Multiscale characterization of biobased materials and applications

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JOURNEES TECHNIQUES ACOUSTIQUE ET VIBRATIONS

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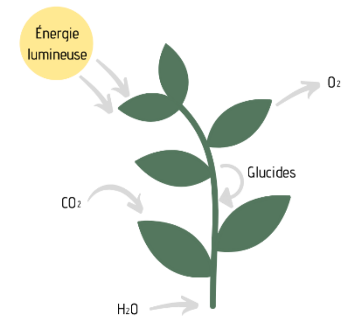
I. Context

1. Resources and environment

- **Challenges for the building sector:**
  - Improve comfort at home (noise, heat, humidity, IAQ, …)
  - Minimizing the ecological impact of construction materials

- **Solution : Bio/Geosourced materials**
  - Locally available and recyclable resources
  - Low gray energy and CO2 storage



- **Hemp-clay** (light earth)
  - Hemp particles + earth (clay) binder
  - Used for renovation and new buildings

I. Context

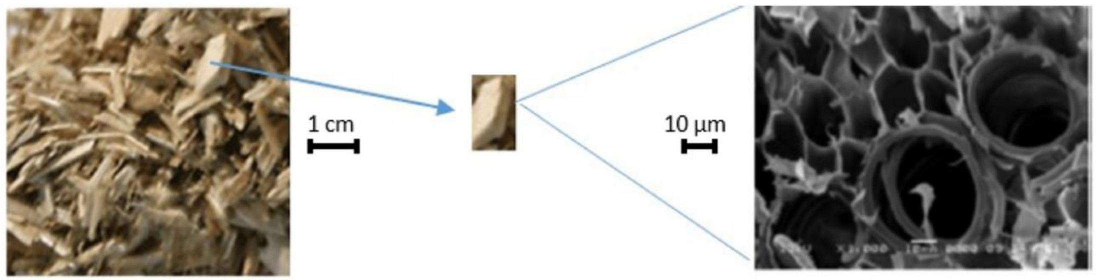
2. Eco-Terra project

Objectives:

- develop light-earth as a thermal and acoustic insulator for buildings, with very low environmental impact and using local resources
- facilitate lightweight earth construction on any territory, with non-standardized local resources and with a certain variability
- knowing and controlling the performances of the materials

I. Context

3. Focus on multiscale densitieS



Packing scale: apparent bulk density and inter-particle porosity	**Particle scale:** apparent particle density	**Cellular scale:** intra-particle porosity and cell walls density

- Shiv is a multiscale material, and is defined by multiple densities and associated porosities
- These various scales of porosity are directly related to:
  - Packing compressibility
  - Thermal properties
  - Vapor diffusion performances
  - Acoustic behavior

I. Context

3. Focus on multiscale densitieS

→ **Open issues and questions limiting the understanding of the physical behavior of shiv and related building materials…**

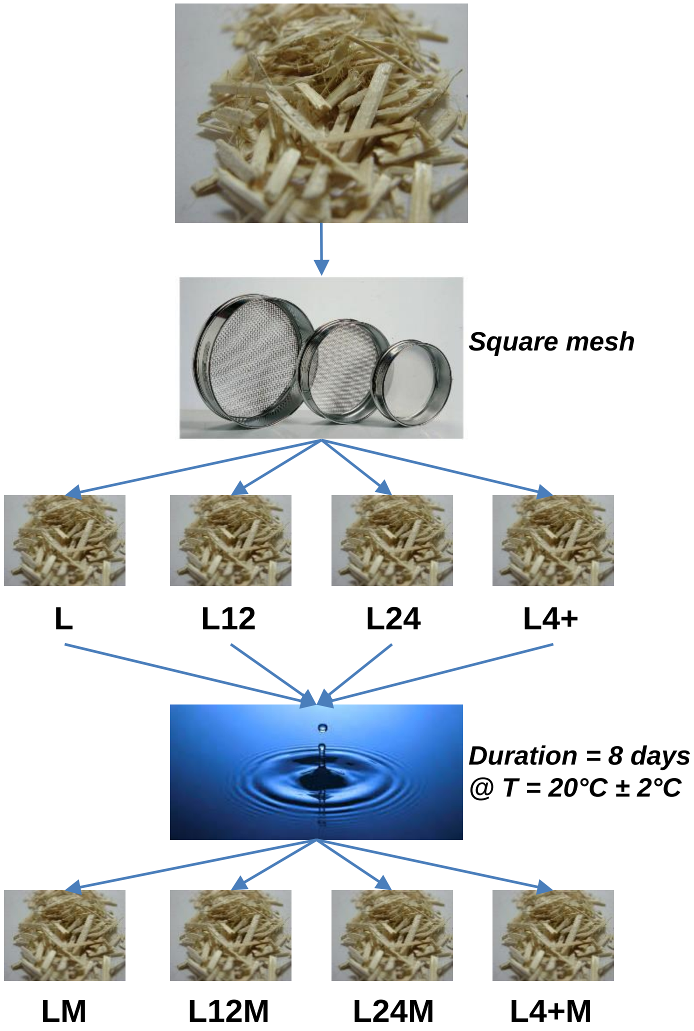
- Physical interpretation of the bulk density
- Measurement method for the particle envelope density
- Size distribution of the porosity
- Coexistence of 'open' and 'closed' pores
- Effects of particle size and aging on these characteristics

Outline

II. Materials and Methods

1. Hemp shiv under study

- Raw shiv:
  - One commercial hemp shiv
  - Origin: France
  - Retting: light
  - Code: L
- Variations by mechanic sieving:
  - **L**: raw sample
  - **L12**: fraction of L between 1 and 2 mm
  - **L24**: fraction of L between 2 and 4 mm
  - **L4+**: fraction of L above 4 mm
- Variations by immersion in water:
  - **LM**
  - **L12M**
  - **L24M**
  - **L4+M**
- Samples conditionning:
  - Mass stabilisation ± 0,01%



II. Materials and Methods

2. Microstructure and density investigation

Packing scale

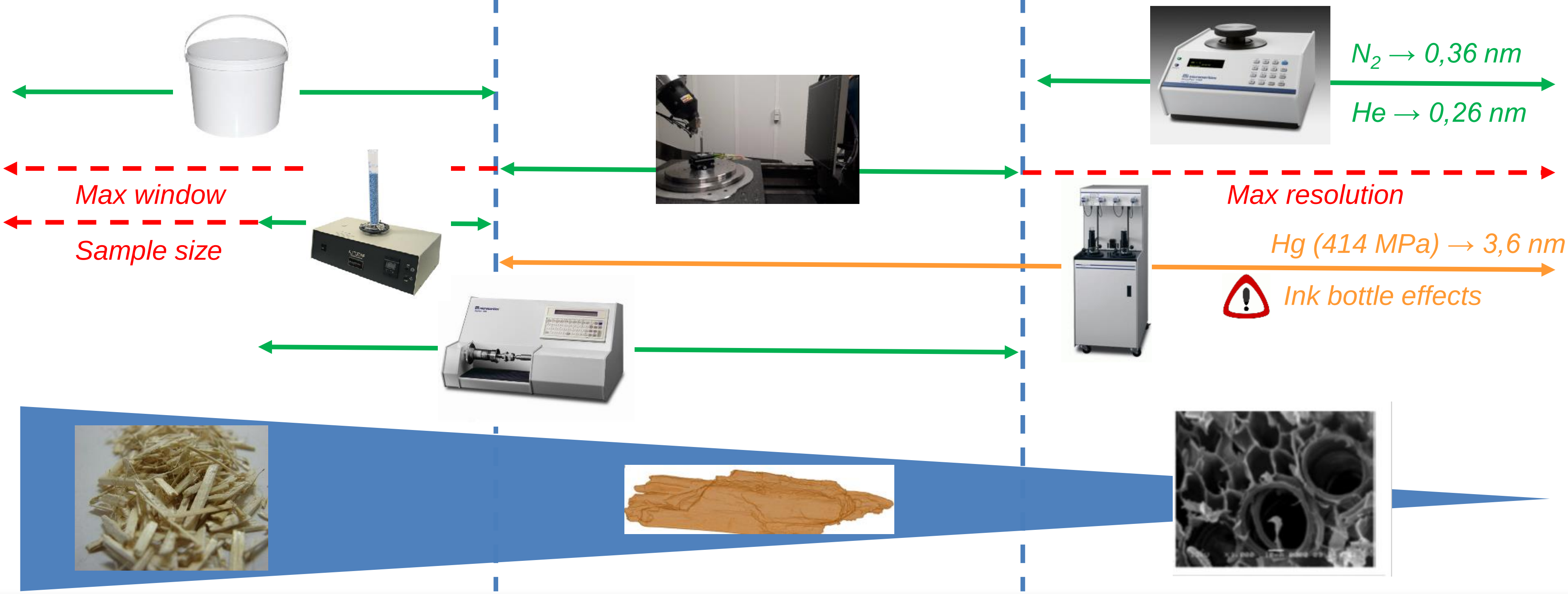
- Trimmed density
- Apparent density
- Tapped density
- Compacted density

Particle scale

- Powder pycnometry
- Mercury intrusion
- X-ray computed tomography

Cellular scale

- He pycnometry (powder)
- He pycnometry
- N_2 pycnometry
- Mercury intrusion



II. Materials and Methods

3. Characterisation of the physical properties

Water sorption

Measurements:

- DVS system

→ Drying at 40°C until mass stab.

→ RH: 0 to 90 %

→ Fit by GAB model

Water absorption

Measurements:

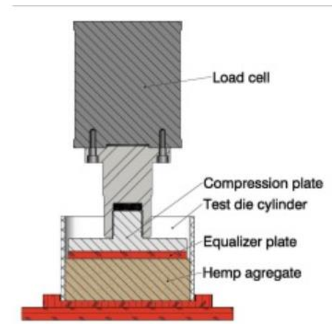
- Immersion in water

→ Drying at 40°C until mass stab.

→ Time: 6 steps from 5’ to 2 days

→ 1g sample

Compression



[Chi. et al, 2021]

Measurements:

- Test with an hydraulic press

→ 500 kN capacity

→ Die diameter: 30mm

→ Lower & Upper punches

→ Displacement control

Thermics

Measurements:

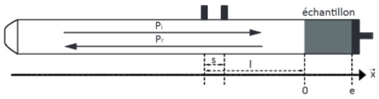
- Two devices

→ Drying at 60°C until mass stab.

→ Guarded hot plate: T at 23°C, sample size 7x30x30 cm

→ Hot disk: T at 20°C, sample volume of 1L

Acoustics



Measurements:

- Kundt tube device

→ Frequency range [50 – 2000 Hz]

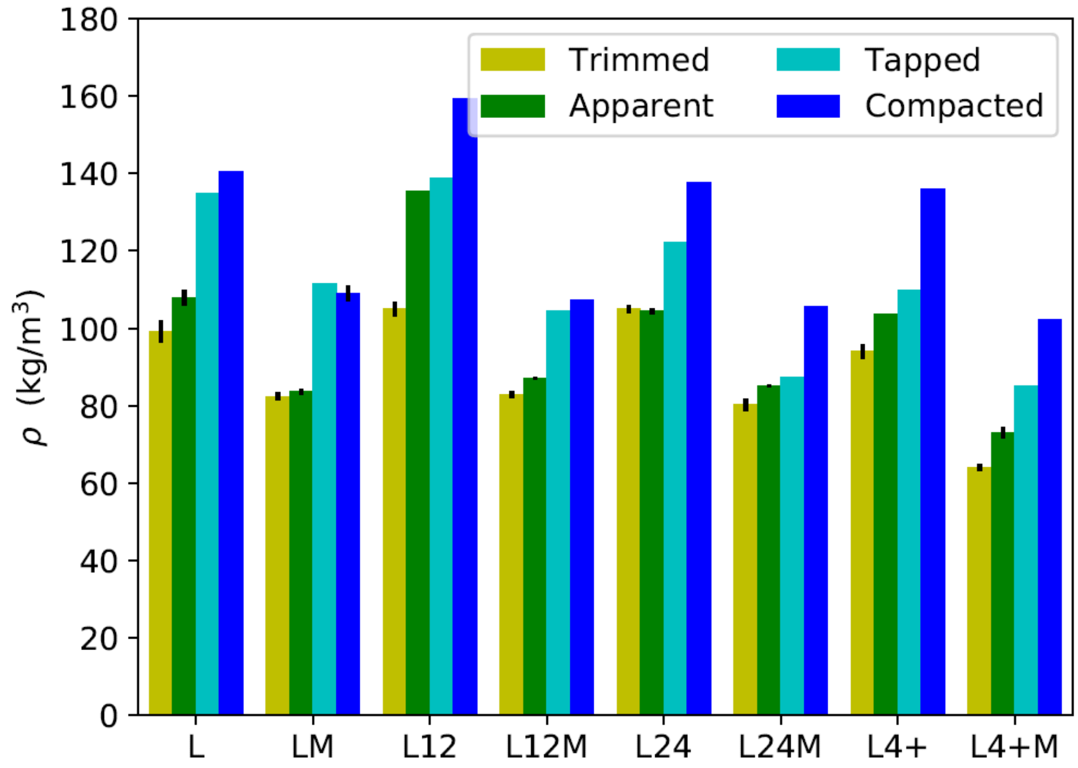
→ α, R, ρ and K measurement

→ Indirect characterisation of tortuosity and characteristic lengths

III. Multiscale characterization

1. Bulk density

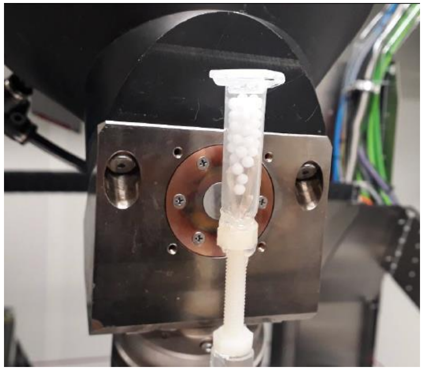
- **Trimmed:** Particles dropped in a cylinder and levelled
- **Apparent:** Particles dropped in a cylinder which is overturned 10 times
- **Tapped:** Automated process using Autotap (vertical shakes) and Geopyc (horizontal rotation and agitation) devices
- **Compacted:** Manual compaction up to saturation

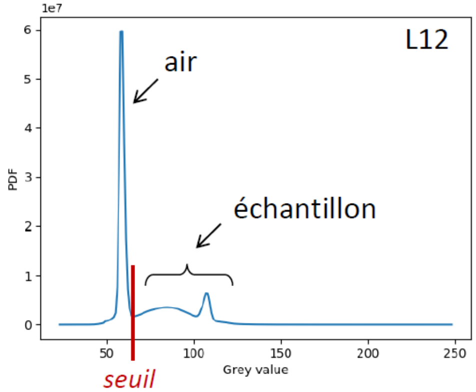


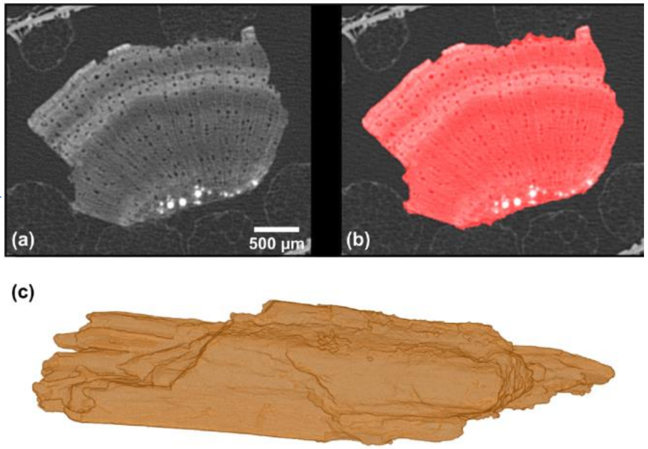
→ The 2 tapped methods lead to similar results
→ ρ in the range [60; 160 kg.m^{-3}]
→ $\rho_{trimmed} < \rho_{apparent} < \rho_{tapped} < \rho_{compacted}$
→ Lower densities for immersed samples (15 to 30%)
→ Lower densities for larger particle size distribution

III. Multiscale characterization

2. Particle density



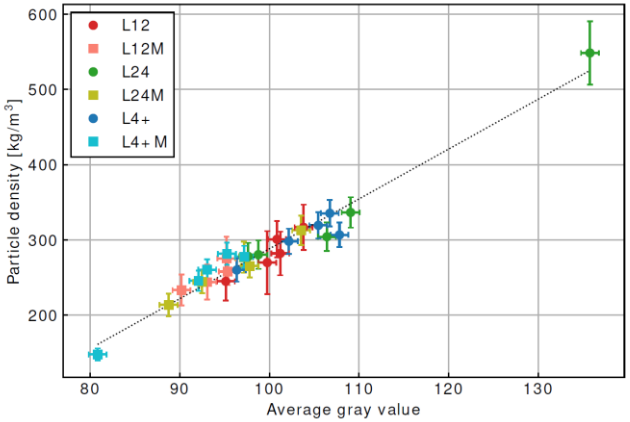




- 230 kV micro focus source
- Voxel size: 12 µm
- Scan duration: 12h

- Denoising
- Segmentation (Gray level)
- Volume evaluation

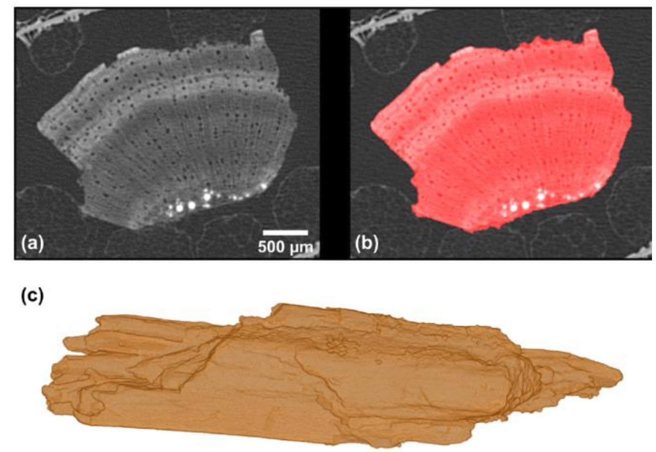
→ Sensitivity to gray level thresholding ~ 1 to 4,5%
→ Uncertainty in volume evaluation estimated at 5%
→ Uncertainty in density evaluation estimated between 1% (L4+) and 11% (L12)
→ Consistency between Gray values and Particle densities (validation of the evaluation, including outliers)

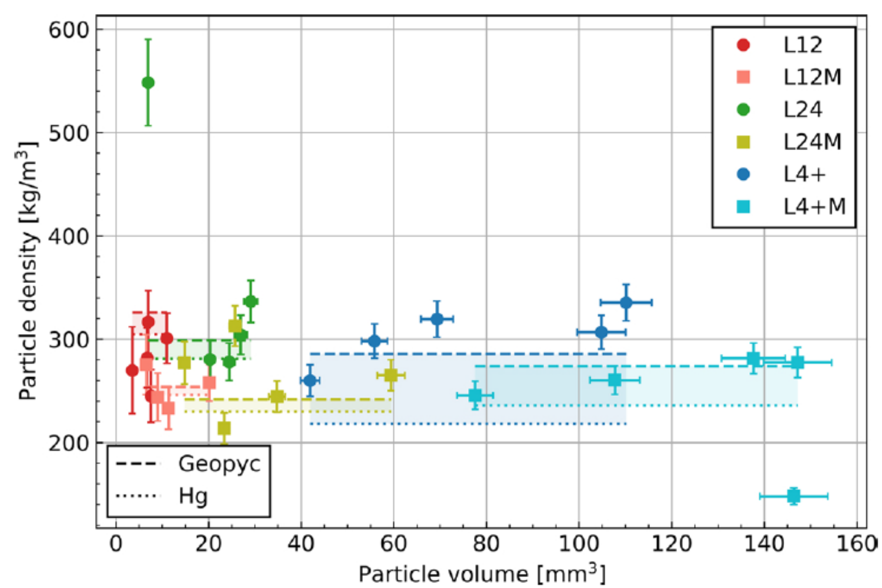


III. Multiscale characterization

2. Particle density

- **Geopyc:** Powder pycnometry (7,6 cm^3 volume)
- **Hg:** Mercury intrusion at low pressure (3,54 cm^3 volume)
- **XRCT:** X-ray computed tomography (5 particles)





→ $\rho_{particle}$ in the range [210; 330 kg.m^{-3}]

→ $\rho_{Hg} < \rho_{Geopyc}$: part of interparticle pores not filled by Hg at low pressure?

→ Regarding XRCT, values globally consistent but not statistically representative

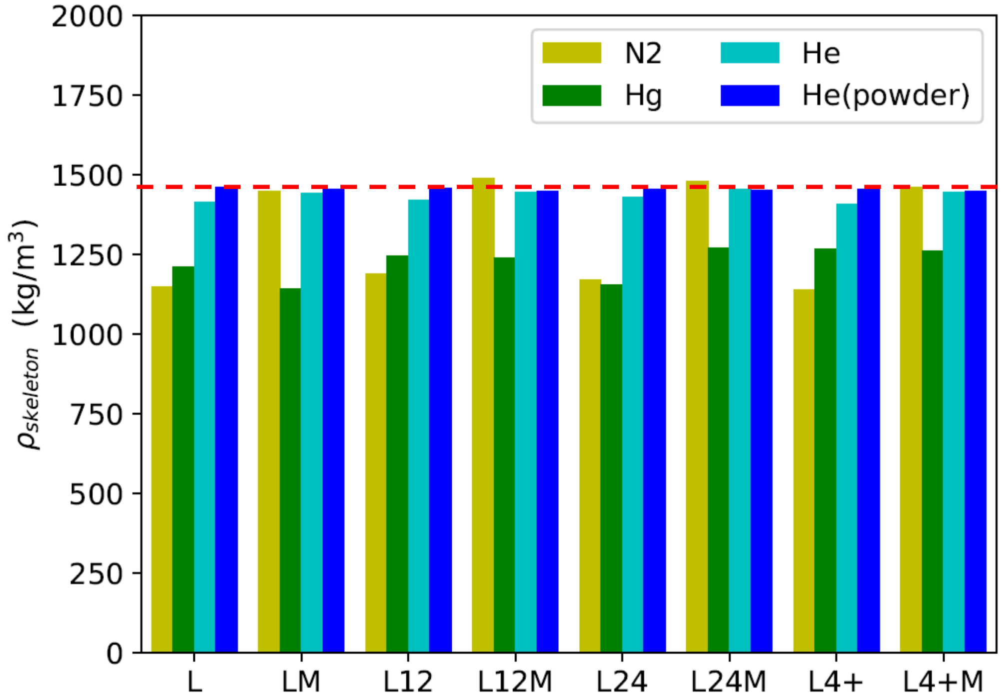
→ Decrease of particle density for immersed shiv

→ Larger particles have lower density (not confirmed by XRCT): original position in stem

III. Multiscale characterization

3. Skeleton density

- **N2:** Nitrogen pycnometry on particles (10 cm^3 volume)
- **Hg:** Mercury intrusion at high pressure (3,54 cm^3 volume)
- **He:** Helium pycnometry on particles (10 cm^3 volume)
- **He (powder):** Helium pycnometry on finely ground particles (100-500 µm)



→ $\rho_{skeleton}$ in the range [1100; 1500 kg.m^{-3}]
→ ρHe (powder) is remarkably constant: 1455 ± 5 kg.m^{-3}
→ ρN2 < ρHg < ρHe < ρHe (powder)
→ ρN2 < ρHe: smaller pores accessible to He, but not to N2
→ Increase of ρN2 and ρHe for immersed shiv: opening of pores

III. Multiscale characterization

4. Pore size distribution

- From Hg intrusion

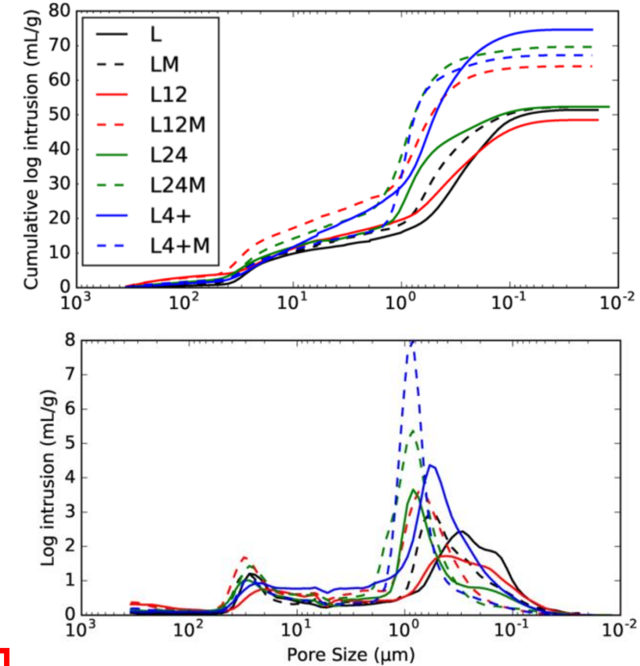
→ Ink bottle effect confirmed (extrusion plot not presented here)

→ Two groups of pores are detected:

  → Between 10 and 50 µm: Vessels (lit: 50 µm), tracheids (lit: 10-20 m)

  → Between 0,02 and 2 µm: Pits (lit: 1-2 µm)

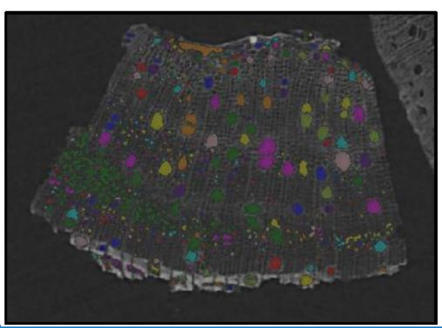
→ Shift towards larger size for immersed shiv : Bigger pores related to the opening of the microstruture

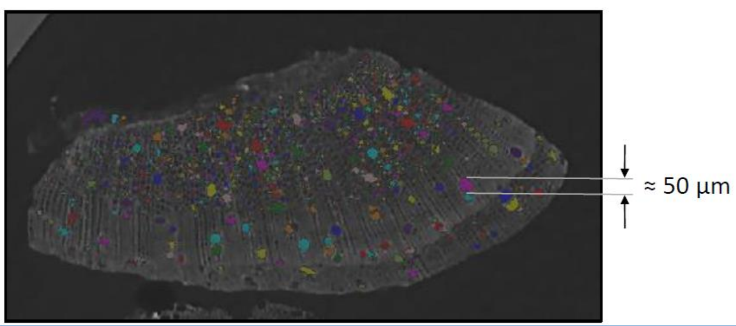


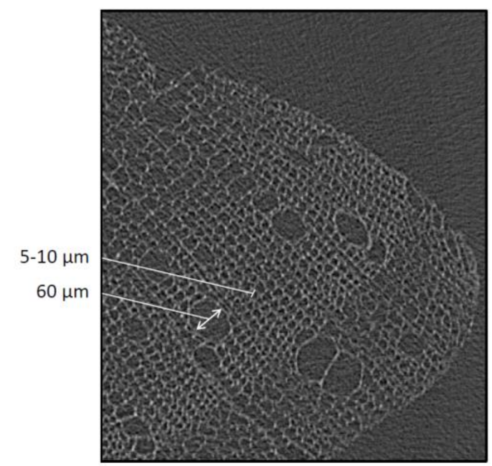
- From XRCT

→ At reference resolution (voxel of 12 µm³): only vessels are detectable

→ Attempt at higher resolution (voxel of 1,3 µm³): week absorption







IV. Modelling applications

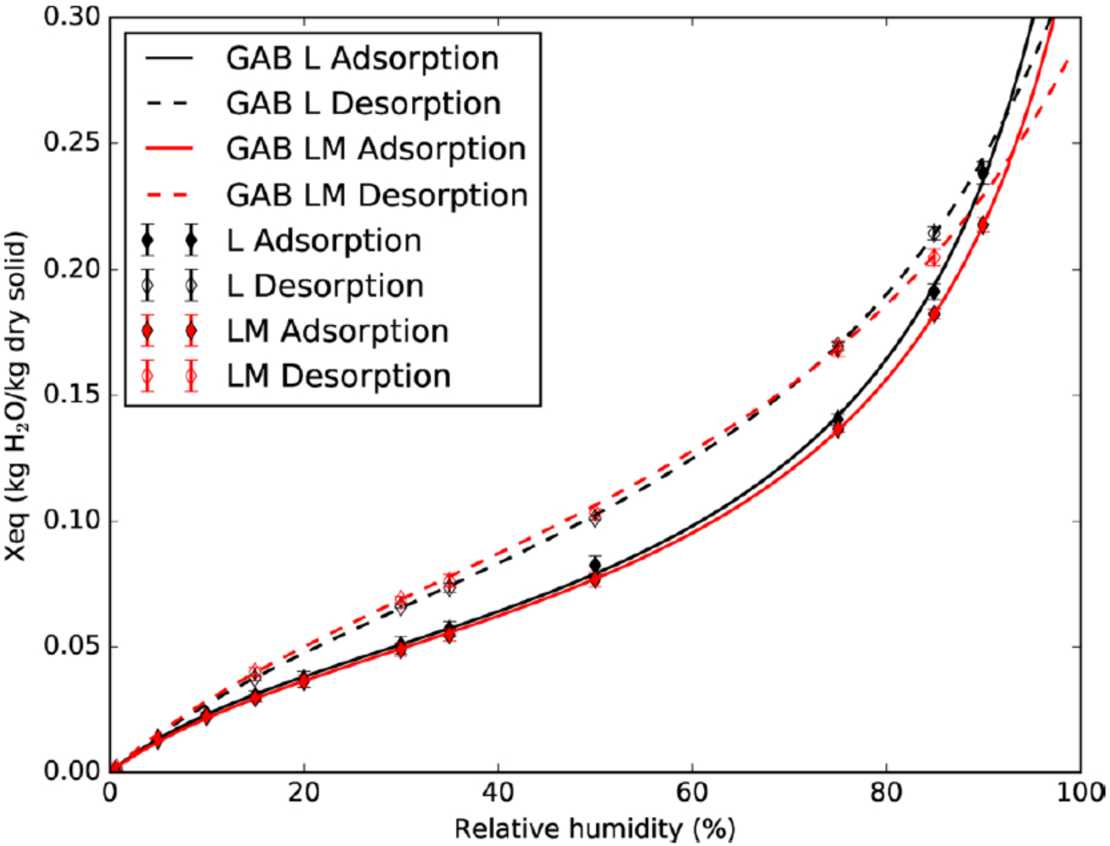
	Bulk density ρ_B			Particle density ρ_P (kg.m^{-3})		Skeleton density ρ_S		
	Trimmed	Apparent	Compacted	Geopyc	Acoustic	N_2	He	He *
L	99	108	140	330	478	1150	1413	1460
LM	82	84	109	210	443	1450	1441	1455
L12	105	136	159	326	515	1190	1420	1457
L12M	83	87	107	254	469	1490	1445	1450
L24	105	104	138	299	502	1170	1430	1456
L24M	80	85	105	242	441	1480	1454	1452
L4+	94	104	136	286	447	1140	1407	1455
L4+M	64	73	102	274	396	1460	1445	1450

* milled/ground particles

IV. Modelling applications

1. Water sorption

- Good fit of the experimental data by GAB model
- No clear difference between raw and immersed samples



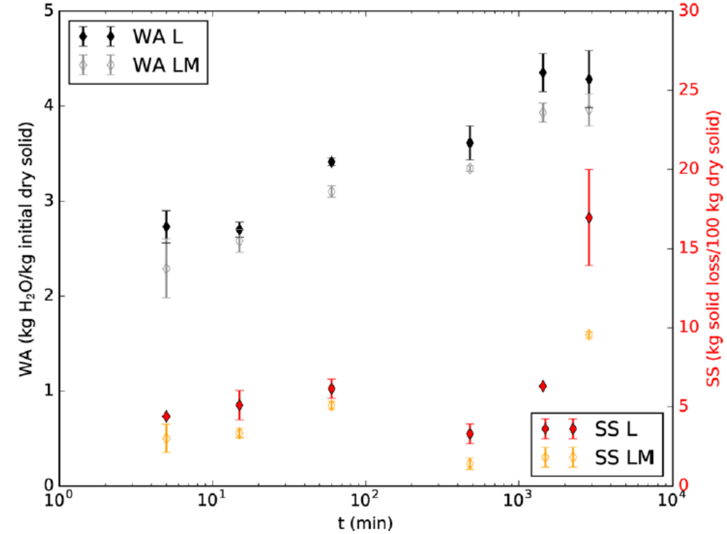
IV. Modelling applications

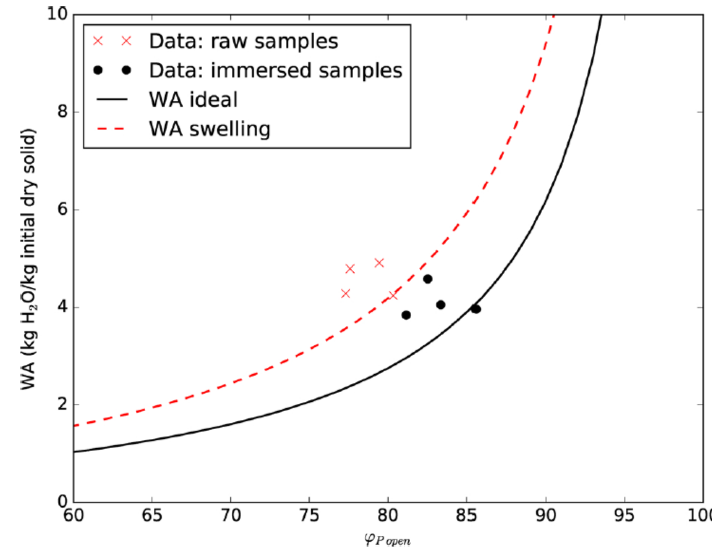
2. Water absorption by immersion

- Maximum water absorption is mainly correlated to intra porosity

- Swelling must be taken into account: preliminary immersed particle keep their initial swelling / not the case for pristine particles

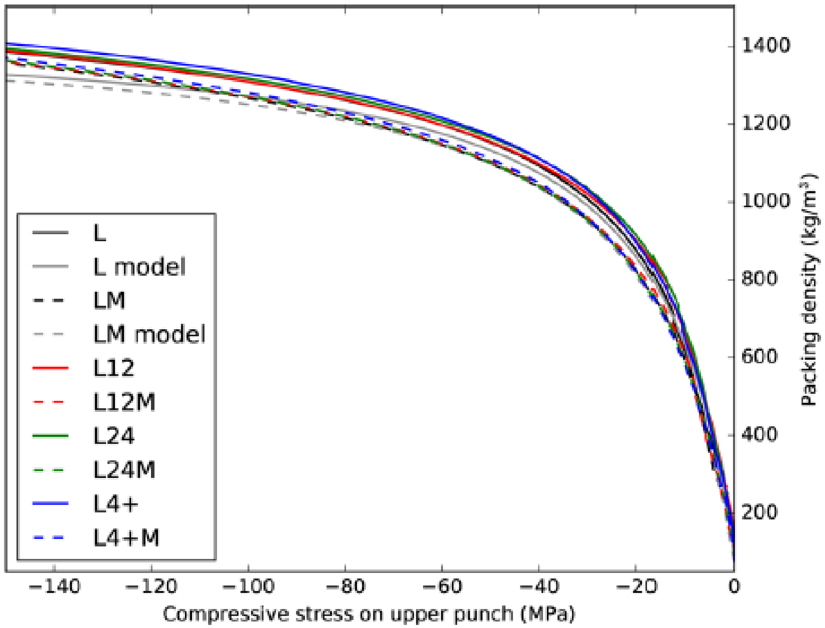
$$WA = \frac{\varphi_{P-open} * \rho_{water} * (1+s)^3}{(1 - \varphi_{P-open})\ \rho_S}$$

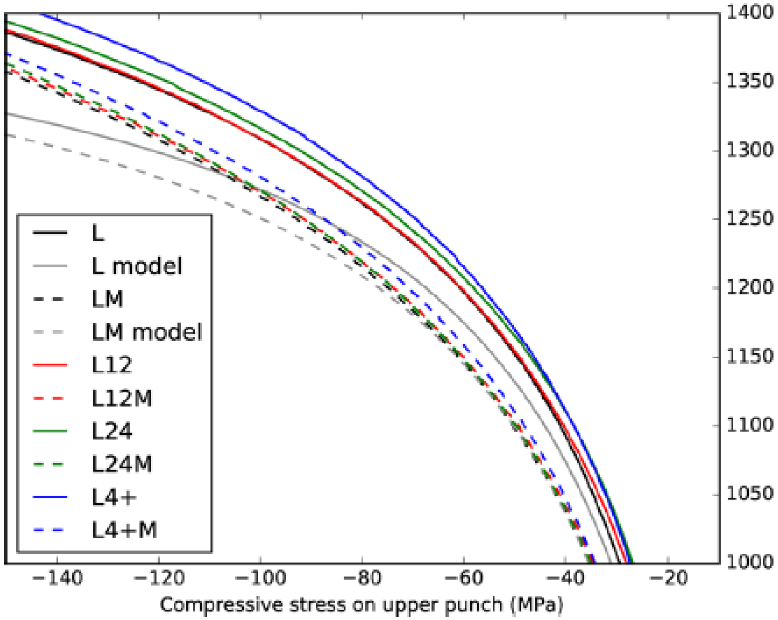




IV. Modelling applications

3. Compression behavior





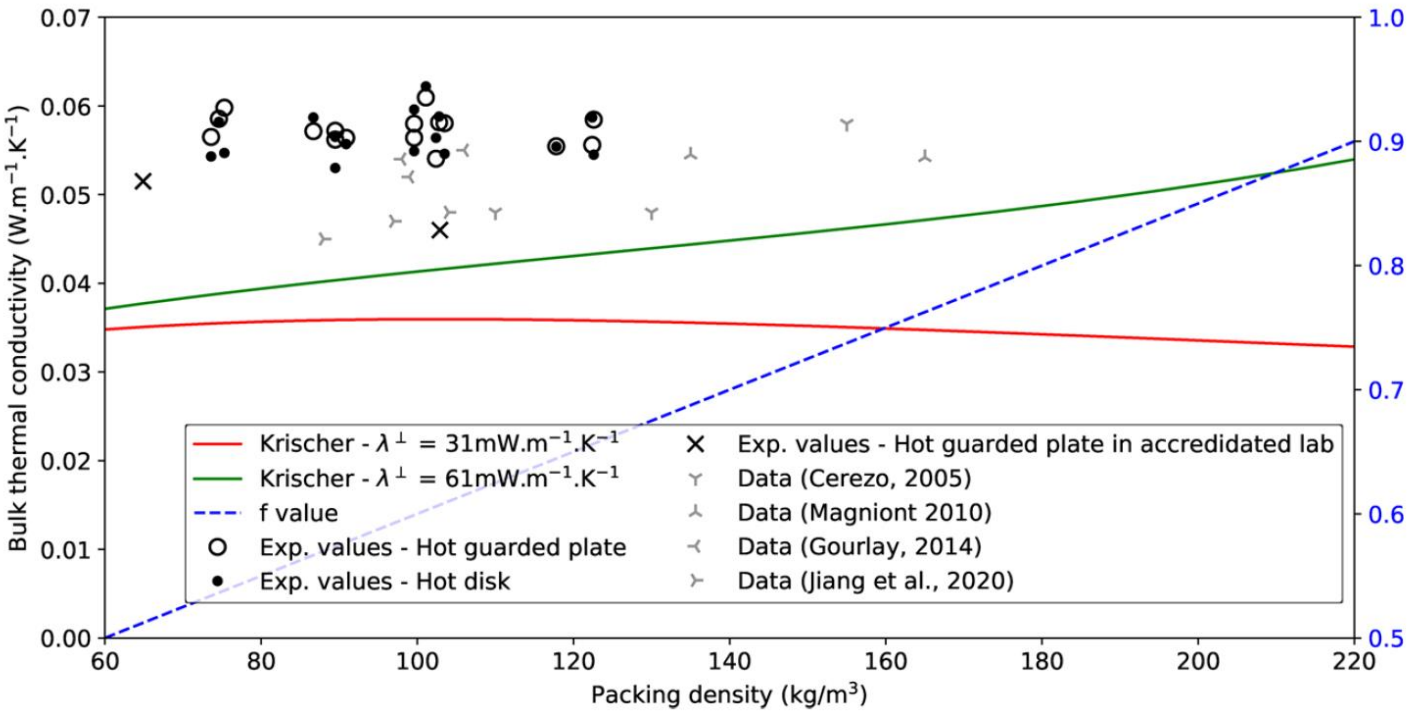
Cooper and Eaton Modeling

$$\rho = \left[\frac{1 - exp(P_r/\sigma_{UP})}{\rho_{B-trim}} + \frac{exp(P_r/\sigma_{UP}) - exp(P_d/\sigma_{UP})}{\rho_P} + \frac{exp(P_d/\sigma_{UP})}{\rho_S}\right]^{-1}$$

IV. Modelling applications

4. Thermal behavior

$$\lambda_B = \left[f * \left(1 \middle/ \lambda_{air} + \frac{\rho \left(1/\lambda_{P\perp} - 1/\lambda_{air}\right)}{\rho_P}\right) + (1-f) \middle/ \lambda_{air} + \frac{\rho}{\rho_P}\left(\lambda_{P//} - \lambda_{air}\right) \right]^{-1}$$



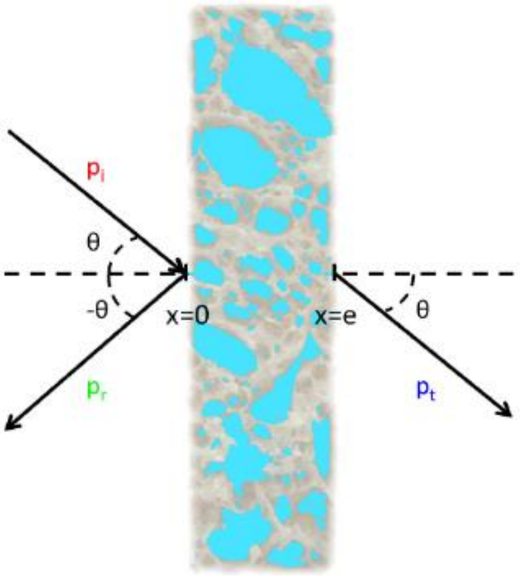
Experimental values quite far from theoretical curves

IV. Modelling applications

5. Acoustic properties

- General case: 2 waves in solid phase + 1 wave in fluid phase [Biot, 1956]
- Simplified approach: Rigid frame is considered (1 wave in fluid phase), with an isotropic behaviour

→ Dissipation through visco-inertial (ρ) and thermal effects (K)
→ 4 parameters are used to describe the material:
  - Porosity: ϕ_{acou}
  - Resistivity: σ
  - Tortuosity: α_∞
  - Characteristic length: Λ

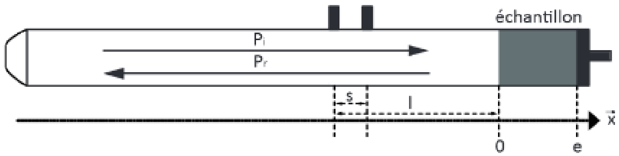


- Acoustic model

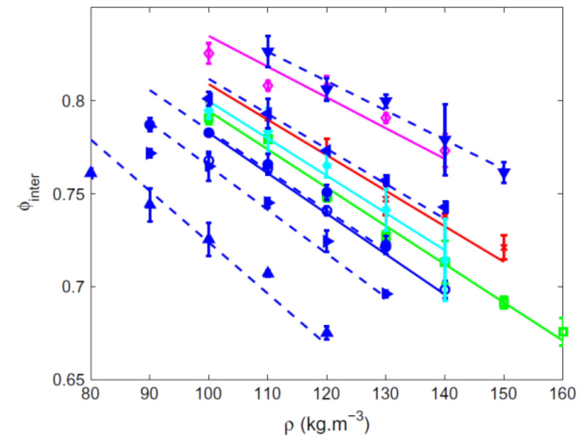
→ Viscoinertial effects [Johnson et al. 1987]
→ Thermal effects [Zwikker & Kosten 1949]

IV. Modelling applications

5. Acoustic properties



Re(K)



Linear regression

$$\phi_{inter} = 1 - \frac{\rho}{\rho_{particle}}$$

- **Initial 'blind' approach:**

- Particle density is not known
- Assumption of a single level of porosity
- Hypothesis H0: $\Phi_{acou} = \Phi_{inter}$
- Evaluation of an acoustical density

	Particle density ρ_P (kg.m^{-3})	
	Geopyc	Acoustic
L	330	478
LM	210	443
L12	326	515
L12M	254	469
L24	299	502
L24M	242	441
L4+	286	447
L4+M	274	396

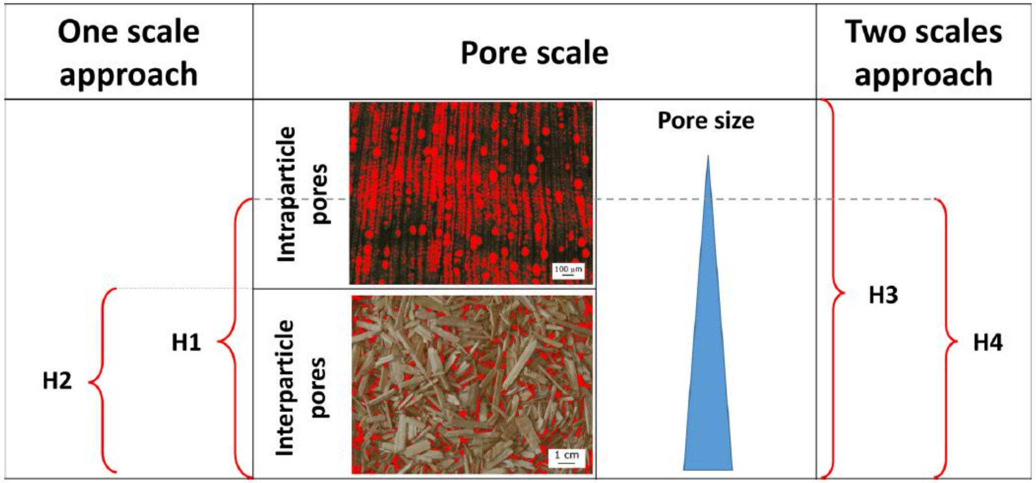
→ Acoustic densities are higher than particle densities
→ The tendencies are respected (size and immersion effects)
→ Hypothesis H0 is not true

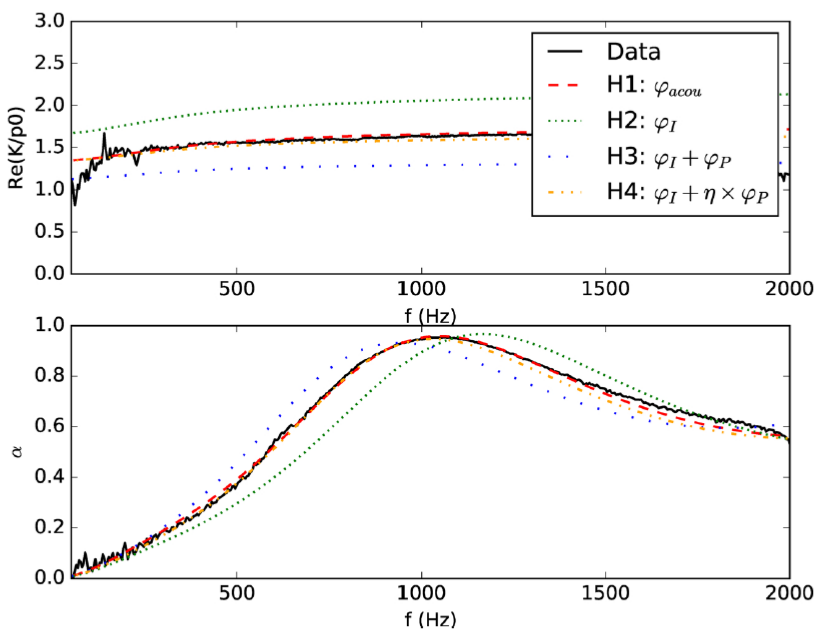
IV. Modelling applications

5. Acoustic properties

- **Corrected approach:**

- Particle density from Geopyc
- 4 hypotheses / behavior
- One or two porosity scales considered [Olny & Boutin 2003]





→ All open pores do not take part to acoustic dissipation
→ Acoustic dissipation is not only due to inter particle pores
→ Interparticle pores + a fraction of the intraparticle pores are involved in sound dissipation
→ Two approaches are satisfying, but the (partial) double porosity is physically more accurate

Conclusion and Outlooks

❑ Conclusions

- ✓ Investigations on the densities and microstructures of plant particles used for biobased building applications at three scales: packing, particle and skeleton
- ✓ Application on complementary methods of characterization:
  - ✓ Powder pycnometry : efficient and representative to characterize particle density.
  - ✓ Mercury intrusion porosimetry : validation of the range of densities + PSD
  - ✓ X-ray computed tomography : characterization of individual particles + PSD
- ✓ Analysis of particle size and immersion effects:
  - ✓ Smaller particles have higher packing and particle densities
  - ✓ Shiv aged through immersion in water present lower packing and particle densities and higher skeletal density
- ✓ Correlation of the multiscale densities with mechanical, hygro-thermal, and acoustical behaviors

❑ Outlooks

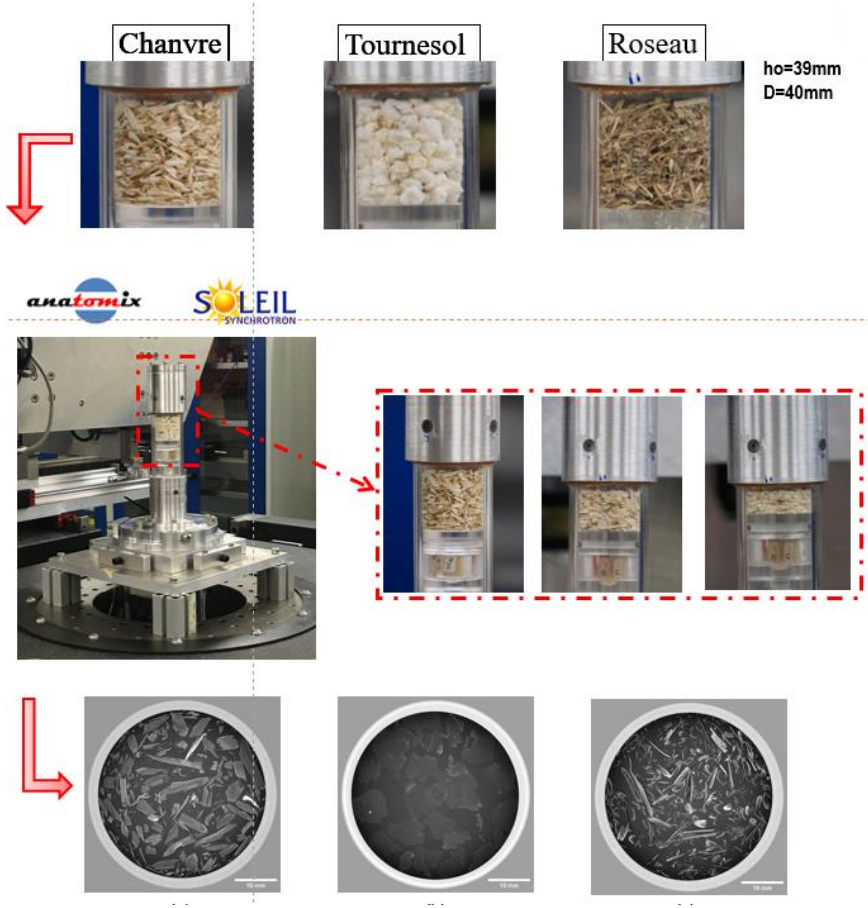
→ Tomographic observations at high resolution to characterize the morphology and the evolution of porosity

→ Assessment of the impact of the densities on the physical properties

In progress

❑ Synchrotron SOLEIL

- ✓ Experimental campaign (2021)
  - ✓ Beamline Anatomix (resolution : 20nm-20µm)
  - ✓ Energy: 5 to 50keV
  - ✓ Beam size ~ 40mm
- ✓ Materials: shiv, sunflower and reed
- ✓ Objectives
  - ✓ Assess the evolution of the particles with compression and moisture level
  - ✓ Characterize interparticle porosity (shape and size)
  - ✓ Understand the micromechanisms inside particles (closing of cells)
- ✓ Work on images
  - ✓ In progress: PhD A. Kouakou (2022-2025)



Thank you for your attention…

…any questions?

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