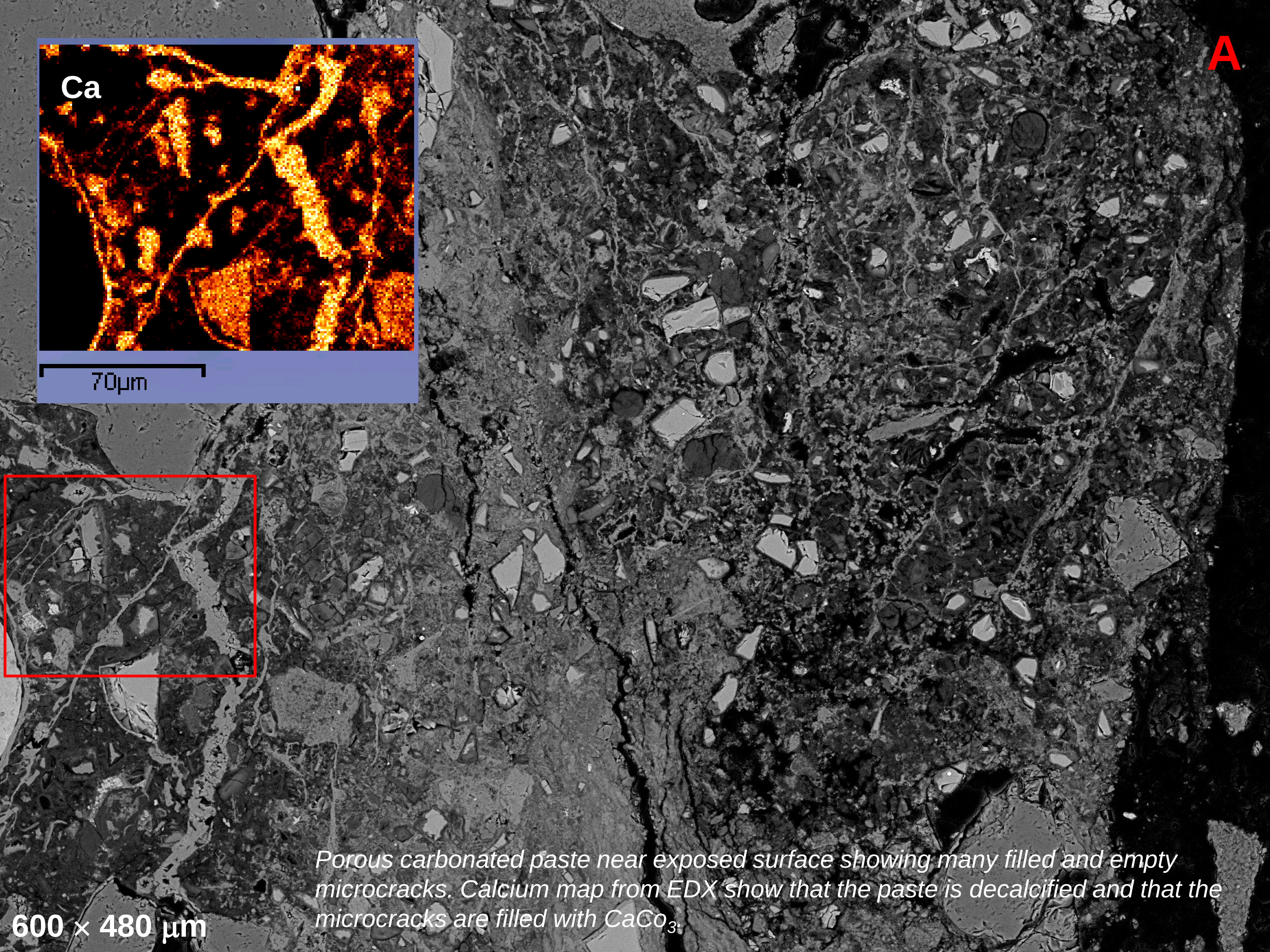


Uncarbonated belite
Relicts of carbonated belite
343 × 273 µm

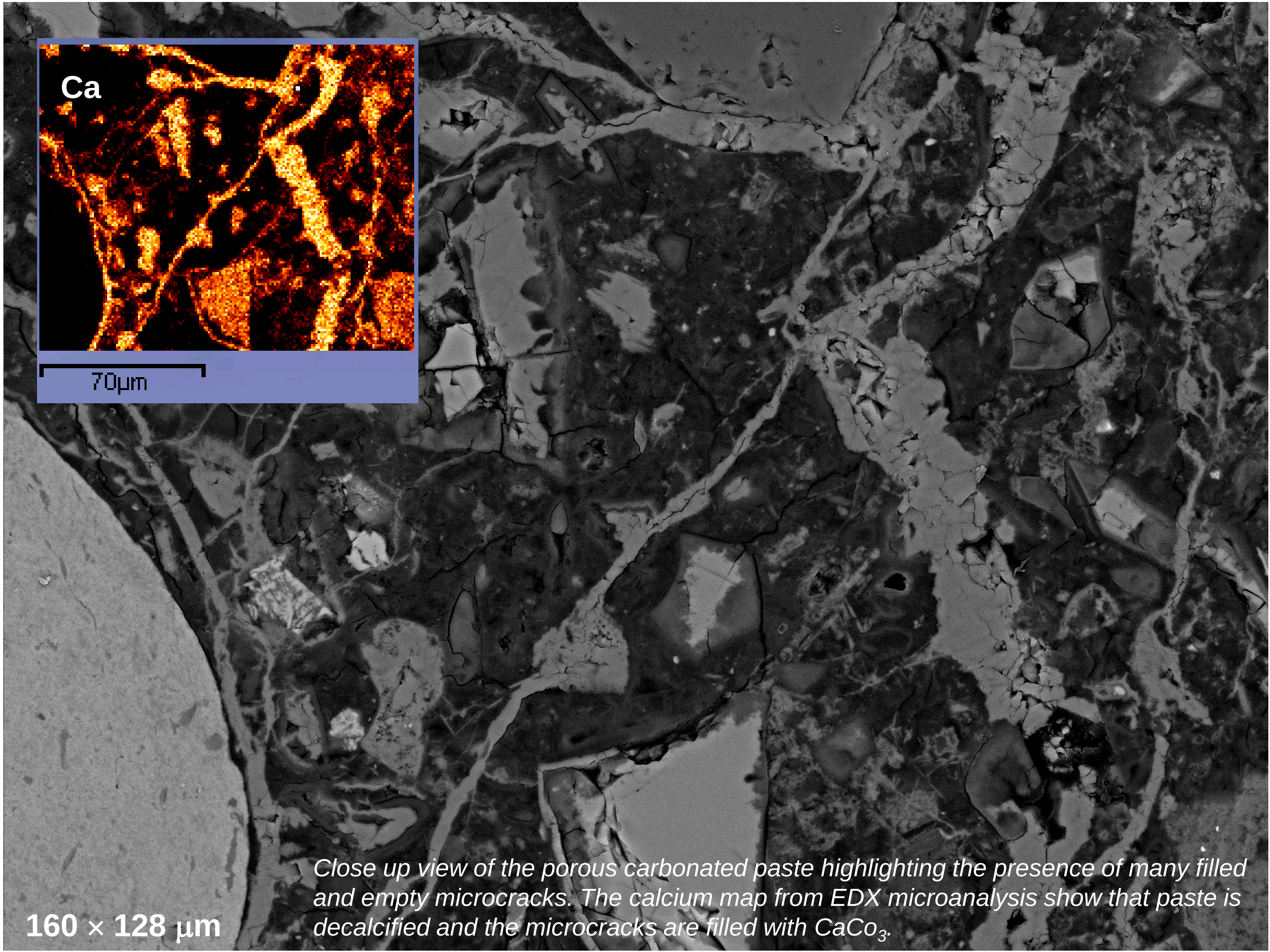
Typical microstructure of internal wall (compost). Paste near the exposed surface is highly porous and cracked



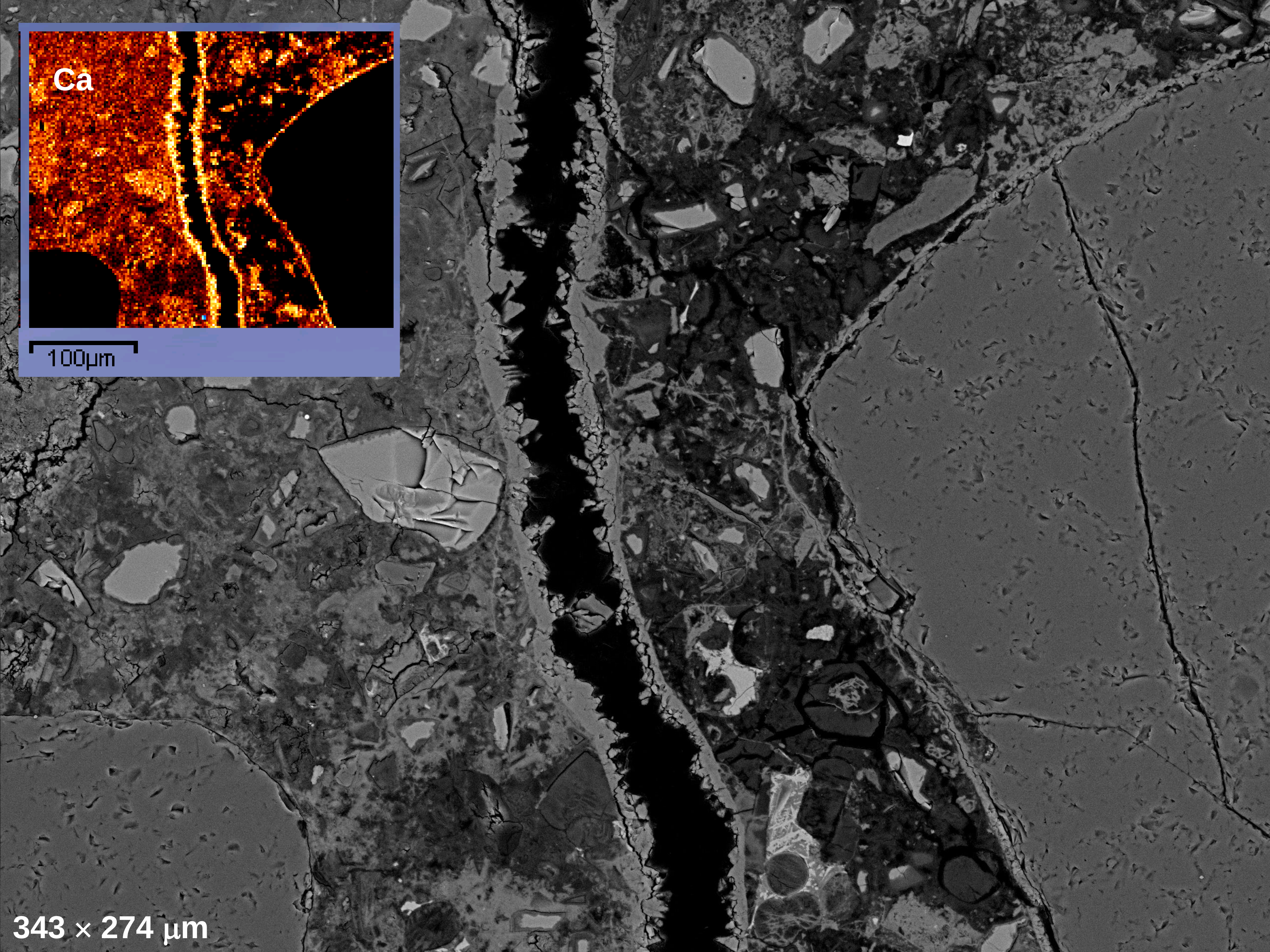




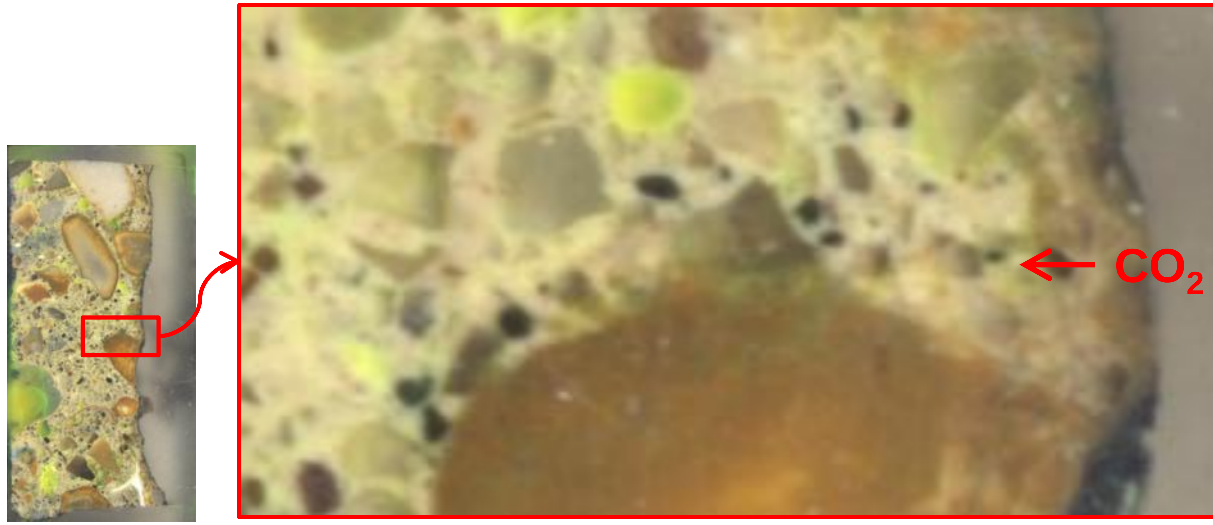
Porous carbonated paste near exposed surface showing many filled and empty microcracks. Calcium map from EDX show that the paste is decalcified and that the microcracks are filled with $CaCo_3$.

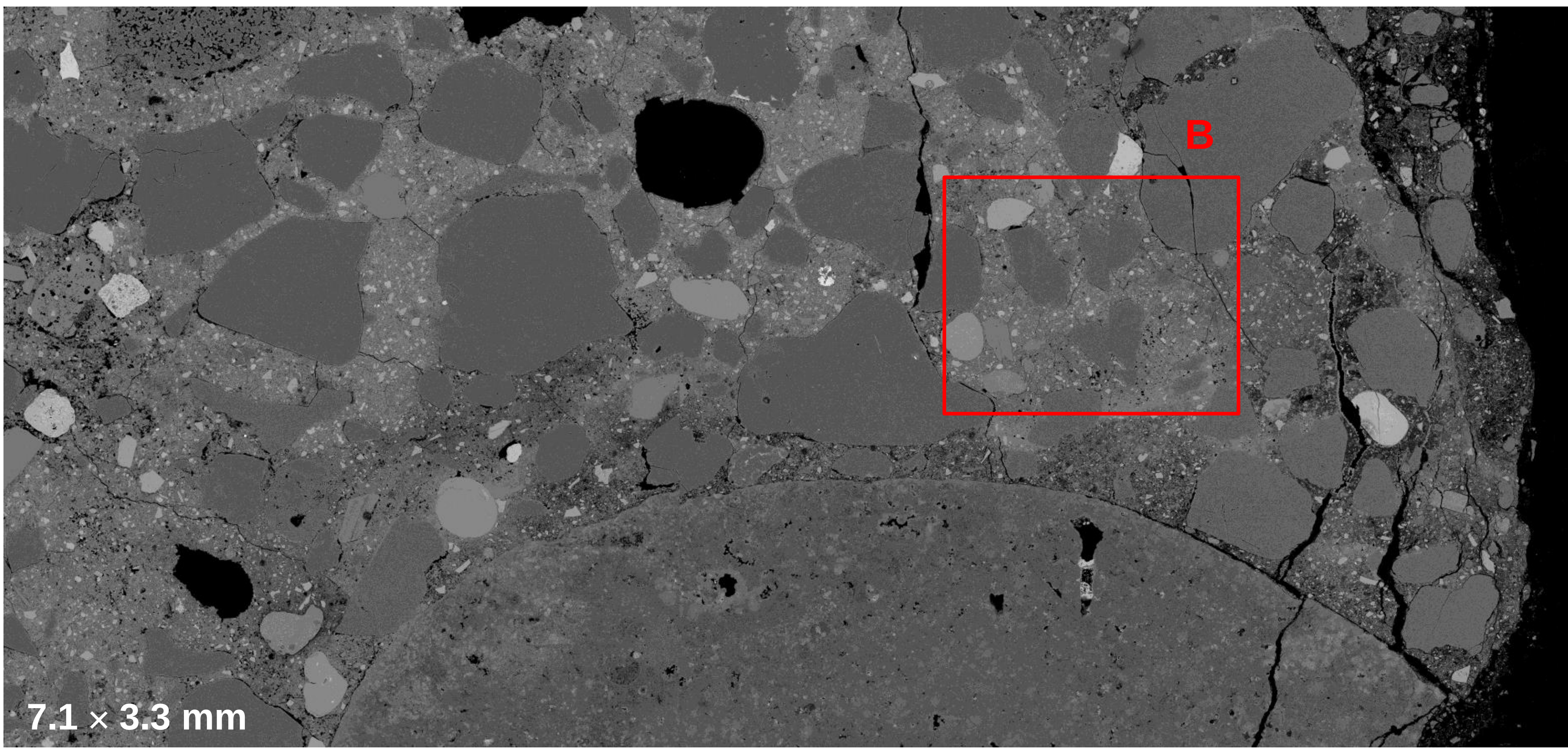


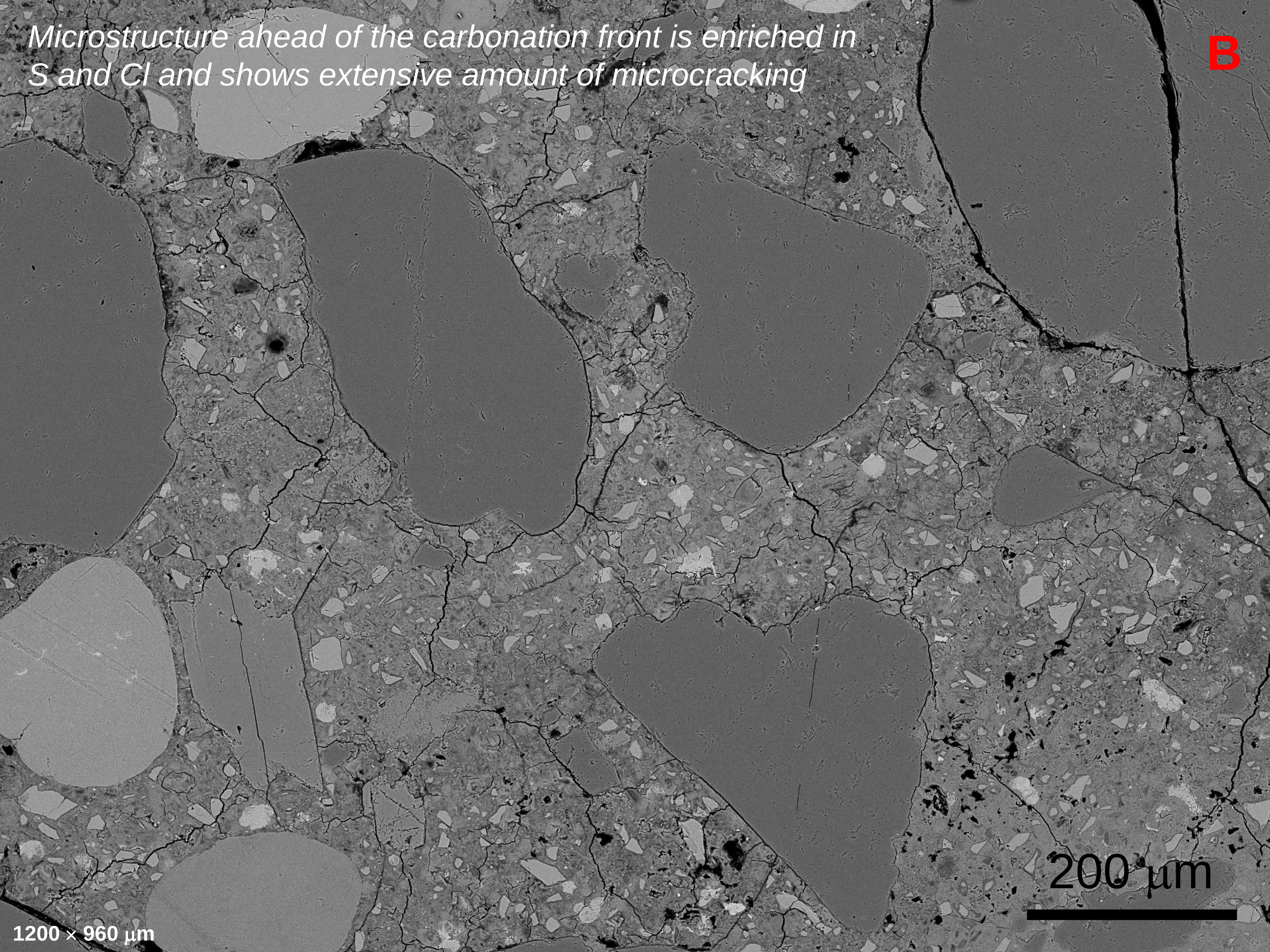
Close up view of the porous carbonated paste highlighting the presence of many filled and empty microcracks. The calcium map from EDX microanalysis show that paste is decalcified and the microcracks are filled with $CaCo_3$.


Ca
100µm
343 × 274 µm

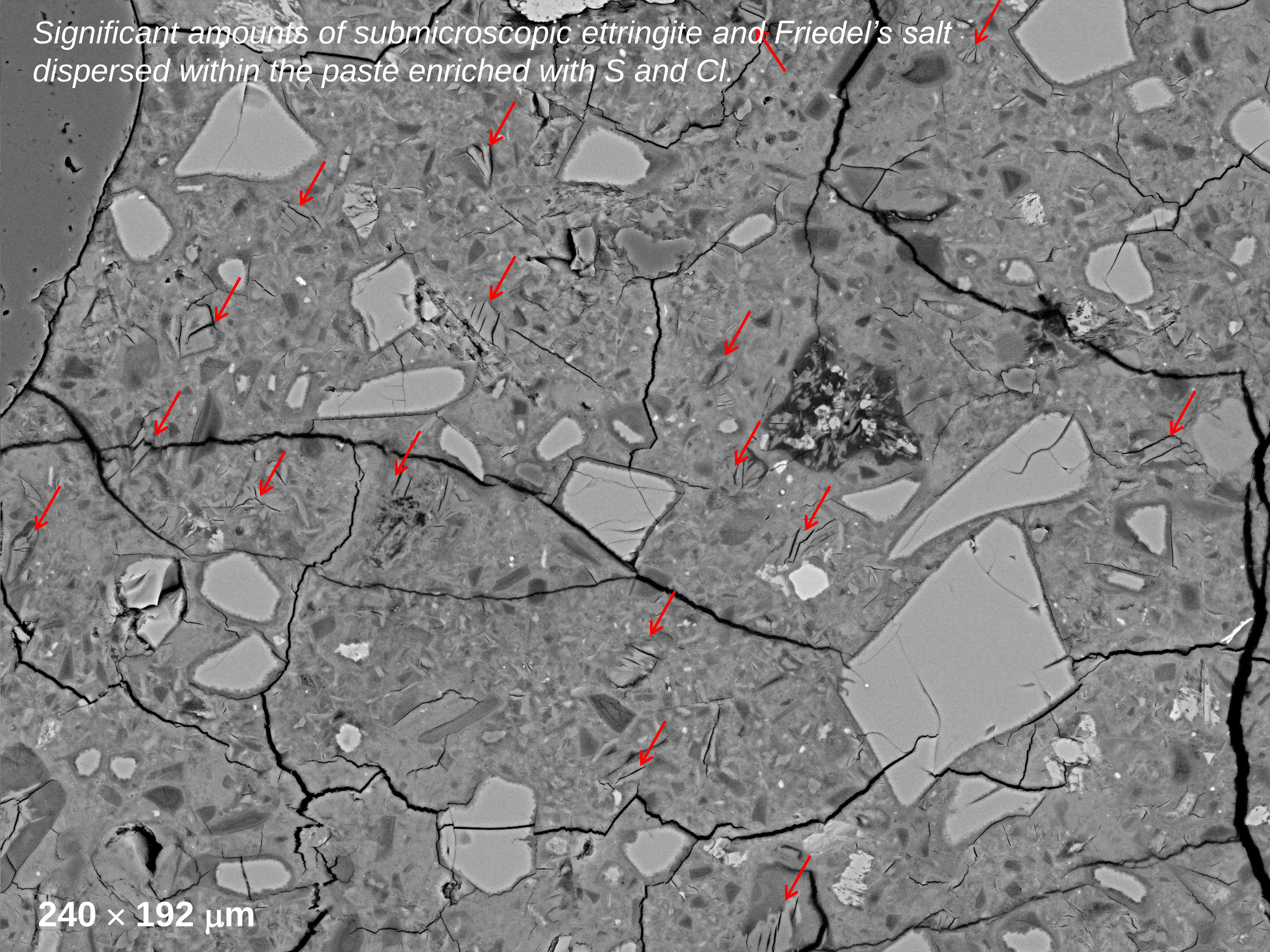
Typical microstructure of internal wall (compost). Paste near the exposed surface is highly porous and cracked



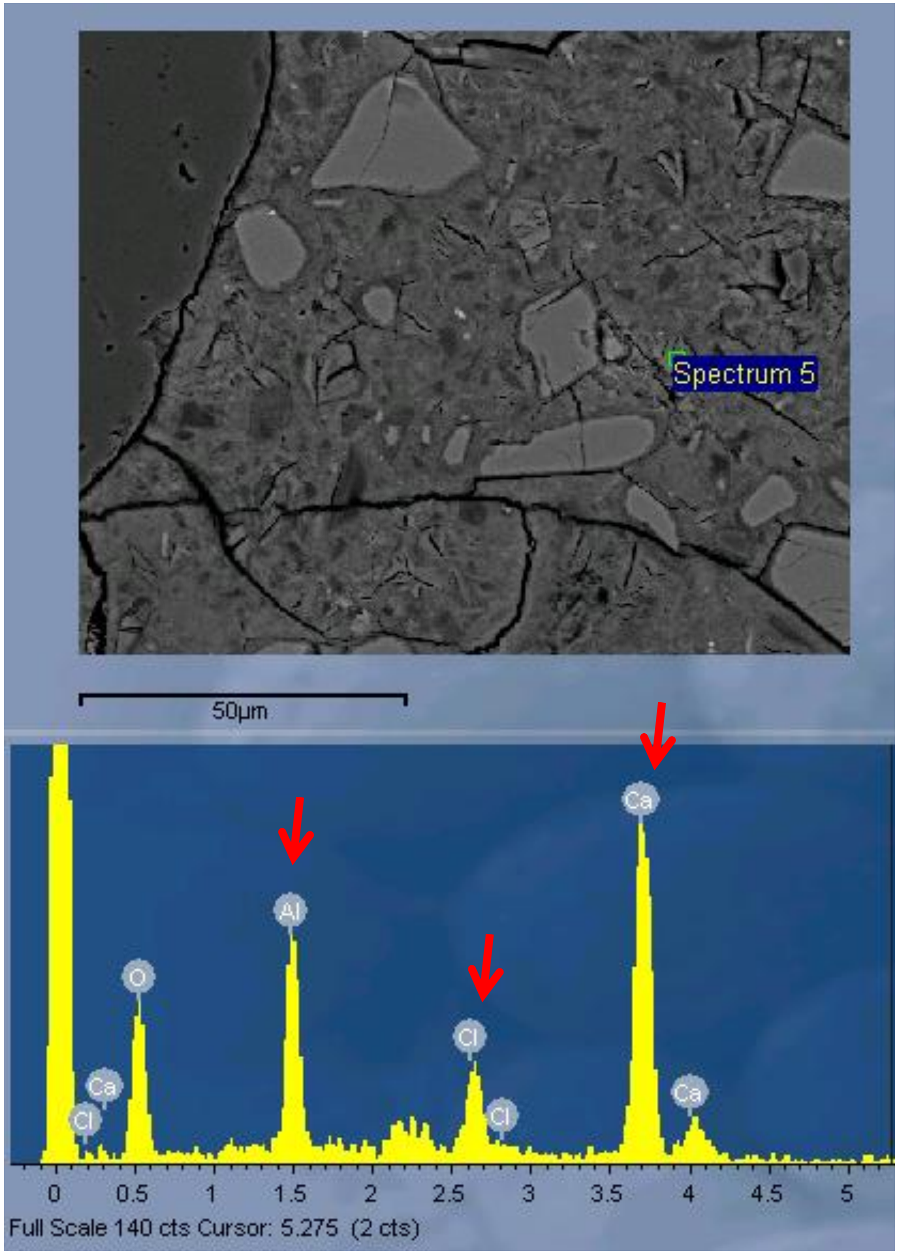




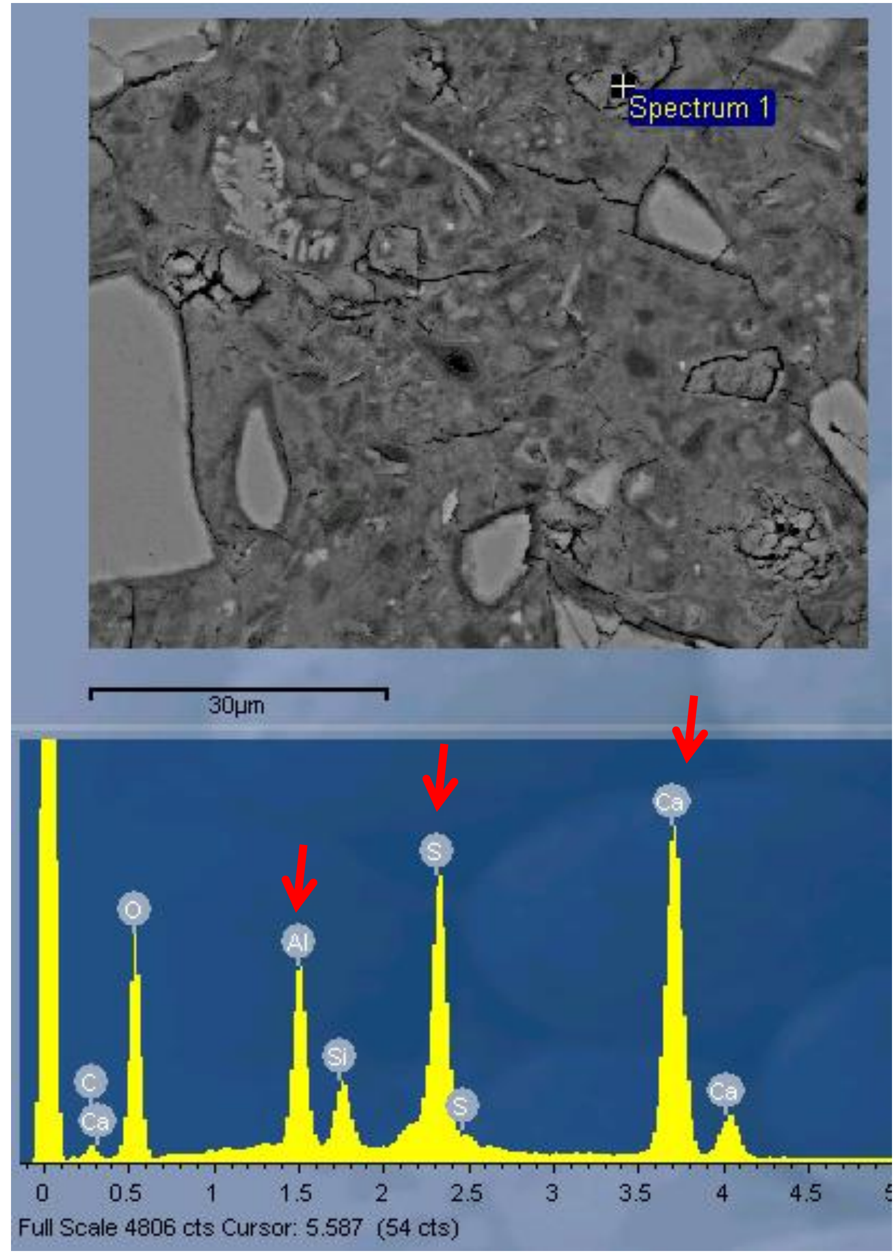
Microstructure ahead of the carbonation front is enriched in S and Cl and shows extensive amount of microcracking
B
200 µm
1200 × 960 µm


Significant amounts of submicroscopic ettringite and Friedel's salt dispersed within the paste enriched with S and Cl.
240 × 192 µm

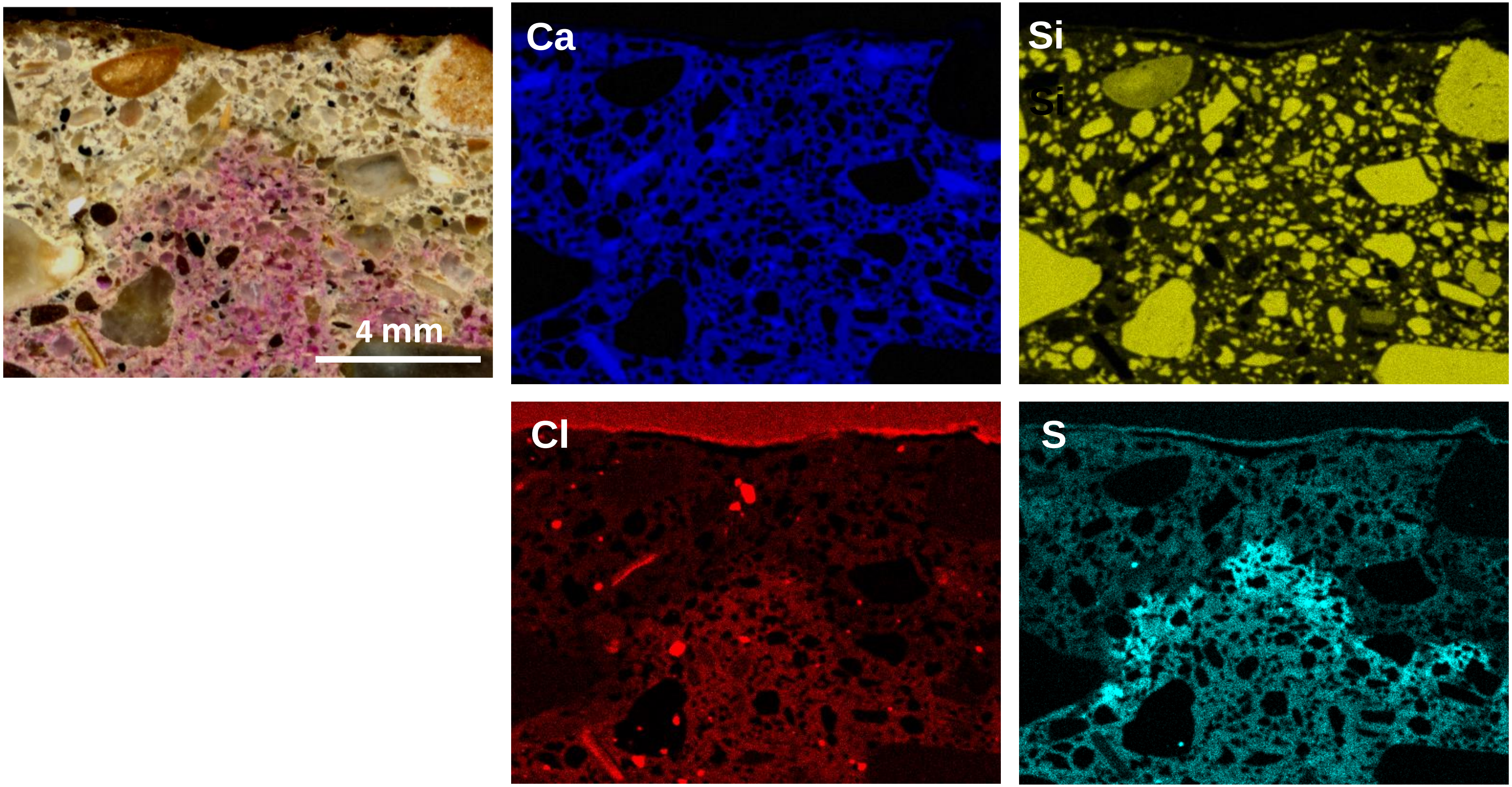
Friedel's salt
($3CaO.Al_2O_3.CaCl_2.10H_2O$)



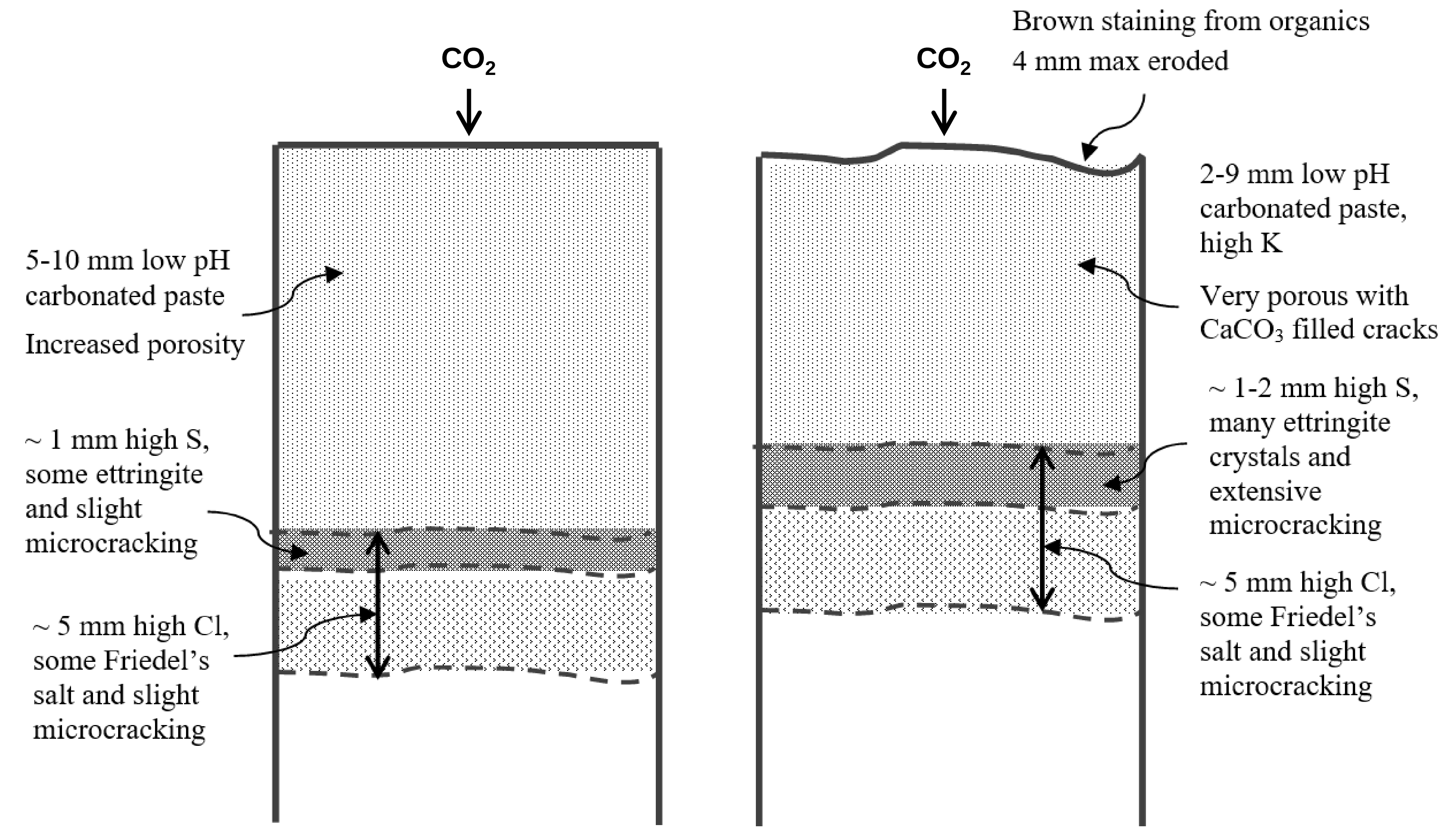
Ettringite
($3CaO.Al_2O_3.3CaSO_4.32H_2O$)



Element mapping shows enrichment of sulphur and chloride ahead of carbonation front, corresponding to where phenolphthalein changes colour.



Schematic & possible mechanism(s)



a) External wall (control) b) Internal wall (compost)

- Impact on reinforcement corrosion?
- How to model this?

Summary & concluding remarks

- Transport under partially saturated conditions is important, but complex.
- Drying induces surface microcracking (< 0.1 mm width, < 10 mm depth).
- Other changes also occur on drying (e.g. C-S-H collapse, pore coarsening, moisture content etc.) → huge effect on transport (*D*: 2-18x, *k*: 3-25x).
- Hysteresis when re-wet, but isolating moisture content → residual effect on transport is small (*D* < 2x, *k* < 4x).
- On re-saturation, residual effect is negligible.
- Impact of microcracks is mitigated by blockage (hydration, self-healing etc.)
- Good correlation between transport vs. saturation degree vs. accessible porosity.
- Transport after carbonation – binder type & microstructural changes.
- Need more data on transport properties and durability of carbonated concretes (SCMs).
- Link/calibrate lab data to field studies – long-term performance in natural environments – benchmark results to field performance of established systems (database needed).

Microstructure and transport properties of partially saturated concrete

International Workshop on Mechanisms of Concrete Carbonation, ENPC, Champs-sur-Marne

27 June 2019

Hong S. Wong

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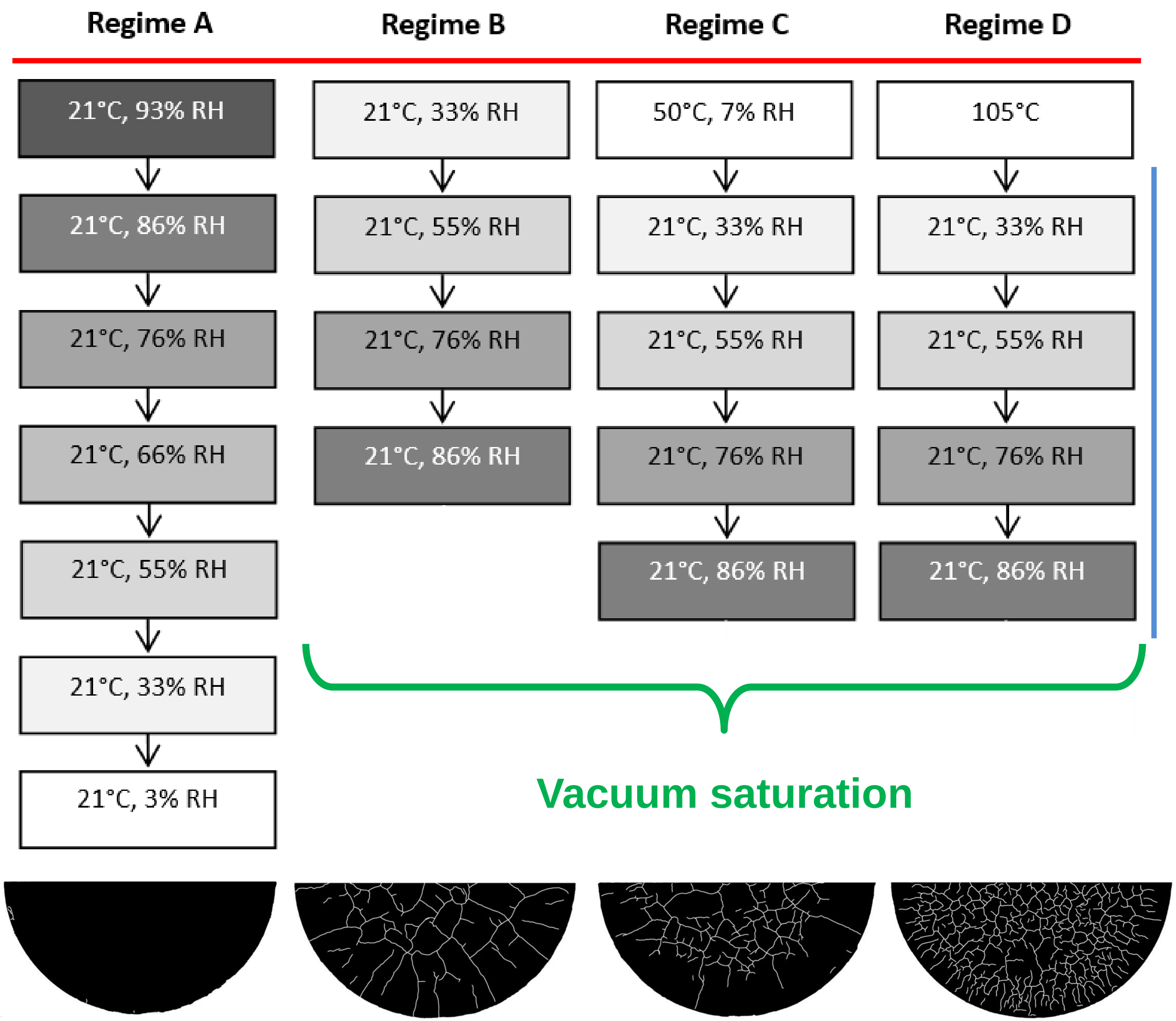
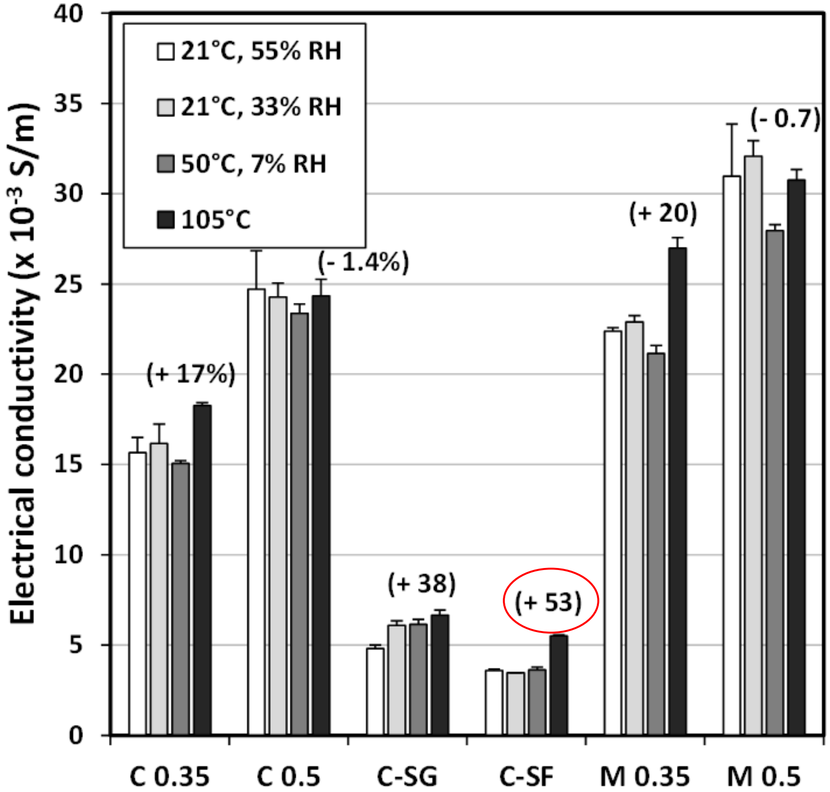
Bonus slides…

Drying + re-wetting + re-saturation

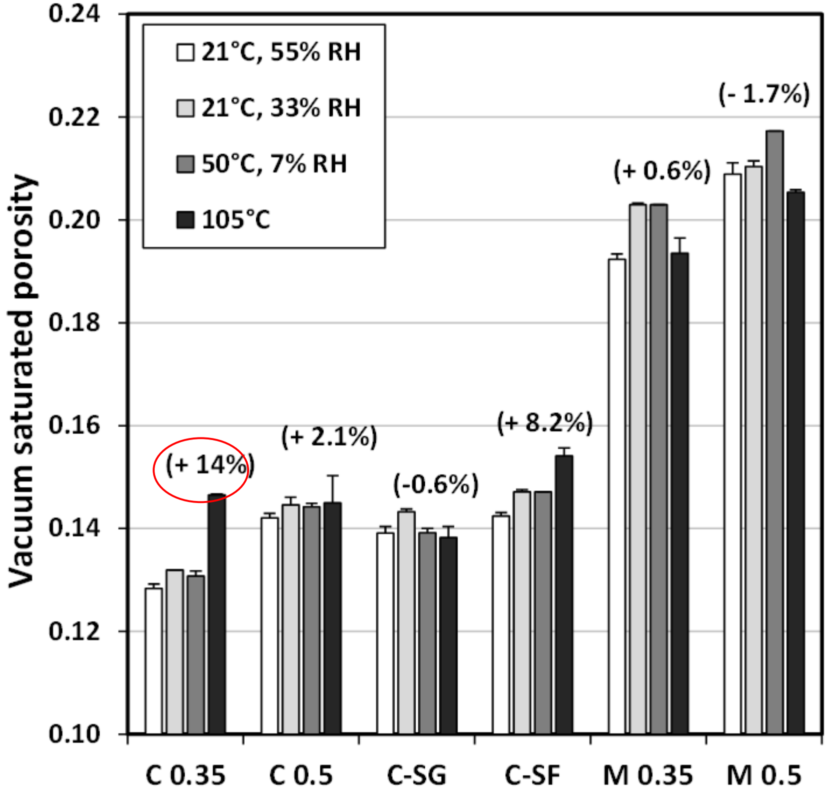


- Drying to induce varying degrees of microcracking
- Re-wetting via step wise increasing RH at 21ºC
- “Equilibrium” = mass loss < 0.01% per day
- Measure transport property at every conditioning step…
- **Vacuum saturation – measure electrical conductivity & porosity**

Electrical conductivity & total porosity after re-saturation



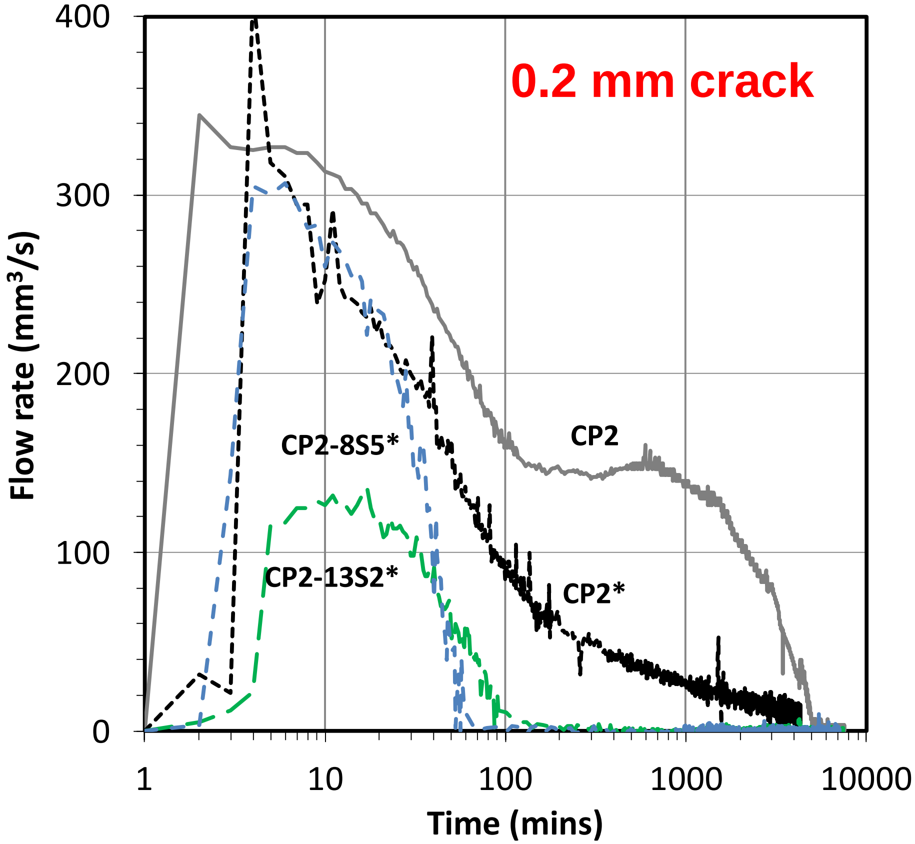
a) Electrical conductivity (90-day cured)

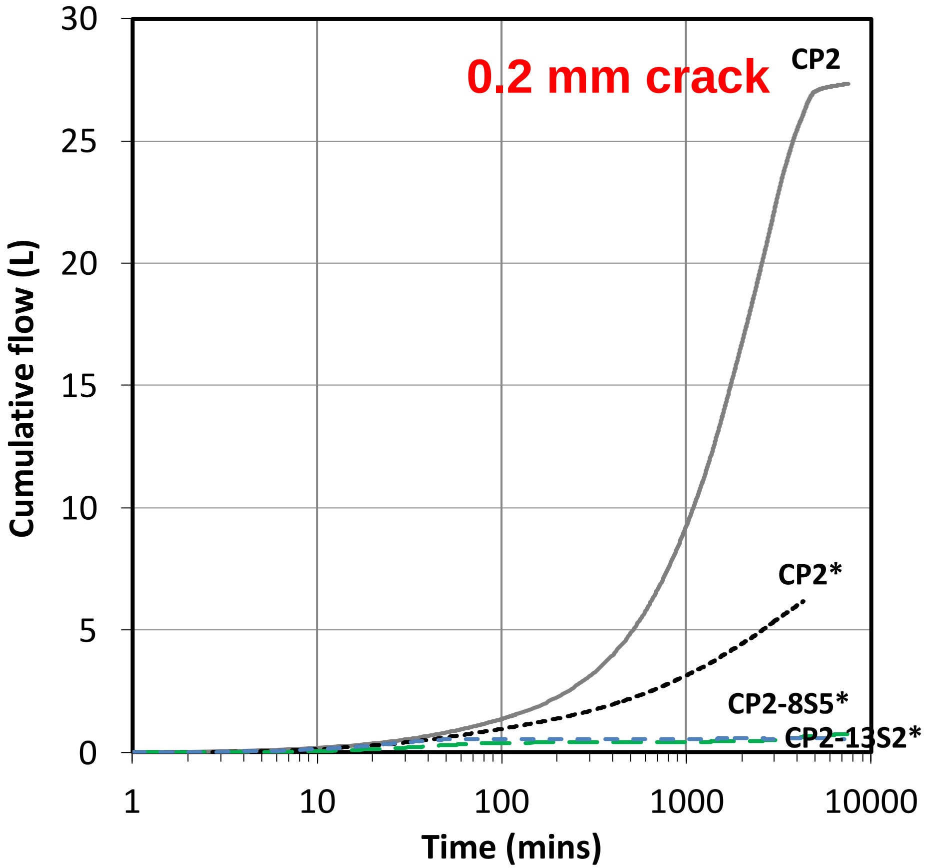


b) Porosity (90-day cured)

- Very little residual effects on electrical conductivity & porosity
- Influence of drying-induced damages is insignificant following re-saturation
- **Why?**
  - *Continued hydration causes crack healing?*
  - *Blockage by condensation at narrow crack constrictions?*
  - *Swelling of C-S-H on re-wetting?*

Self-healing





- Flow rate peak at ~ 5 min, then decline over time
- Smaller cracks heal faster
- Mechanisms for self-healing:
  - Continued hydration, swelling
  - Carbonation-induced precipitation
  - Crack blockage by loose debris