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X-RAY COMPUTED TOMOGRAPHY

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Servicios Centrales de Apoyo a la Investigación (SCAI)
26/10/2023

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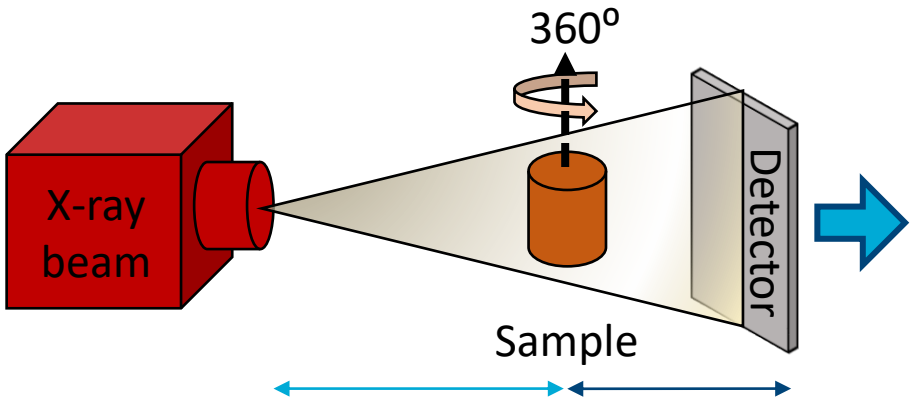
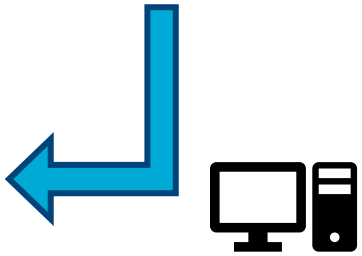
INDEX

ACQUISITION & RECONSTRUCTION



Proyections 2D

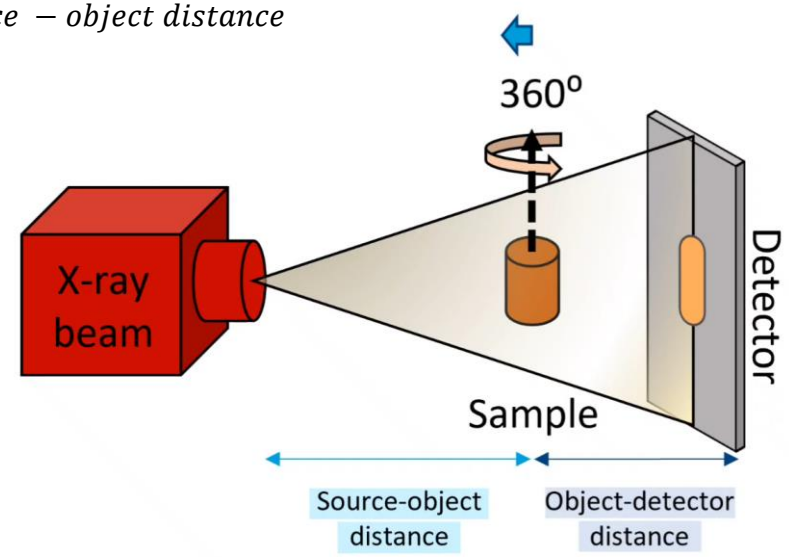
3D



Feldkamp backprojection algorithm

MAGNIFICATION

$$Magnification = \frac{\boldsymbol{Object - detector\ distance}}{Source\ - object\ distance}$$



Geometrical Magnification

- Higher source-object distance → Bigger voxel size → Lower spatial resolution
- Lower source-object distance → Smaller voxel size → Higher spatial resolution

Smaller **voxel** size → smaller **object** size

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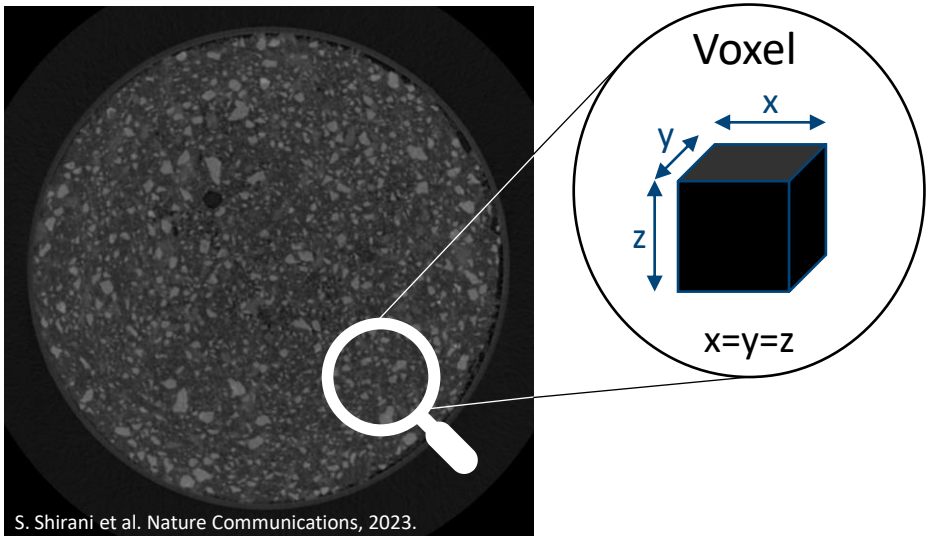
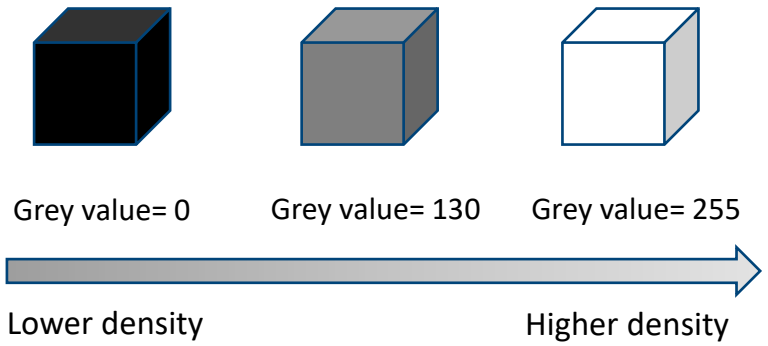
VOXEL SIZE & GREY VALUE





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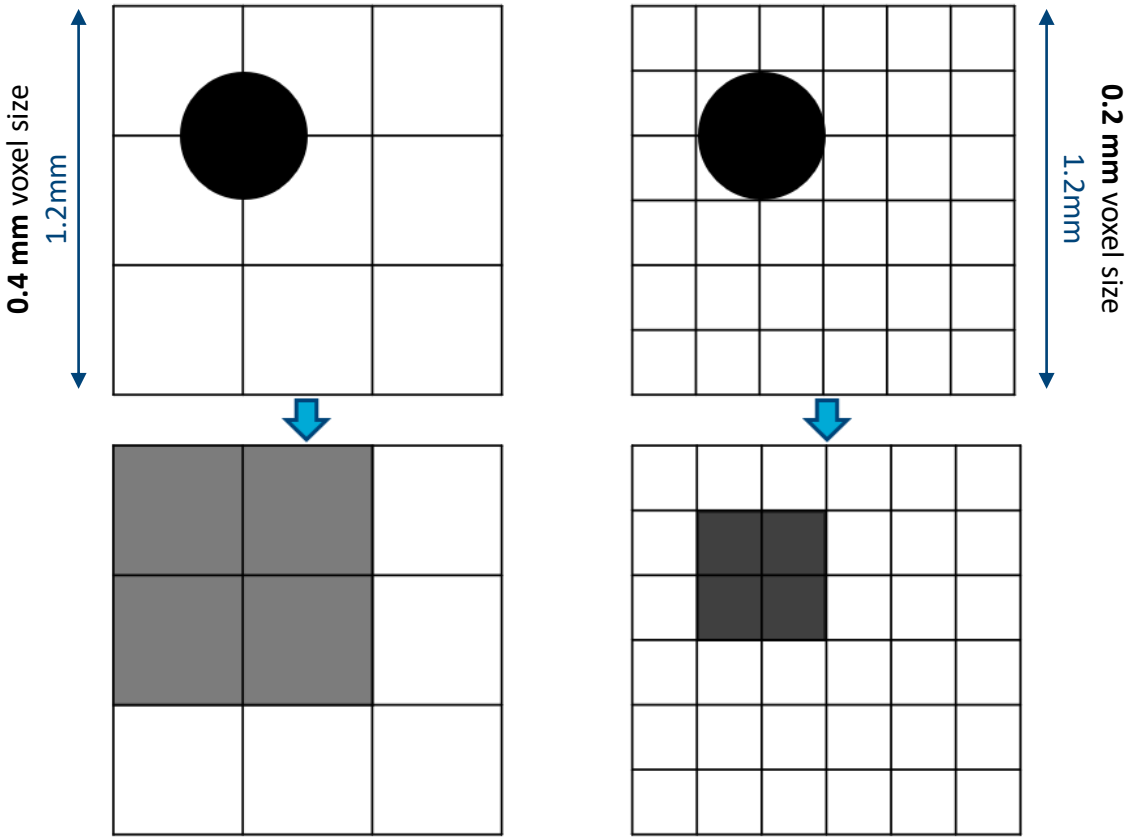
VOXEL SIZE & GREY VALUE



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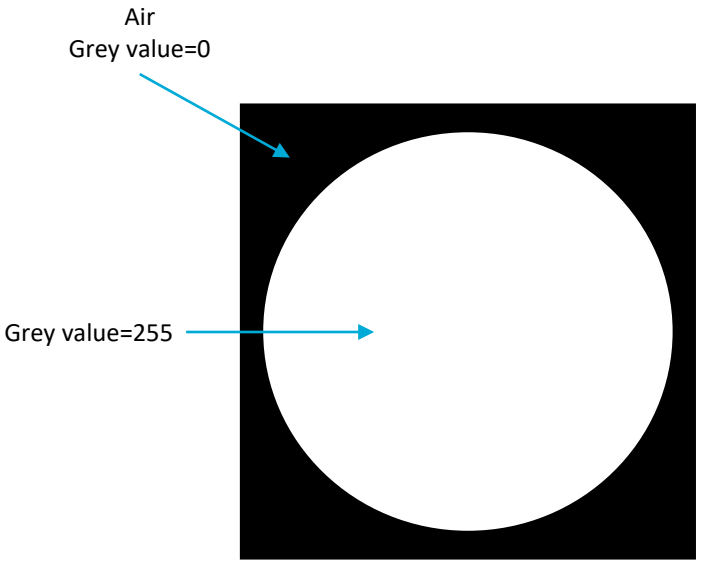
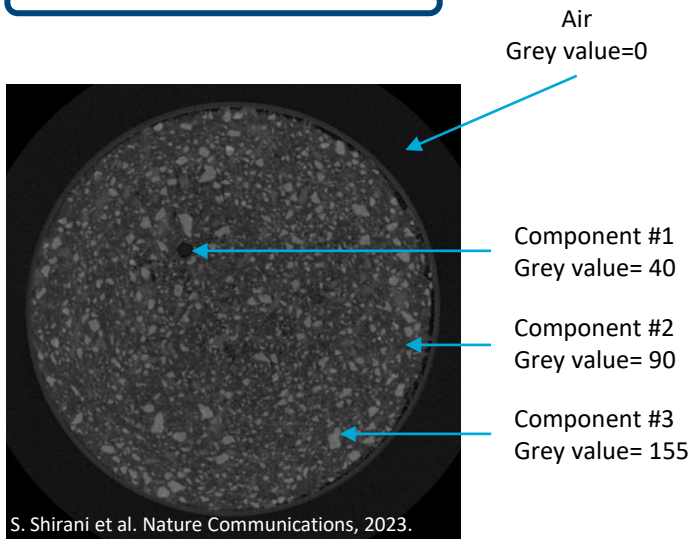
CONTRAST

X-rays pass through material and get attenuated ⇨ different absorption ⇨ **CONTRAST**

Absorption is related to:
- Density
- Atomic mass





3D segmentation → Visualization and quantification

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SUMMARY

- ✓ Non-destructive technique
- ✓ Information about internal details (tens of nanometers)
- ✓ Follow the structural evolution of materials in three dimensions in real time or in a time-lapse manner

- ! No information related to crystalline structure
- ! Sample size can compromise the resolution
- ! Contrast

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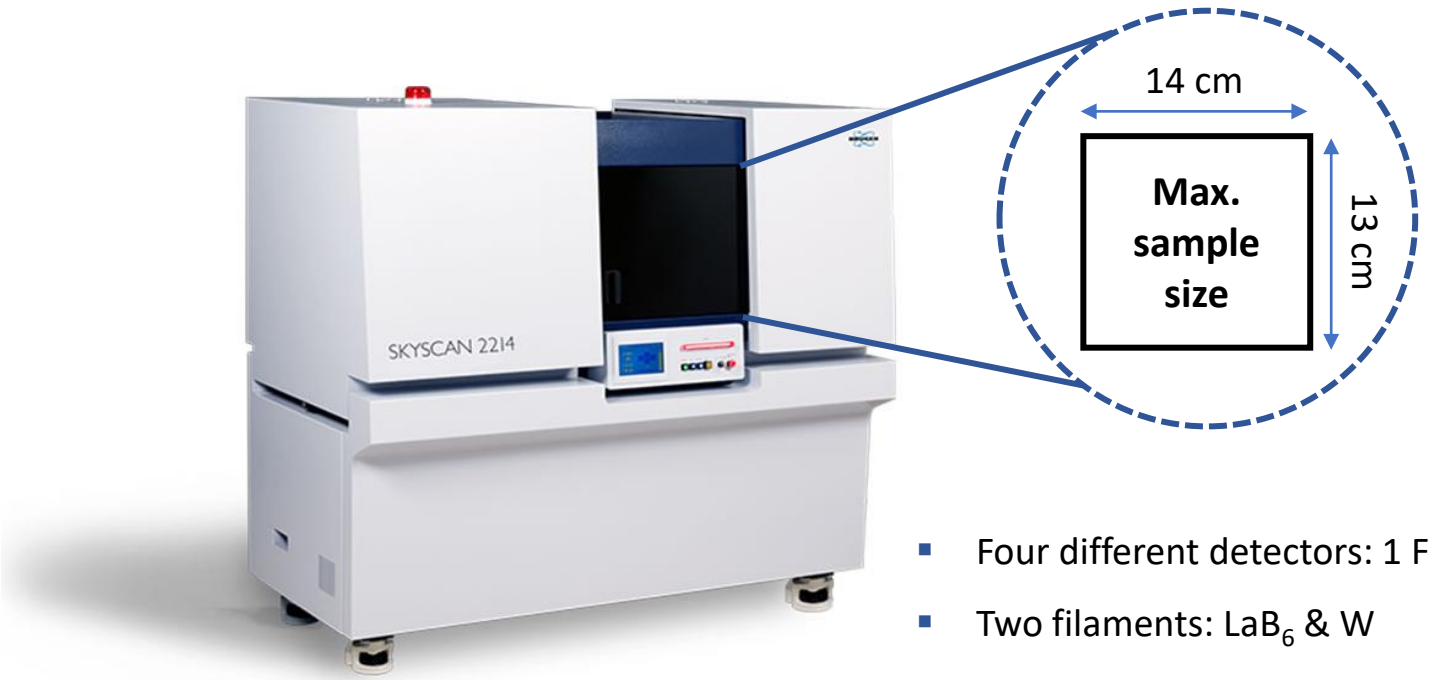

SkyScan 2214 (Bruker)

- Four different detectors: 1 Flat panel + 3 CCD cameras
- Two filaments: LaB_6 & W
- Temperature stages
- Tensile and compression: Deben Stage
- Down to 60 nm pixel size

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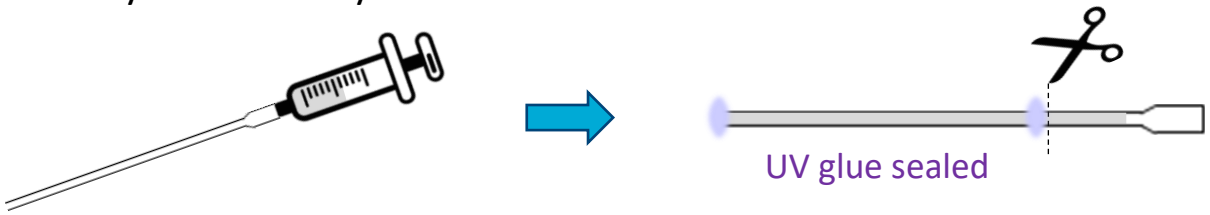
XRD & CT COMBINATION

Main goal: Better understanding of the hydration process of cements

- Evolution of the crystalline phases during hydration → **LXRPD** (Rietveld method)
- Porosity and amorphous content development → **µCT**

In situ cement hydration study:



Glass capillary Φ= 2mm → Thick capillary to avoid self-desiccation

Salcedo, I.R.; Cuesta, A.; Shirani, S.; León-Reina, L.; Aranda, M.A.G. Accuracy in Cement Hydration Investigations: Combined X-ray Microtomography and Powder Diffraction Analyses. *Materials* **2021**, *14*, 6953. https://doi.org/10.3390/ma14226953

Shirani, S.; Cuesta, A.; Morales-Cantero; A.; Santacruz, I.; Diaz, A.; Trtik, P.; Holler, M.; Rack, A.; Lukic, B.; Brun, E.; Salcedo, I. R.; Aranda, M. A. 4D nanoimaging of early age cement hydration. *Nature Communications* **2023**, *14*(1), 2652. https://doi.org/10.1038/s41467-023-38380-1

Shirani, S.; Cuesta, A.; De la Torre, A.G.; Santacruz, I.; Morales-Cantero, A.; Koufany, I.; Redondo-Soto, C.; Salcedo, I. R.; León-Reina, L.; Aranda, M.A.G. Mix and measure - combining *in situ* X-ray powder diffraction and microtomography for accurate hydrating cement studies. Submitted to *Cement and Concrete Research* **2023**

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XRD & CT COMBINATION

µCT setup

UV glue sealed

Wax

Capillary holder

+

2 sample stage adaptors: XRD & CT adaptors

Custom designed sample holder

XRD setup

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XRD & CT COMBINATION

µCT setup

Capillary holder

+

2 sample stage adaptors: XRD & CT adaptors

Custom designed sample holder

XRD setup

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XRD & CT COMBINATION

SKYSCAN 2214
(Bruker)

Capillary holder

+

2 sample stage adaptors: XRD & CT adaptors

Custom designed sample holder

D8 ADVANCE
(Bruker)
Mo $K\alpha_1$ radiation

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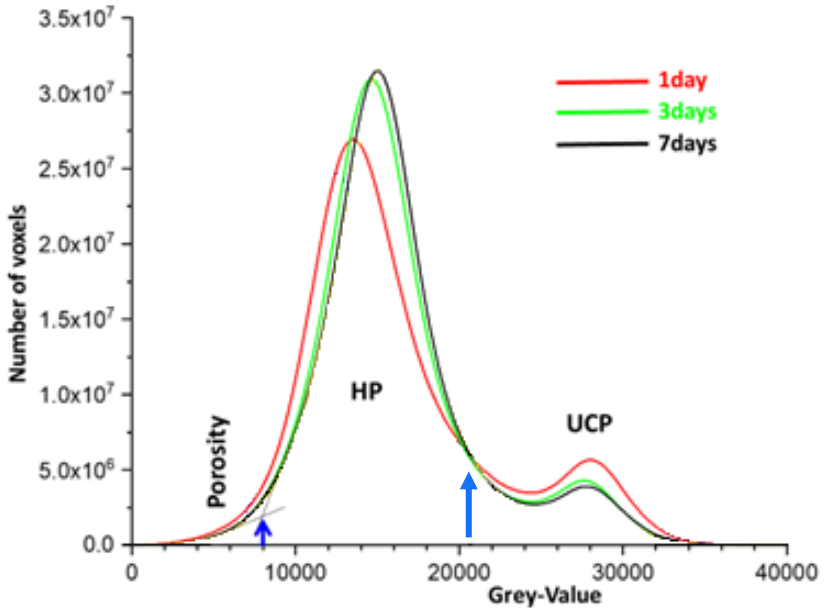
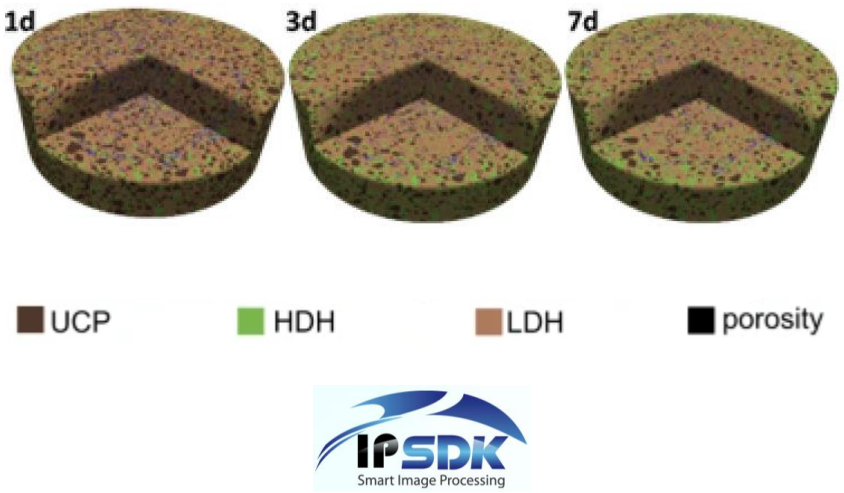
XRD & CT COMBINATION

Global Thresholding Segmentation → **3 components**



Machine Learning Segmentation → **4 components**



IPSDK Smart Image Processing

Porosity = air + water
HP (Hydrated Particles) = **HDH** (high-density hydrates: mainly portlandite + calcite) + **LDH** (low-density hydrates: mainly C-S-H gel + ettringite)
UCP (Unhydrous Cement Particles) = all unreacted clinker phases

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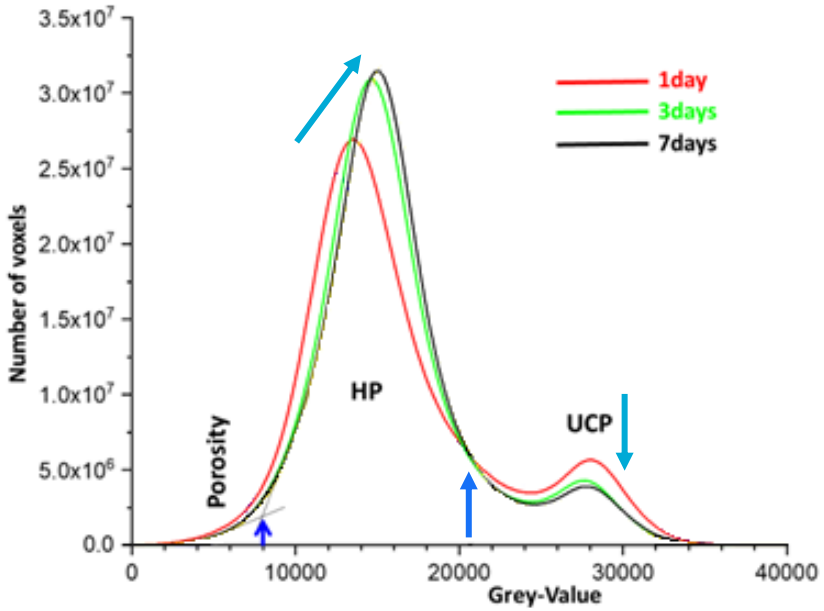
XRD & CT COMBINATION

Global Thresholding Segmentation → **3 components**



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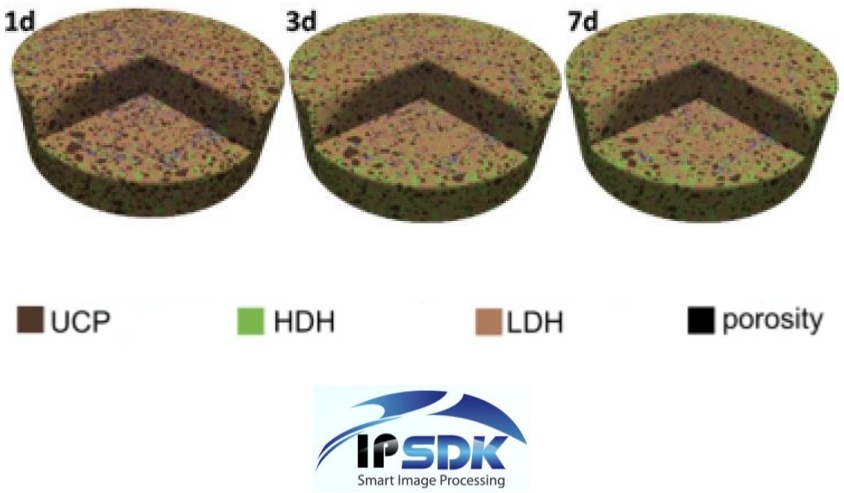

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XRD & CT COMBINATION

Comparison of RQPA and µCT results (vol%):

Hydration age	Components	LXRPD	Global Thresholding	Machine Learning*
1d	HP	78.5	79.9	76.2
	UCP	21.5	20.1	23.8
3d	HP	84.1	84.0	83.2
	UCP	15.9	16.0	16.8
7d	HP	85.5	84.8	84.2
	UCP	14.5	15.2	15.8

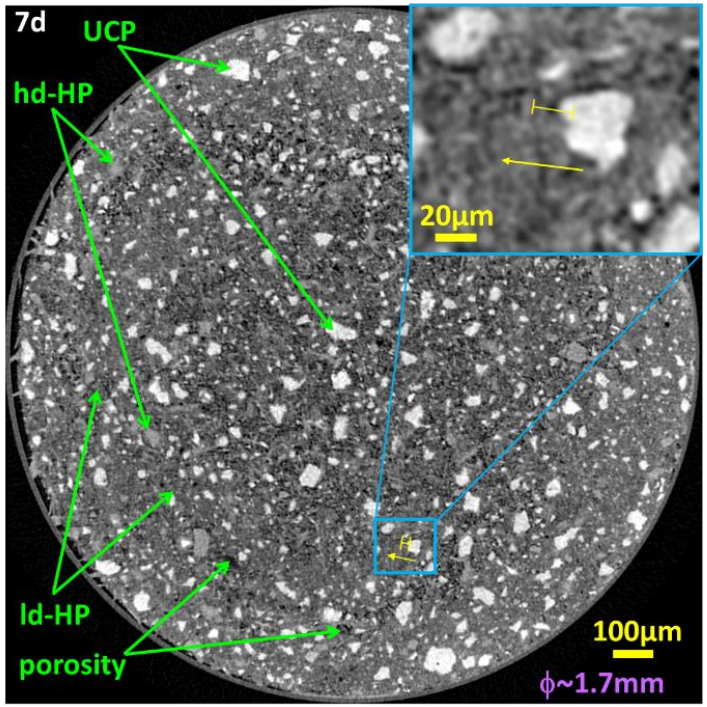
*LDH and HDH derived from ML have been summed as a single hydrate phase (HP) for comparison to global thresholding and LXRPD results.

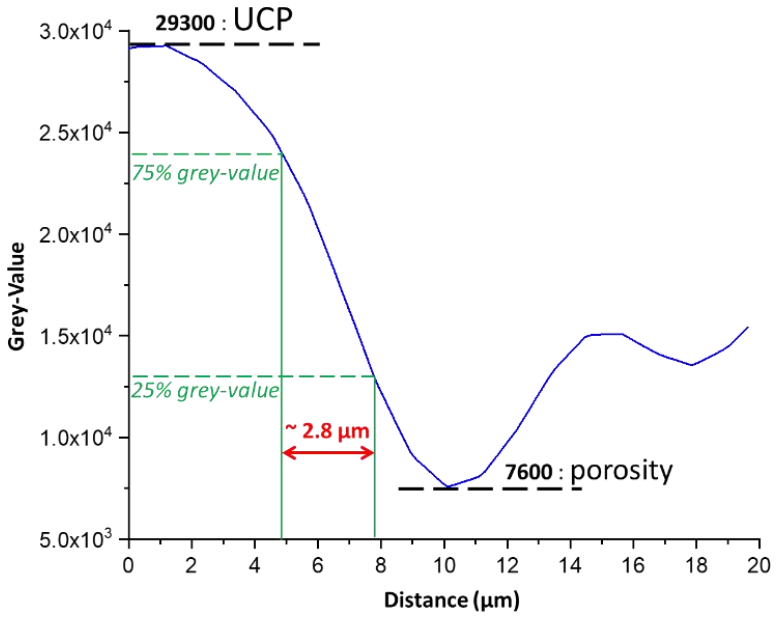
Shirani, S.; Cuesta, A.; De la Torre, A.G.; Santacruz, I.; Morales-Cantero, A.; Koufany, I.; Redondo-Soto, C.; Salcedo, I. R.; León-Reina, L.; Aranda, M.A.G. Mix and measure - combining *in situ* X-ray powder diffraction and microtomography for accurate hydrating cement studies. Submitted to *Cement and Concrete Research* **2023**

XRD & CT COMBINATION

Spatial Resolution:

Voxel size = 1.1µm
Spatial resolution ~ 2.8µm





Shirani, S.; Cuesta, A.; De la Torre, A.G.; Santacruz, I.; Morales-Cantero, A.; Koufany, I.; Redondo-Soto, C.; Salcedo, I. R.; León-Reina, L.; Aranda, M.A.G. Mix and measure - combining *in situ* X-ray powder diffraction and microtomography for accurate hydrating cement studies. Submitted to *Cement and Concrete Research* **2023**

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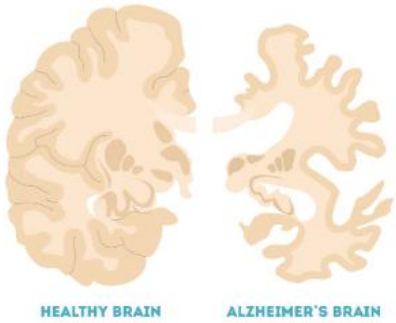
MORPHOMETRIC STUDY IN BONES



DCI + water

5xFAD
Age: 6-10 months

D-Chiro-inositol (DCI)

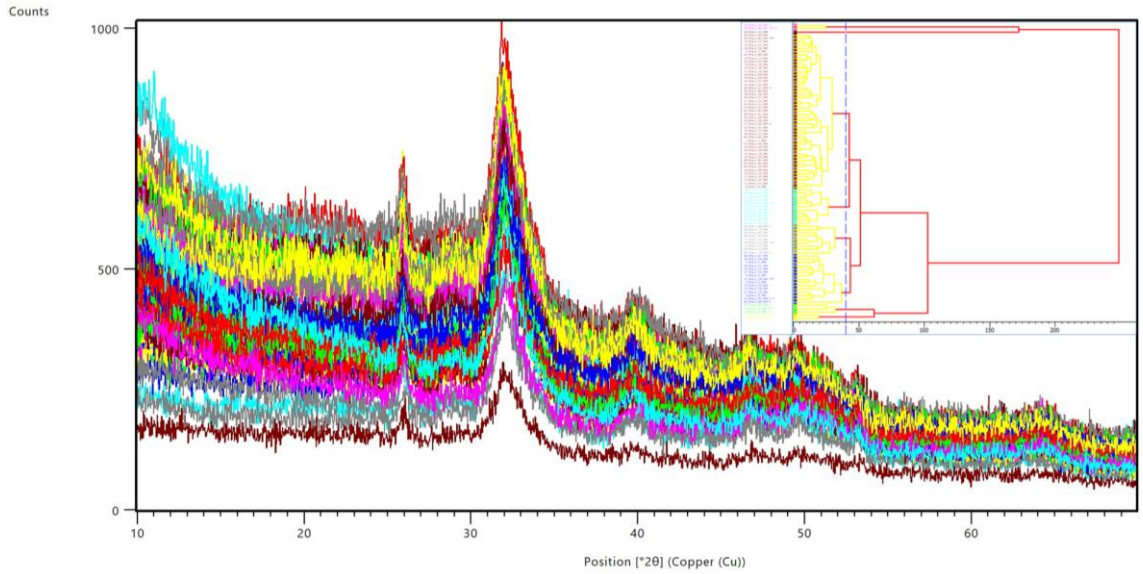
- Insulin signalling
- Antioxidant
- Anti-inflammatory

López-Gambero, A. J. (2022). Estudio de la eficacia de la administración dietética del D-Chiro-inositol en la señalización de insulina, neuroinflamación y alteraciones metabólicas en un modelo animal de la enfermedad de Alzheimer. Universidad de Málaga.

MORPHOMETRIC STUDY IN BONES

Alzheimer disease → lower bone quality

DRX: to study the hydroxyapatite crystal structure and the amorphous content variations.



Micro-CT: to determine possible structural alteration of bone microarchitecture.

López-Gambero, A. J. (2022). Estudio de la eficacia de la administración dietética del D-Chiro-inositol en la señalización de insulina, neuroinflamación y alteraciones metabólicas en un modelo animal de la enfermedad de Alzheimer. Universidad de Málaga.

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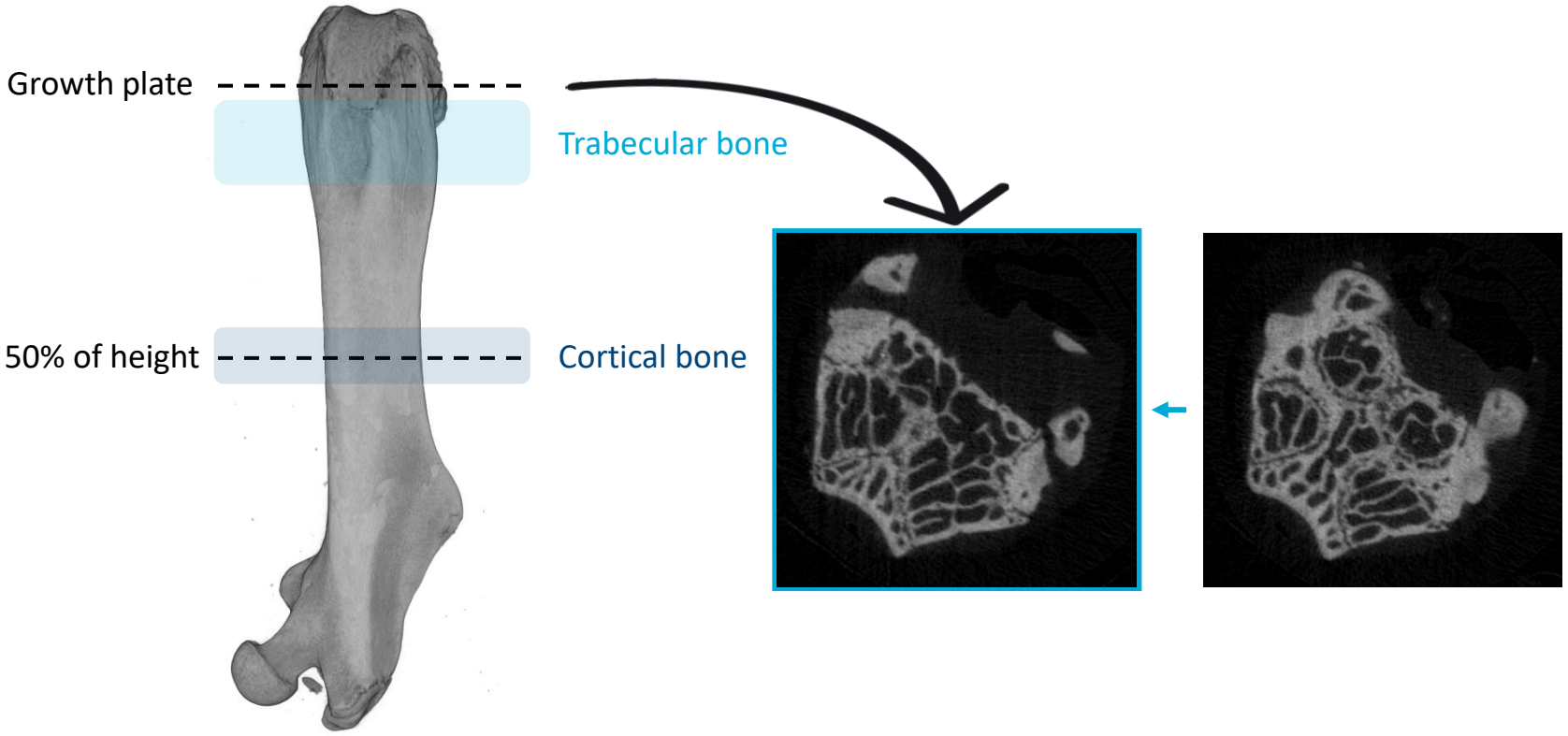
MORPHOMETRIC STUDY IN BONES



López-Gambero, A. J. (2022). Estudio de la eficacia de la administración dietética del D-Chiro-inositol en la señalización de insulina, neuroinflamación y alteraciones metabólicas en un modelo animal de la enfermedad de Alzheimer. Universidad de Málaga.

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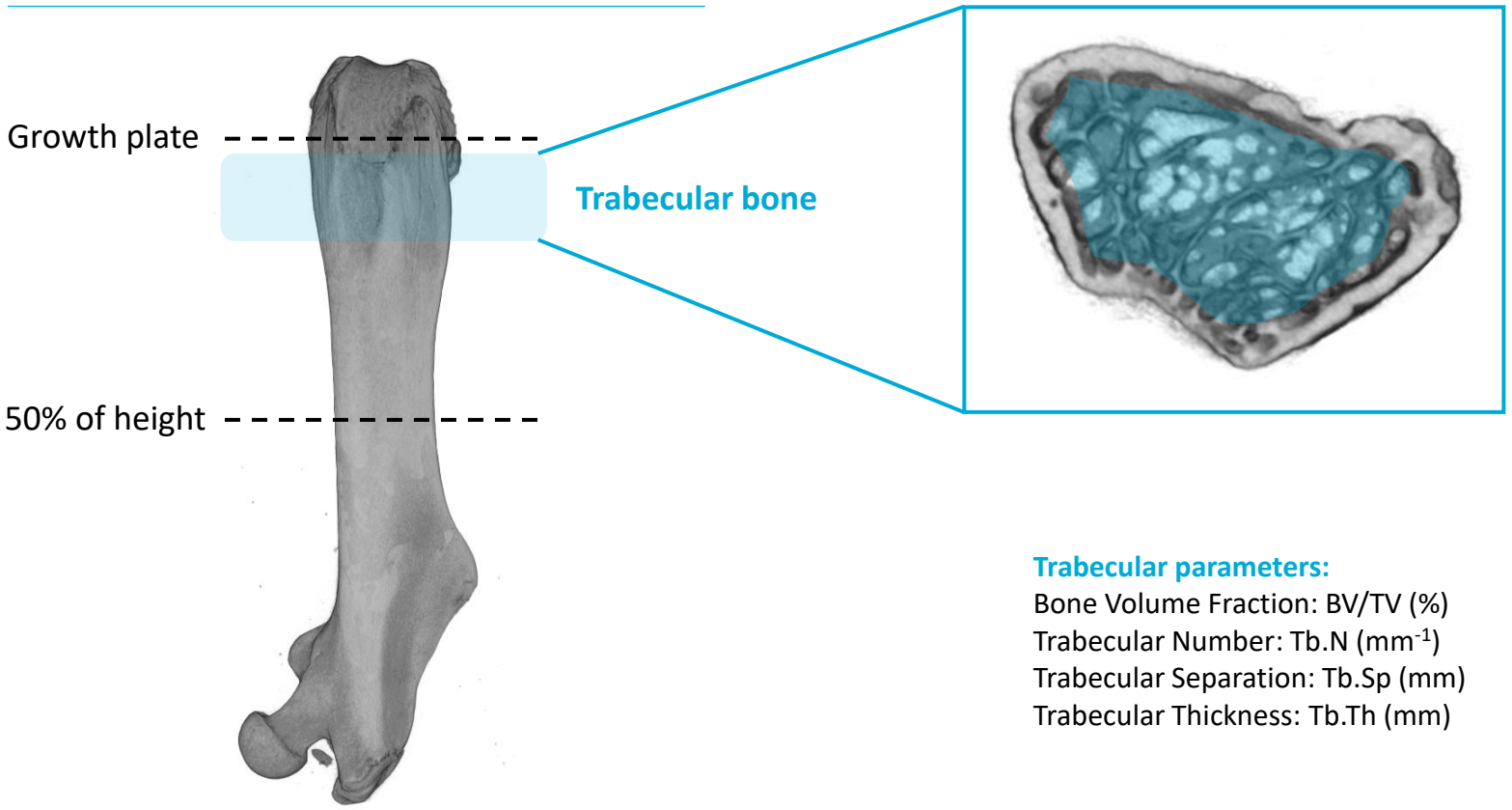
MORPHOMETRIC STUDY IN BONES



Trabecular parameters:

Bone Volume Fraction: BV/TV (%)
Trabecular Number: Tb.N (mm^{-1})
Trabecular Separation: Tb.Sp (mm)
Trabecular Thickness: Tb.Th (mm)

López-Gambero, A. J. (2022). Estudio de la eficacia de la administración dietética del D-Chiro-inositol en la señalización de insulina, neuroinflamación y alteraciones metabólicas en un modelo animal de la enfermedad de Alzheimer. Universidad de Málaga.

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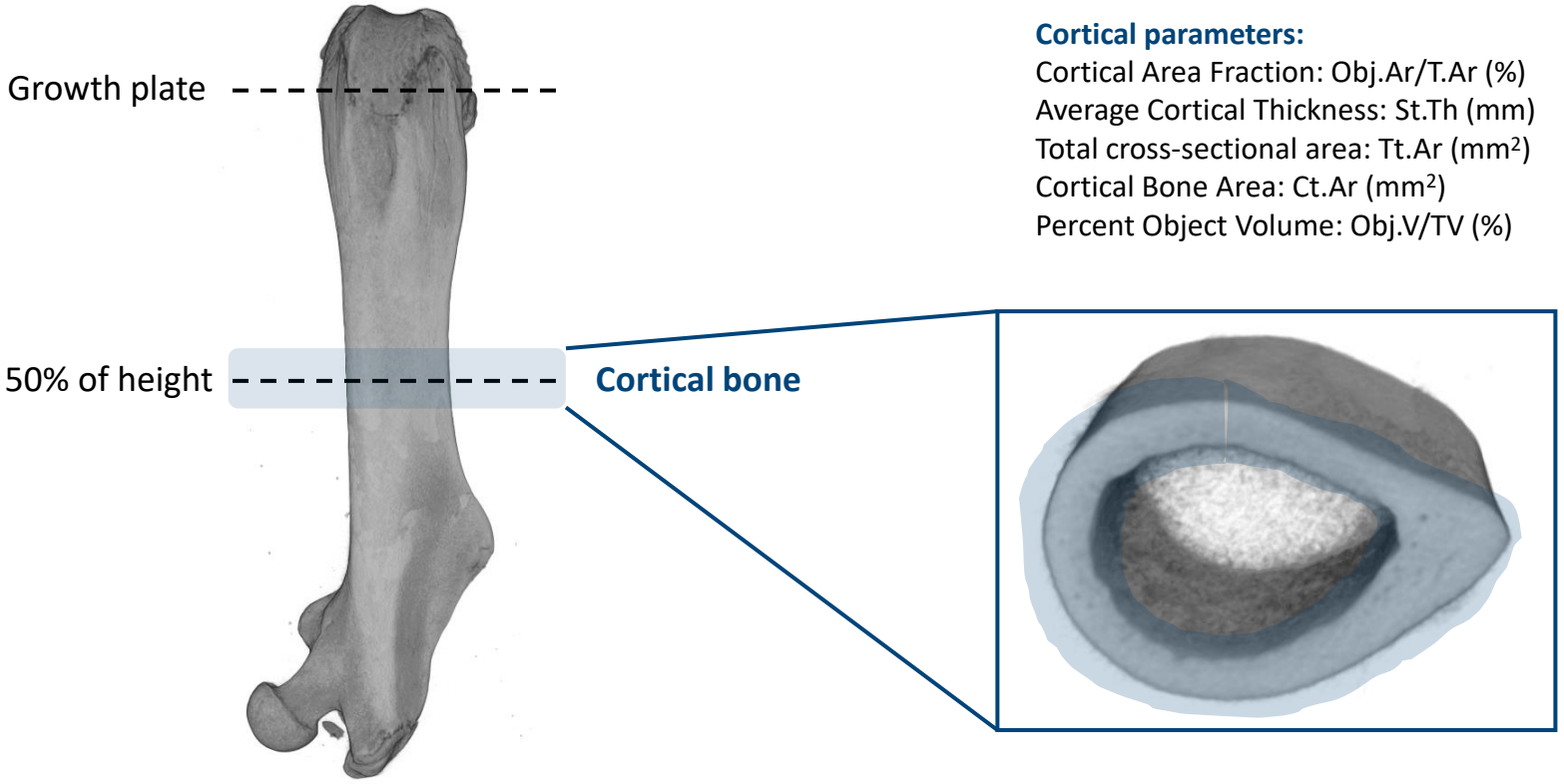
MORPHOMETRIC STUDY IN BONES



Cortical parameters:
Cortical Area Fraction: Obj.Ar/T.Ar (%)
Average Cortical Thickness: St.Th (mm)
Total cross-sectional area: Tt.Ar (mm^2)
Cortical Bone Area: Ct.Ar (mm^2)
Percent Object Volume: Obj.V/TV (%)

López-Gambero, A. J. (2022). Estudio de la eficacia de la administración dietética del D-Chiro-inositol en la señalización de insulina, neuroinflamación y alteraciones metabólicas en un modelo animal de la enfermedad de Alzheimer. Universidad de Málaga.

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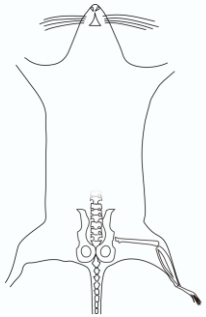
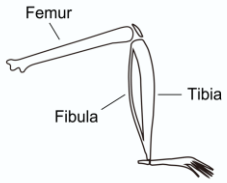
BONE MARROW ADIPOSE TISSUE

1

Mouse dissection:



Remove soft tissue



2

Fixation of the bone:

- 10% neutral buffered formalin (O/N, 4°C with gentle agitation)
- Wash in running cool tap water (1h)

3

Decalcification of the bones:

- EDTA decalcification solution (~ 14 days) → remove mineral to allow optimal penetration of the Os
- Wash in cool running tap water (1h)

4

Osmium staining of the decalcified bones:

- Osmium tetroxide (48 h) at RT
- Wash under cool running tap water (2h)

Scheller, E. L., Troiano, N., VanHoutan, J. N., Bouxsein, M. A., Fretz, J. A., Xi, Y., ... & Horowitz, M. C. (2014). Use of osmium tetroxide staining with microcomputerized tomography to visualize and quantify bone marrow adipose tissue in vivo. In *Methods in enzymology* (Vol. 537, pp. 123-139). Academic Press.

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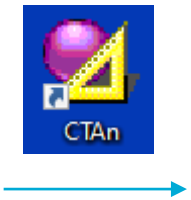
BONE MARROW ADIPOSE TISSUE



3D Volume Rendering

Distribution of MAT

16.3% MAT

Scheller, E. L., Troiano, N., VanHoutan, J. N., Bouxsein, M. A., Fretz, J. A., Xi, Y., ... & Horowitz, M. C. (2014). Use of osmium tetroxide staining with microcomputerized tomography to visualize and quantify bone marrow adipose tissue in vivo. In *Methods in enzymology* (Vol. 537, pp. 123-139). Academic Press.

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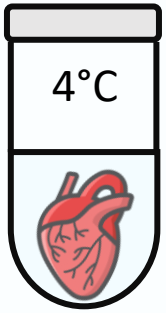
SOFT TISSUE

1

Dehydration process:

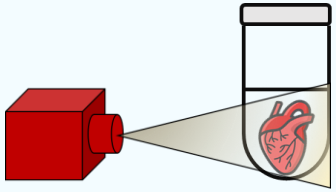
- PBS (1 day)
- EtOH 30% (1 day)
- EtOH 50% (1 day)
- EtOH 70% (1 day)
- EtOH:MeOH:PBS 4:4:3 (1h)
- MeOH 80% (1h)
- MeOH 90% (1h)



2

Staining process:

- 0.7% Phosphotungstic acid (PTA) + MeOH 90%

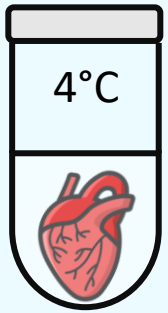


Rapid scan to ensure adequate stain penetration

3

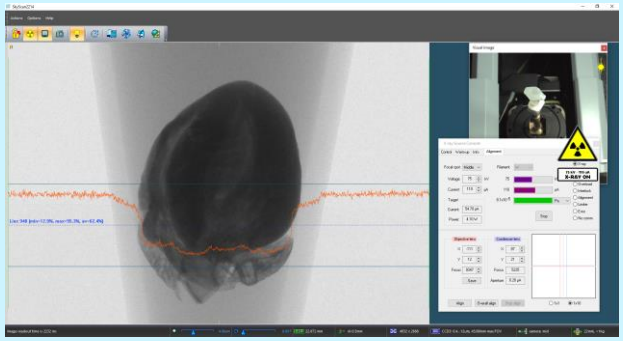
Rehydration process:

- MeOH 90% (6h)
- MeOH 80% (O/N)
- MeOH 70% (6h)
- MeOH 50% (O/N)
- MeOH 30% (1 day)
- PBS (1 day)



4

CT scan:



Lesciotto, K. M; Motch Perrine S. M.; Kawasaki, M.; Stecko, T.; Ryan, T.M.; Kawasaki, K.; Richtsmeier, J. T. Phosphotungstic acid-enhanced microCT: Optimized protocols for embryonic and early postnatal mice. *Developmental Dynamics* **2020**, 249(4):573-585. doi: 10.1002/dvdy.136.

SOFT TISSUE

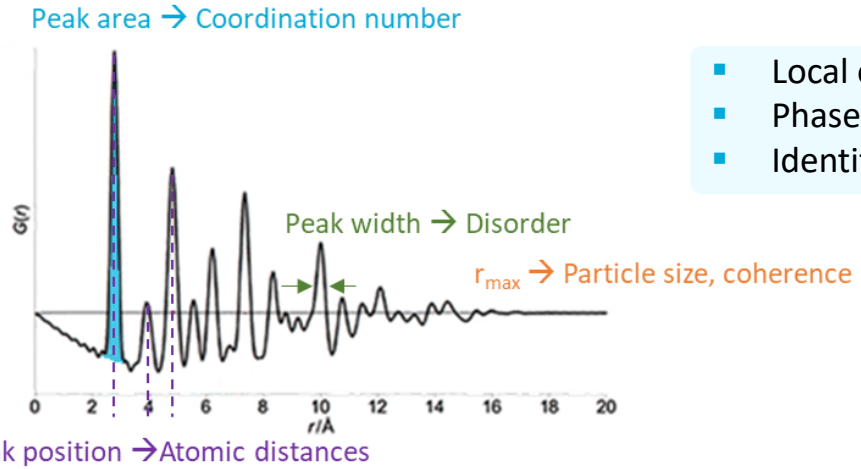
ELECTROCATALYSTS

Iron/Cobalt Phosphonate → (Pyrolytic treatment: H_2 or N_2) → Pyrophosphate- or phosphide-based iron/cobalt electrocatalysts

Characterization:

- Rietveld & Pair Distribution Function Analysis → Phase composition
- CT → Sample distribution in Carbon Paper (Spray and drop coating)

PDF information:



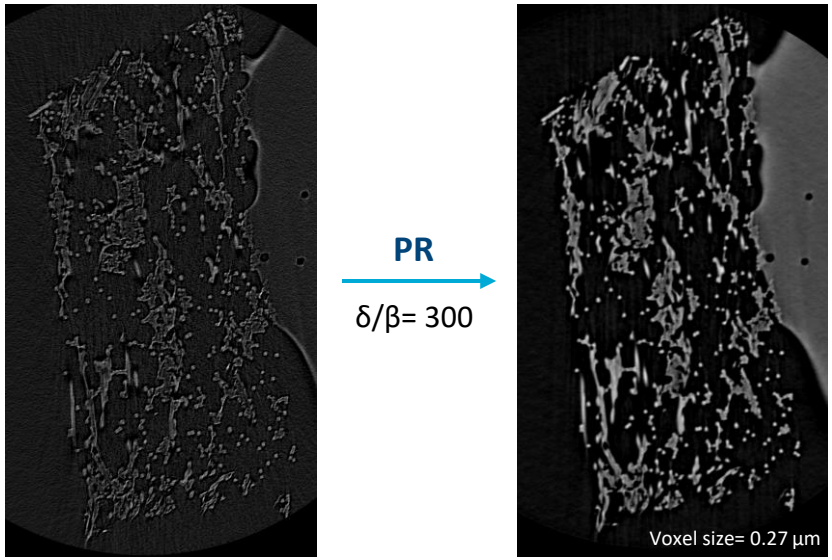
- Local order
- Phase quantification
- Identification of amorphous phase

Vílchez-Cózar, Á.; Colodrero, R. M.; Bazaga-García, M.; Marrero-López, D.; El-refaei, S. M.; Russo, P. A.; Pinna, N.; Olivera-Partos, P.; Cabeza, A. Tuning the activity of cobalt 2-hydroxyphosphonoacetates-derived electrocatalysts for water splitting and oxygen reduction: insights into the local order by pair distribution function analysis. *Applied Catalysis B: Environmental*, **2023**, 122963. https://doi.org/10.1016/j.apcatb.2023.122963

ELECTROCATALYSTS

Phase Retrieval (PR):

- Employed to enhance contrast from non-absorption interactions
- Method of processing X-ray projection images
- Useful for low dense materials



Paganin, D.; Mayo, S. C.; Gureyev, T. E.; Miller, P. R.; Wilkins, S. W. Simultaneous phase and amplitude extraction from a single defocused image of a homogeneous object. *J. Microsc.* **2002**, 206, 33-40.

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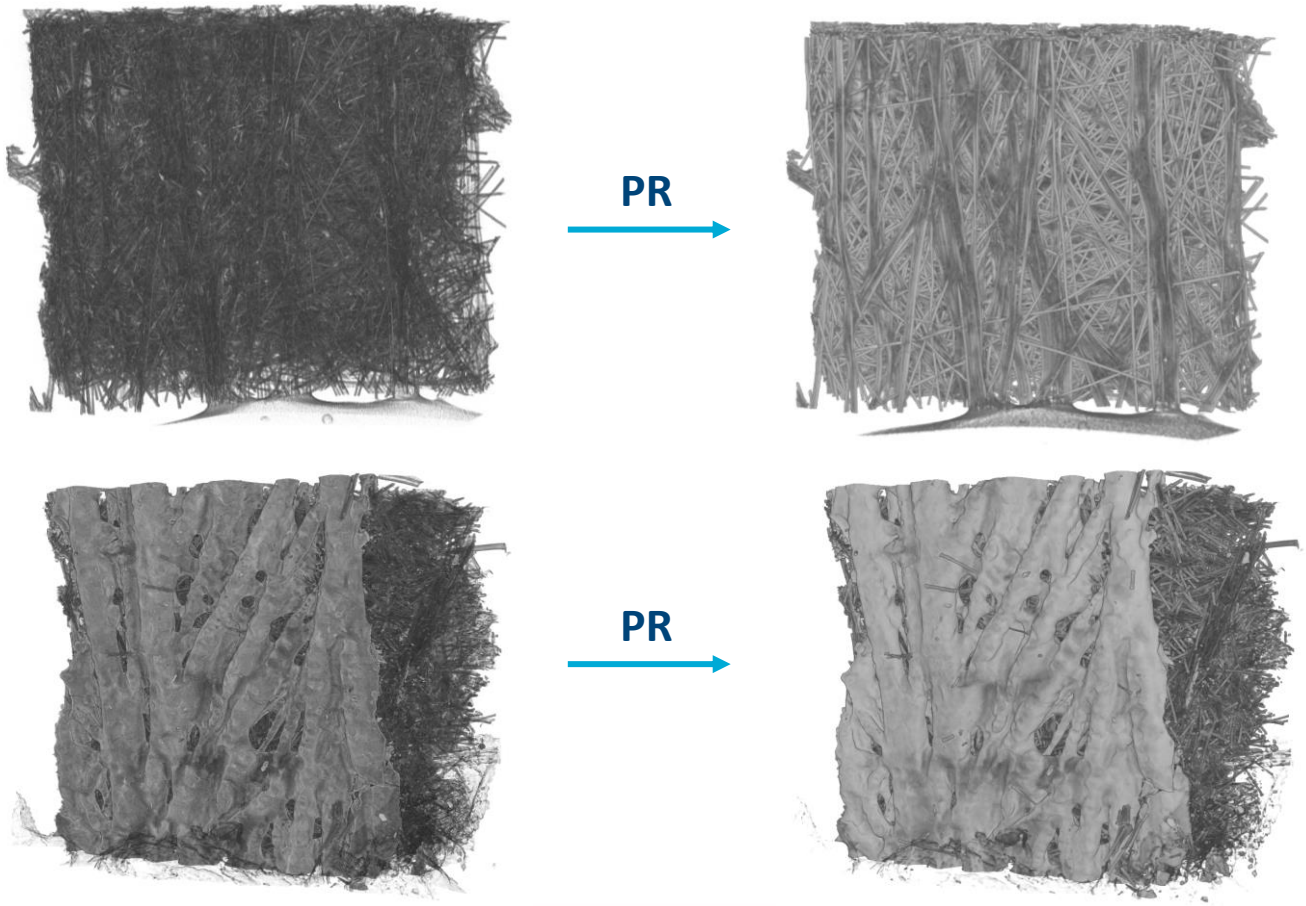
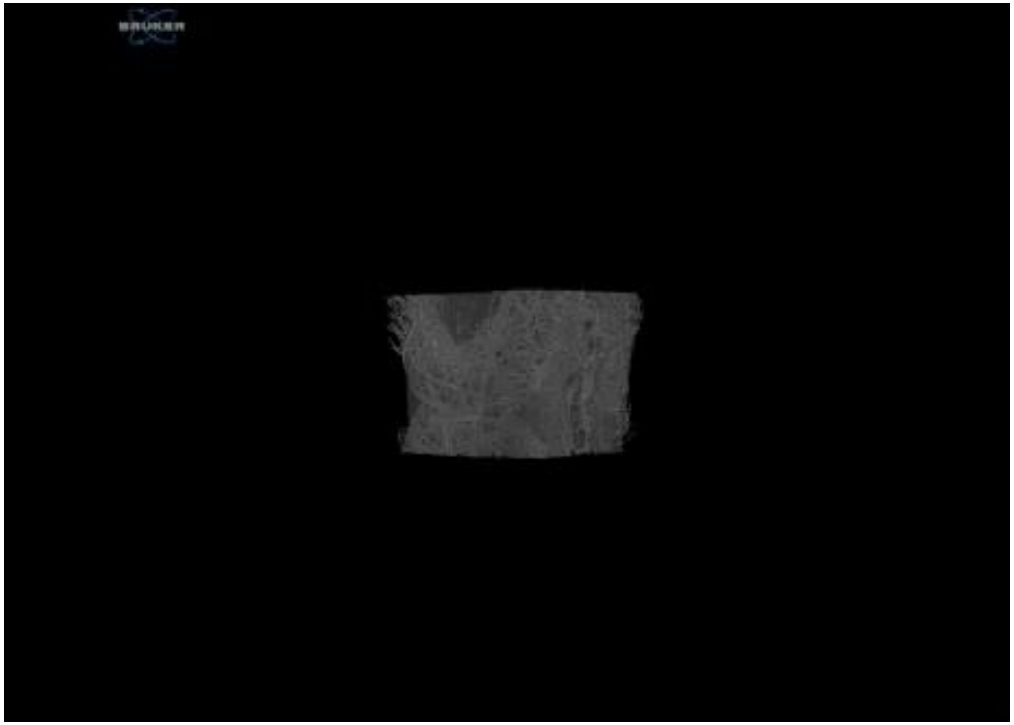
ELECTROCATALYSTS



IMAGING AND ANALYSIS OF FIBER



Fiber Manufacturing | Bruker

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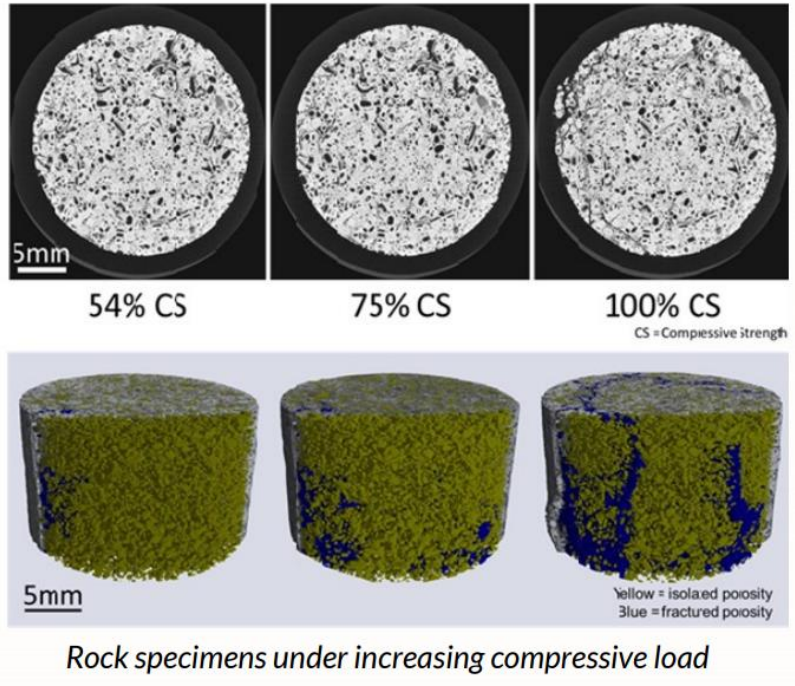
IN-SITU AND DYNAMIC EXPERIMENTS

Tensile and compression stage
DEBEN CT5000RT
Up to 5 kN

Temperature stages



Rock specimens under increasing compressive load

University-of-Ghent-CT5000-Geomaterials-Application-Story.pdf (deben.co.uk)

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