Microstructural characterization and numerical evaluation of the effective elastic properties of oolitic rocks

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GeoRessources Laboratory
University of Lorraine (France), CNRS

- GeoRessources laboratory combines most of the Nancy applied geology resources
- Total staff = 180 persons

- Research activities focused on geological resources
- Resources, exploration and exploitation: cover all phases from initial processing through to delivery and use and would help to evaluate its impacts on society and the environment
- Pluridisciplinary laboratory: geology, geochemistry, sedimentology, geophysics, rock mechanics and heat and mass transfer processes, geomodelling...
“Multi-Scale Hydrogeomechanics” team

Experimentation

- **Leaders**: A. Giraud/ F. Golfier
- **3 PR/ 6 Ass. PR / 4 technical staff**
- **High level experimental platform**
- **Tools for modelling and parallel computing**

**Research themes:**
- Behaviour of fractured media
- Reactive transport
- Multiscale coupled modelling

**Applications**: Underground storage (radioactive wastes, gas: CO₂, H₂), depollution and vulnerability of aquifers, petroleum reservoirs (hydraulic fracturing...), safety of underground works, slope stability...

Multi-scale modelling

- **Pollutant plume and gravitational digitation**
- **Bacterial growth in porous medium**
- **Cracks network non-compliant to the mesh and modelling of the hydromechanical behaviour with FM/XFEM methods**
- **Simulation of the hydraulic stimulation with coupled method DEM/FVM**

- **Nano-indenteter** (characterization of micromechanical properties of rocks)
- **Rock sample with displacement sensors**
- **Percolation test**
- **Gas chromatography**
General framework: THMBC coupling in porous rocks

**THMBC** modelling and laboratory experiments whose date are used to better understand physical mechanisms and to feed multi-scale models:
- micro models,
- macro models (i.e., phenomenological),
- macro models enriched with micromechanics.

**Concerned rocks:** porous sedimentary rocks
- carbonated (ooliticimestones, micritic limestones, dolostones),
- detritic (oolitic iron ore, sandstone),
- argillaceous (argillites, marls).


**Oolitic iron ore**

**Oolitic limestone from Euville**

**Claystone**
- **Underground works/structures:**
  - Storage reservoirs ($CO_2$ and $H_2$, radioactive wastes, hydrocarbons)
  - Mines (iron ore, coal, salt…)

- Rocks are exposed to **Thermo-Hydro-Mechano-BioChemical solicitations** that can lead to a very complex overall response of the system via couplings:
  - Excavation
  - Ventilation, flooding
  - Heating
  - Gaz injection
  - Mineralogical transformations

- Need characterizations of the porous medium:
  - Mechanical
  - Geophysical
  - Petrophysical
  - Physico-chemical
  - Microstructural, Mineralogical

*Schematization of HMBC couplings around underground mines ($T$ is constant)*
Poromechanical Problems: Underground Mines

- Underground iron mines in Lorraine (North-Eastern France), depth up to 250m

- Rooms and pillars system (partial extraction method) → Safety pillars under sensitive zones.

- Mine drainage (dewatering) during mining work → Lowering of the ground water-table.

- Underground mine ventilation → Relative Humidity $RH = 70$-$85\%$, → Drying of rocks.

- Mines are abandoned during 1990s → End of mine dewatering, → Water-table rising, → Flooding of the mine, → Resaturation of rocks.

- Mine collapses (failure of iron ore pillars).
Poromechanical Problems: Underground Mines

- Posed problem: excavation + hydrous cycle (initial saturation-ventilation-flooding) \(\rightarrow\) HM coupling

- **Initial state**: saturation

- **1st perturbation**: excavation
  \(\rightarrow\) EDZ
  \(\rightarrow\) Permeability

- **2nd perturbation**: ventilation (drying)
  \(\rightarrow\) Parsat zone
  \(\rightarrow\) Stresses \(\Delta\)
  \(\rightarrow\) EDZ
  \(\rightarrow\) Permeability

- **3rd perturbation**: flooding (wetting-resaturation)
  \(\rightarrow\) Mine flooding
Poromechanical Problems: Underground Mines

1. Introduction
2. Micromechanical properties
3. Microscopic observations
4. Microstructural characterizations
5. Effective properties
6. Conclusions

Intact iron ore \((Fe^{2+})\)

Aged oxidized iron ore \((Fe^{3+})\)

Ferriarenite (iron-rich) facies

Initial iron ore

Aged (70-100 years) iron ore

Oxidation time

<table>
<thead>
<tr>
<th>Oxidation Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 years</td>
</tr>
<tr>
<td>70 years</td>
</tr>
<tr>
<td>100 years</td>
</tr>
</tbody>
</table>
Poromechanical Problems: Radioactive Waste Repository

- Deep geological formation: Callovo-Oxfordian claystone (argillite) of the Paris (France) basin.
- Meuse/Haute Marne Underground Research Laboratory (Bure, North-Eastern France) of ANDRA (French agency for nuclear waste management): 500 m deep
- Posed problem: **Thermo-Hydro-Mechanical couplings**

Desaturation and induced fractures
→ **EDZ** (Excavation Disturbed/Damaged Zone)
→ Alteration of mechanical and hydraulic (permeability increase) properties

Corrosion of metal containers
→ Production of \( \text{H}_2 \) → Pressure increase
→ Damage of surrounding rock
→ Leakage of radionuclides

Heating of saturated poroelastic rock by waste packages
→ **Thermal fracturing** by effective stress
→ Permeability increase

**Vertical cross section of the EDZ with the different induced fractured zones**
Poromechanical Problems: Geological Storage of CO₂

- CO₂ injected in supercritical state.

- Different options:
  Deep saline aquifers
  Depleted oil and gas reservoirs
  Coal seems…

1.Introduction
2.Micromechanical properties
3.Microscopic observations
4.Microstructural characterizations
5.Effective properties
6.Conclusions

Geological Storage Options for CO₂
1 Use of CO₂ in Enhanced Coal Bed Methane Recovery
2 Deep unminable coal seams
3 Depleted oil and gas reservoirs
4 Use of CO₂ in Enhanced Oil Recovery
5 Deep unused saline water-saturated reservoir rocks
6 Other suggested options (basalts, oil shales, cavities)

Different options for storing CO₂ in deep underground geological formations (Cook 1999)

P-T phase diagram of CO₂
Poromechanical Problems: Geological Storage of CO\(_2\) in deep saline aquifers

- CO\(_2\) injection (HP/HT) in saline water-saturated reservoirs rocks with impermeable caprocks.

- Reservoir rocks: **Hydro-Mechanical** problem due to variations in effective stress.

- Caprocks: **Hydraulic fracturing** since CO\(_2\) plume can reactivate/create fractures.
Poromechanical Problems: Geological Storage of \( \text{CO}_2 \)

**Focus**
- Study of long-term **mechanical and petrographic evolutions** during \( \text{CO}_2 \) storage.
- Studied rocks: carbonated (oolitic limestone from Lavoux).

**Low- and high-magnification scanning electron microscopy observations on initial oolitic Lavoux limestone**

*(the first image corresponds to a polished thin section)*
Poromechanical Problems: Geological Storage of CO₂

Experimental device
- Injection of CO₂ under different states (diffusion, dynamic percolation under gaseous/supercritical state, dissolved in an aqueous solution).

- Application of high triaxial stresses, pressures and temperatures.

**Experimental device for the percolation of fluids (aqueous solutions, gas) under high stresses, pressures and temperatures.**
Poromechanical Problems: Geological Storage of CO₂

X-ray 3D nano Computed Tomography
Poromechanical Problems: Geological Storage of CO₂ in coal seams

- **Double porosity** medium
  - Macro: face and but cleats (fractures)
    - Darcy flow
  - Micro: inside coal matrix
    - Sorption mechanism

- Hydro-Chemical coupling:
  Sorption on coal matrix depends on gas pressure in fractures

- Permeability/porosity depend on:
  - **Swelling/shrinkage of matrix**
    - Langmuir adsorption isotherm
  - **Variations of gas pressure in macro-pores**
    - Hydro-Mechanical coupling (effective stress)

- Swelling/shrinkage = main mechanism of gas production/sequestration in coal matrix

- Geological Storage of CO₂ in coal seams:
  - (Thermo-)Hydro-Mechano-Chemical problem
Poromechanical Problems: Geological Storage of CO₂ in coal seams

- Non-linear isotropic poroelastic equation of the stress tensor as a function of drained parameters:
  \[
d\sigma = \lambda_0 \text{Tr}(d\varepsilon)I + 2Gd\varepsilon - (b LP_l + b GDP_g)I - 3\alpha_0 K_0 dT I
\]
  with
  \(b_L, b_G\) Biot’s coefficient for liquid and gas
  \(\alpha_0\) coefficient of drained thermal expansion

- Variation of Eulerian porosity \(\varphi\):
  \[
d\varphi = (b - \varphi) \left(d\varepsilon_v - 3\alpha_0 dT + \frac{dP_g - S_l dP_c}{K_s}\right)
\]

- Langmuir adsorption isotherm: written in terms of volumetric strain \(\Sigma_V\)
  \[
  \varepsilon_V = \varepsilon_L \beta_L P_g / (1 + \beta_L P_g)
  \]

- Homogenized equation for the Eulerian porosity:
  \[
d\varphi = (b - \varphi) \left(d\varepsilon_v - 3\alpha_0 dT + \left(1/K_s - \frac{\varepsilon_L \beta_L}{(1 + \beta_L P_g)^2}\right) dP_g - S_l/K_s dP_c\right)
  \]
  **Temperature** \hspace{1cm} **Langmuir isotherm**

- Permeability/porosity empirical relation (Kozeny-Carman’s formula):
  \[
k/k_0 = (\varphi/\varphi_0)^3
  \]
Poromechanical Problems: Geological Storage of CO₂ in coal seams

- Modelling of Enhanced Coal Bed Methane (ECBM) Recovery

→ Homogenized equation for the Eulerian porosity with the generalized Langmuir isotherm

\[
d\varphi = (b - \varphi) \left( d\varepsilon_v - 3\alpha_0 dT + \frac{1}{K_s} \frac{d}{dP_g} \left( \frac{\varepsilon_{CH_4} \beta_{CH_4} y_{CH_4} - \varepsilon_{CO_2} \beta_{CO_2} y_{CO_2}}{1 + \left( \beta_{CO_2} y_{CO_2} + \beta_{CH_4} y_{CH_4} \right) P_g} \right) \right) dP_g - \frac{S_I}{K_s} dP_c
\]

- 2D numerical simulation with Code_Aster (EDF):
  Model with 2 CO₂ injection wells

Adsorption of CO₂

→ Decrease of porosity 😞

Hydro-Mechanical coupling

→ Increase of porosity 😊

Isovalues of the porosity variation in rock mass: (a) for 1 year, (b) for 5 years, (c) for 10 years and (d) for 100 years.
Poromechanical Problems: Hydraulic Fracturing

- Well stimulation technique (gas, oil), fracking (unconventional reservoir such as shale gas), enhanced geothermal systems (hot dry rock), stimulation of groundwater production…

- Injection of a high pressure fluid
  → 1. Tensile fracture first created
  → 2. Contained fracture continuously driven in the (e.g., hydrocarbon containing) formation

- Fluid *leakoff* into the formation due to the pore pressure rise
  → Expansion of the flooded region
  → Closing of the fracture due to the *backstress* (e.g., extra confining stress at the far-field)
### Poromechanical Problems: building stones

**Ages, origins, thin-section microphotographs and examples of use of selected limestones**

(Eslami et al., Construction and Building Materials, 2018)

<table>
<thead>
<tr>
<th>Stone</th>
<th>Age</th>
<th>Origin</th>
<th>Thin-section microphotographs</th>
<th>Example of Construction</th>
</tr>
</thead>
<tbody>
<tr>
<td>Massangis</td>
<td>Middle Jurassic, Bathonian stratum</td>
<td>Yonne department</td>
<td>97% calcite; 3% quartz</td>
<td>Cloister of the Abbey of Fontenay</td>
</tr>
<tr>
<td>Lens</td>
<td>Lower Cretaceous, Barremian stratum</td>
<td>Gard department</td>
<td>&gt;99% calcite</td>
<td>Maison Carré of Nîmes</td>
</tr>
<tr>
<td>Migné</td>
<td>Miocene, Burdigalian stratum</td>
<td>Vienne department</td>
<td>&gt;99% calcite</td>
<td>Church Notre-Dame-la-Grande of Poitiers</td>
</tr>
<tr>
<td>Savonnières</td>
<td>Upper Jurassic, Portlandian stratum</td>
<td>Meuse department</td>
<td>&gt;99% calcite</td>
<td>Cathedral of Saint-Dizier</td>
</tr>
<tr>
<td>Saint-Maximin</td>
<td>Eocene, Lutetian stratum</td>
<td>Oise department</td>
<td>90% calcite; 10% quartz</td>
<td>Chapel of Chantilly</td>
</tr>
</tbody>
</table>
Poromechanical Problems: building stones

Building stone abundance ranked by the number of churches in which the stones are found.

Note that this not reflect the total quantity of stone use. Black bar: West Sussex stone; grey bar: non-local stone.

(Bone, Proceedings of the Geologists’ Association 127, 2016)
Poromechanical Problems: building stones

Weathering/ageing

- **Weathering** of stones (limestones, sandstones, marble...) used in historical monuments after several decades in an urban/ambient environment.
- **Temperature** (fire), **humidity** can cause significant **physical, chemical and mineralogical changes** to stones.
- The **alteration** leads to a heterogenization of the limestone, in term of morphology (roughness and porosity) and phases distribution.
- Mechanical behaviour of microporous carbonates are strongly controlled by the microporosity distribution within the grains, at the origin of variations in elastic properties, mechanical strength and failure mode.

Micromechanical approach

- Produce new lime-based mortars in the same way as the historic ones (monuments) were made.
  ➔ Comprehension of microstructural effects and upscaling with micromechanics (or effective media theory) is useful to predict basic properties of lime-based mortars in tension and compression.
- The multi-scale methodology (e.g., upscaling models) allows to establish the links between nano- to micro-observations and the macroscopic behaviour of the stone.
  ➔ Micromechanical or poro-micromechanical (because porosity is very important) models are necessary.
General Framework:
- Macroscopic thermo-mechanical behavior of geomaterials
- Micro-macro relationship / micromechanical models
- Homogenization theory (Maxwell)
- Effective properties

Methodology:
- Characterization of the geometry of the microstructure of porous heterogeneous rocks
- Micromechanical properties of components
- Simplified Model: approximation of components
- Verification of approximation: compliance contribution tensors
- Use of these tensors to evaluate effective properties

Micromechanical properties: Nanoindentation tests

- CSM device nanoindenter is used: Evaluation of Elastic Modulus.

Oliver Pharr theory

\[
\frac{E}{E_r} = \frac{S \sqrt{\pi}}{2 \beta \sqrt{Ap(hc)}}
\]

\[
\frac{1}{E_r} = \frac{1 - \nu_s^2}{E_{it}} + \frac{1 - \nu_i^2}{E_i}
\]

\[
A_p = 24.5 h_c^2
\]

E\_r: the reduced elastic modulus
E\_it and \( \nu_s \): elasticity modulus and Poisson’s coefficient of the material
E\_i and \( \nu_i \): elasticity modulus and Poisson’s coefficient of the indenter.

P: Load
S: Contact stiffness
Ap: projected contact area
Protocol:
set of nanoindentation tests covering all the phases of the material:

400 indentations / entire sample using grid indentation technique.

<table>
<thead>
<tr>
<th>Set</th>
<th>I</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximal load (mN)</td>
<td>75</td>
</tr>
<tr>
<td>Loading rate (mN/min)</td>
<td>100</td>
</tr>
<tr>
<td>Unloading rate (mN/min)</td>
<td>100</td>
</tr>
<tr>
<td>Pause (seconds)</td>
<td>10</td>
</tr>
</tbody>
</table>
Elastic modulus from 7 to 71 Gpa

- 3 Peaks means 3 material phases (separated from the whole histogram with a deconvolution algorithm):
  - Phase 1: Inner layers of oolites (micro-calcite).
  - Phase 2: Outer layers of oolites (micro-calcite).
  - Phase 3: Inter-oolitic sparry calcite (macro-calcite).
Microscopic observations

1- X-Ray Nanotomography:
Nanotomograph in GeoRessources Laboratory is used: phoenix nanotom with 500 nm minimal resolution.

2- SEM (Scanning electron microscopy):
Microscope of GeoRessources laboratory with resolution of 1 nm for 15 Kv.

Application on oolitic rocks:
- Lavoux limestone
- Iron ore

![Lavoux limestone](image1)
![SEM observations](image2)
![Iron ore](image3)
X-Ray tomography observations:

Image processing concept
REV selection

- Threshold algorithm
- Surface determination (determination of voids and material)
- Grayvalue histogram analysis
- Defects (voids or inclusion) detection (VGStudio Max module)

Lavoux limestone
Sample: Height = 10 mm
Voxel resolution = 5 μm

REV: Side-cube: 2200 – 2400 micrometers

Porosity: 8.3%
Comparison of tomography porosity (5 µm resolution) with mercury intrusion porosimetry

Porosity ~ 7%

Accessible porosity with x-ray tomography because of the resolution (5 µm)

Porosity distribution based on the pore radii access
2400 Elements
Min= 6,12 μm
Max= 130 μm
Mean=21,72 μm
Standard dev= 15,12

2400 Elements
Min= 1000 μm³
Max= 2,27*10^6 μm³
Mean=28 181,66 μm³
Standard dev= 126500

Pores Volume Distribution

Pores Radius Distribution

Lavoux limestone

Pores: statistical analysis
Distance between pores - distribution:

Pores: statistical analysis

Number of pores: 3088
Average = 73.9 µm
Standard deviation = 40.86 µm

Distance (µm)

Density

Data
Analysis of REV by VGStudio Max © (image processing software)

Lavoux limestone is composed by 3 components:

- **Oolites**: Spherical shape?
- **Pores**: Ellipsoidal and/or concave shape?
- **Inter-oolitic cement**

|----------------|-----------------------------|-----------------------------|-----------------------------------|----------------------|-----------------|

![Image of microscopic observations](image-url)
Homogenization Theory

3 phase material composed of:
- poroelastic grains (oolites): Spheres
- inter-granular solid crystals (cement)

Simplified model*:
- Spherical shape for oolites
- Ellipsoidal approximation for all pores

X-ray nano-tomography → more realistic shape of components

Novelty of this work:
take into account the irregularly shaped 3D pores which are modeled by ellipsoids with the principal component analysis PCA

Step I:
micro (intra-oolitic pores) → meso
Homogenization of micropores in oolites (o)
Self consistent approximation

Step II:
meso (oolite + inter-oolitic pores + cement) → macro (REV)
Maxwell homogenization scheme

*Effective thermal conductivity of oolitic rocks using the Maxwell homogenization method, 2015
A. Giraud, I. Sevostianov, F. Chen, D. Grgic

Approximation of an irregular shape by an ellipsoid:

- Based on PCA (principal component analysis)
- Based on Inertia Moments
- Gives the principal directions of the ellipsoid

Principle of PCA method

\[
C = \begin{bmatrix}
cov(X,X) & cov(X,Y) & cov(X,Z) \\
cov(Y,X) & cov(Y,Y) & cov(Y,Z) \\
cov(Z,X) & cov(Z,Y) & cov(Z,Z) \\
\end{bmatrix}
\]

\[
C = Q \Lambda Q^T
\]

\[
\begin{bmatrix}
a \\
b \\
c
\end{bmatrix} = 2 \times \sqrt{\text{EigenValues}} (C)
\]

C: covariance matrix
Q : eigenvectors matrix
\Lambda: eigenvalues (diagonal matrix)

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3D acquisition of the shape

Mesh and extracting surface points

Statistical Procedure (PCA)

Ellipsoidal approximation

Lavoux Limestone (98% calcite) and tomography is based on Graylevel (density of materials)

Difficulty of distinguishing different components automatically

Oolites characterization

Problems:
Time consuming !!!
Low sphericity (specific surface)

For the same oolite volume:
surface S2 (specific surface) > surface S1 so the sphericity ratio decreases

1st test: extract oolites manually with VGStudio Max tools

Sphericity ratio

3D view of extracted oolites

Percentage

Sphericity

0.58 0.61 0.64 0.67 0.7 0.73 0.76 7.79
Solution: Grinding Process

1. Oolites extracted manually under the binocular microscope.
2. Oolites put into a gel
3. Nanotomography scan for the total sample (gel + oolites)
4. Oolites are then approximated by ellipsoids using the PCA method
Approximation of an oolite by an ellipsoid

Oolites: statistical analysis

Sphericity ratio close to 1

→ Oolites can be approximated by spheres in micromechanical models
### Approximation of an oolite by an ellipsoid

<table>
<thead>
<tr>
<th>Oolite Ref.</th>
<th>3D acquisition</th>
<th>Mesh and extraction of surface points</th>
<th>Ellipsoidal approximation (PCA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td><img src="image1.png" alt="Image" /></td>
<td><img src="image2.png" alt="Image" /></td>
<td><img src="image3.png" alt="Image" /></td>
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<tr>
<td>5</td>
<td><img src="image4.png" alt="Image" /></td>
<td><img src="image5.png" alt="Image" /></td>
<td><img src="image6.png" alt="Image" /></td>
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<tr>
<td>4</td>
<td><img src="image7.png" alt="Image" /></td>
<td><img src="image8.png" alt="Image" /></td>
<td><img src="image9.png" alt="Image" /></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Oolithe Ref.</th>
<th>Real volume</th>
<th>Approximated volume</th>
<th>Radius</th>
<th>Approximated semi-axes</th>
<th>Sphericity ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>0.0753</td>
<td>0.0821</td>
<td>0.26</td>
<td>(0.29;0.27;0.24)</td>
<td>0.995</td>
</tr>
<tr>
<td>5</td>
<td>0.1244</td>
<td>0.1404</td>
<td>0.32</td>
<td>(0.33;0.32;0.30)</td>
<td>0.998</td>
</tr>
<tr>
<td>4</td>
<td>0.1145</td>
<td>0.141</td>
<td>0.32</td>
<td>(0.34;0.33;0.30)</td>
<td>0.997</td>
</tr>
</tbody>
</table>
Verification of the approximation

- Determination of property contribution tensors of irregular pores (numerically)
- Determination of property contribution tensors of ellipsoidal approximation (analytically and numerically)
- FEM is used
- Mesh is done using Comsol Multiphysics
- Code_Aster (EDF) is used to perform the simulations

\[ \Delta \varepsilon = \frac{V}{V^*} \ H : \sigma^\infty \]

\[ \Delta \varepsilon_{ij} = \frac{-1}{2V} \int_{\delta \Omega} \left( u_i n_j + u_j n_i \right) dS \]

\( \Delta \varepsilon \): extra strain generated by the inhomogeneity

= volume integral of strain tensor (may be replaced by surface integral)

\( u, n \): displacements at the boundary of the pore and normal vector

Theoretical introduction

Approximation of a pore by an ellipsoid
Approximation of a pore by an ellipsoid

3D acquisition of real pores

Ellipsoidal approximation
E=1 MPa \,
\nu=0.3

Numerical solution: FEM

Pore is placed in a cube → Mesh is done for all the model (Comsol) → Application of boundary conditions → Displacements on every node → Normal vectors on Gauss points → Extra Strains

Volume discretization:
10-nodes tetrahedrons

Surface discretization of pore and ellipsoid:
6-nodes triangles

Quadratic interpolation in both cases
**Boundary conditions:** 6 load cases are needed

- 3 for compression or tension \((\sigma_{11}, \sigma_{22}, \sigma_{33})\)
- 3 for shear \((\sigma_{12}, \sigma_{13}, \sigma_{23})\)

\[
\Delta \varepsilon_{ij} = \frac{-1}{2V} \int_{\delta\Omega} (u \cdot n_j + u_j n_i) dS
\]

**Analytical solution for the normal vector**
- Gradient of implicit function of surface: \(\left(\frac{x-x_i}{a}\right)^2 + \left(\frac{y-y_i}{b}\right)^2 + \left(\frac{z-z_i}{c}\right)^2 = 1\)
- Product of 2 vectors of the surface

**Analytical solution:** Eshelby’s theory

\[ S^E = \mathbb{P} : C_0 \]
\[ \mathbb{P} = S^E : S_0 \]

**Eshelby tensor**

**Hill tensor**

**Numerical solution**
- Calculation of the normal: Product of 2 vectors of the surface

**Ellipsoidal Pores**

**Irregular Pores**
Results for a selected irregular pore and its corresponding ellipsoid

**3D view of a selected pore and its approximated ellipsoid using the PCA method**

**Compliance contribution tensor for approximated ellipsoid:**
Analytical solution (implicit function)

\[
\begin{pmatrix}
1.3754 & -0.3774 & -0.4341 & 0 & 0 & 0 \\
-0.3774 & 1.9807 & -0.6171 & 0 & 0 & 0 \\
-0.4341 & -0.6171 & 3.0447 & 0 & 0 & 0 \\
0 & 0 & 0 & 3.1473 & 0 & 0 \\
0 & 0 & 0 & 0 & 2.7669 & 0 \\
0 & 0 & 0 & 0 & 0 & 2.082 \\
\end{pmatrix}
\]

**Compliance contribution tensor for approximated ellipsoid:**
Analytical solution (Eshelby's solution)

\[
\begin{pmatrix}
1.3761 & -0.3794 & -0.4354 & 0 & 0 & 0 \\
-0.3794 & 1.9815 & -0.6185 & 0 & 0 & 0 \\
-0.4354 & -0.6185 & 3.0458 & 0 & 0 & 0 \\
0 & 0 & 0 & 3.1488 & 0 & 0 \\
0 & 0 & 0 & 0 & 2.7681 & 0 \\
0 & 0 & 0 & 0 & 0 & 2.092 \\
\end{pmatrix}
\]

**Compliance contribution tensors for an irregular pore and its approximated ellipsoid:**
Numerical solution

\[
\begin{pmatrix}
1.3863 & -0.3731 & -0.4218 & -0.0044 & -0.0109 & 0.0195 \\
-0.3731 & 1.9821 & -0.6212 & 0.0181 & 0.0009 & 0.0211 \\
-0.4218 & -0.6212 & 3.0828 & 0.0174 & -0.0027 & -0.0048 \\
-0.0044 & 0.0181 & 0.0174 & 3.192 & 0.0273 & -0.0064 \\
-0.0109 & 0.0009 & -0.0027 & 0.0273 & 2.8522 & 0.0136 \\
0.0195 & 0.0211 & -0.0048 & -0.0064 & 0.0136 & 2.1136 \\
\end{pmatrix}
\]

- Matrices should be symmetric for mechanical considerations:
- Numerical values of the non-diagonal components of each matrix data are less than 1% of each other
- The terms of these two tensors are very close, with a maximum relative error of 3%
Frobenius distance in % between analytical and numerical compliance contribution tensors for irregular and ellipsoidal pores compared to the analytical solution based on Eshelby’s theory

- "Frobenius Norm"

\[ \| A \|_F = \sqrt{\sum_{i=1}^{m} \sum_{j=1}^{n} |a_{ij}|^2} \]

- To evaluate the accuracy of these approximations, we define \( \Delta \) the distance between two matrices:

\[ \Delta = \frac{|A_{F}^{exact} - A_{F}^{approximation}|}{|A_{F}^{exact}|} \]

<table>
<thead>
<tr>
<th>H tensor</th>
<th>Ellip (Eshelby)</th>
<th>Ellip (implicit function)</th>
<th>Ellip (cross product)</th>
<th>Irregular pore</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ellip (Eshelby)</td>
<td>0</td>
<td>0.18</td>
<td>0.61</td>
<td>2.04</td>
</tr>
<tr>
<td>Ellip (implicit function)</td>
<td></td>
<td>0</td>
<td>0.82</td>
<td>1.99</td>
</tr>
<tr>
<td>Ellip (cross product)</td>
<td>0</td>
<td></td>
<td>2.24</td>
<td></td>
</tr>
<tr>
<td>Irregular pore</td>
<td></td>
<td></td>
<td>0</td>
<td></td>
</tr>
</tbody>
</table>
Frobenius distance in % between analytical and numerical compliance contribution tensors for irregular and ellipsoidal pores compared to the analytical solution based on Eshelby’s theory

- The values of the distance produce discrepancy of max. 4.5%

- The distance between compliance tensors obtained numerically and from the 2 analytical solutions does not exceed 3%

→ Proposed computational method is suitable for all the shapes of irregular pores from a mechanical point of view

→ The approximation of an irregularly shaped pore by an ellipsoid is relevant within the simplified model framework (Maxwell homogenization scheme)
Reformulation of Maxwell homogenization scheme in terms of compliance contribution tensors

- Material modeled as 3 Phase composite medium
- Compliance contribution tensors $H$ used to evaluate effective properties
- Comparison of Irregular Pores vs Ellipsoid approximation

$$S^{\text{eff}} = S_c + \left\{ [f_o H_o + f_b H_b]^{-1} - Q_\Omega \right\}^{-1}$$

- Stiffness tensor of matrix $f(k_o, \mu_o)$
- By FEM $f(\text{Hill's tensor})$

$$\bar{H}_b = \bar{H}_b^k J + \bar{H}_b^\mu K$$
$$Q = C_c : (I - P : C_c)$$
$$S^{\text{eff}} = 3k^{\text{eff}} J + 2\mu^{\text{eff}} K$$

- Calcite: Isotropic background
- Elastic properties of calcite are determined from nano-indentation tests
Pore orientation study

<table>
<thead>
<tr>
<th>Pore</th>
<th>Radius (μm)</th>
<th>Euler angles</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>a</td>
<td>b</td>
<td>c</td>
</tr>
<tr>
<td>1</td>
<td>130,7</td>
<td>80,3</td>
<td>37,9</td>
</tr>
<tr>
<td>2</td>
<td>149,866</td>
<td>108,492</td>
<td>82,5738</td>
</tr>
<tr>
<td>3</td>
<td>136,608</td>
<td>101,932</td>
<td>70,67</td>
</tr>
<tr>
<td>4</td>
<td>185,255</td>
<td>90,79</td>
<td>41,6</td>
</tr>
<tr>
<td>5</td>
<td>137,427</td>
<td>104,304</td>
<td>69,5891</td>
</tr>
<tr>
<td>6</td>
<td>104,117</td>
<td>84,1252</td>
<td>73,819</td>
</tr>
<tr>
<td>7</td>
<td>140,777</td>
<td>75,5153</td>
<td>67,8832</td>
</tr>
<tr>
<td>8</td>
<td>140,52</td>
<td>66,78</td>
<td>57,958</td>
</tr>
</tbody>
</table>


Pores randomly oriented
Dependencies of effective elastic properties of 10 randomly oriented irregularly shaped pores on volume fraction

Effective elastic properties for a selected pore: verification of the ellipsoidal approximation
Conclusions/Perspectives

- Extension of this method to other oolitic rocks (iron ore)

- Calculation of H tensors for both pores and ellipsoids for a N number of pores for 2 materials: Lavoux Limestone and iron ore

- Study of the influence of different parameters on the approximation (Volume, Surface, Specific surface, inertia moment, etc)

- Application and comparison of others homogenization methods

- Coupling of acoustic + thermic (optical scanning) measurements at centimetric scale

- Micro- and nano-indentation measurements → mechanical properties of constituents at micro-scale.