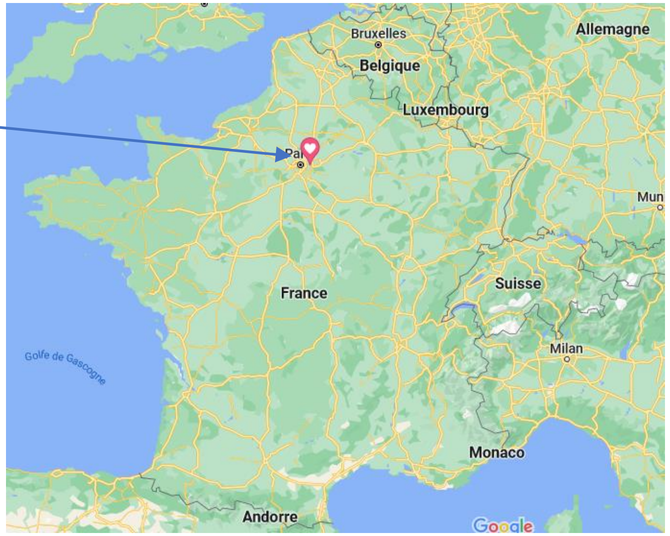
Interpore 2026 (Nantes) MS05
22 may 2026

InterPore2026

Fluid transfers through versatile *dynamic NMR relaxometry*

***Benjamin Maillet** (oral presenter), Philippe Coussot, Rahima Sidi-Boulenouar (Navier laboratory)*



Rheology and Porous Media team building

Ecole des Ponts Paris Tech

Champs-sur-Marne France

Main topic of Navier laboratory : **Material for construction by plenty of approaches.**

Why understand the interaction between water and solid matrix ?

Plenty of approaches but approach often …

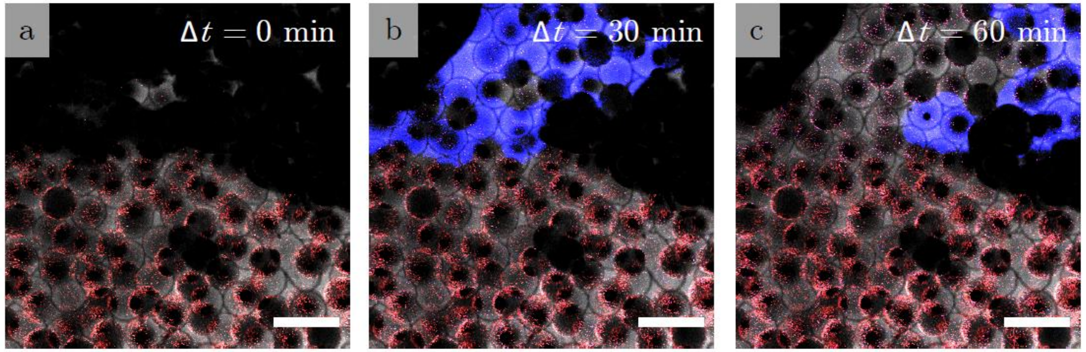
→ **not temporally and/or spacially** resolved (only 1 image or a set of global information)

→ **not « multiscale »**

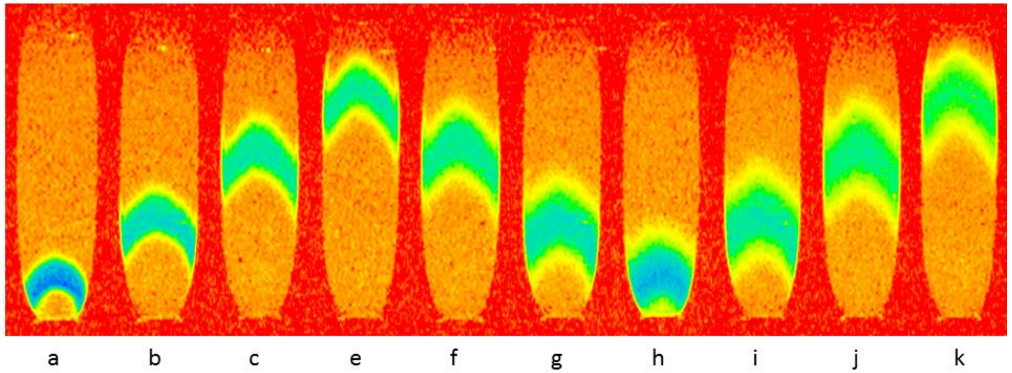
→ **not quantitative**

→ needs to **hydric equlibrium** (in contrary to the « real life »).

→ **not so versatile** (only specific material)



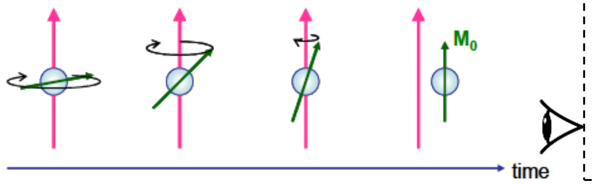
Using colloidal deposition to mobilize immiscible fluids from porous media
Joanna Schneider, Rodney D. Priestley, and Sujit S. Datta
Phys. Rev. Fluids, 2021



Lehoux et al, Physical Review E, 2016

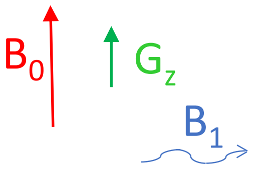
→ The **« Dynmaic NMR relaxometry »** is an innovative and general approach to answer to all these limitations **for porous media and complex fluids**

The most standard NMR setup at Navier laboratory…

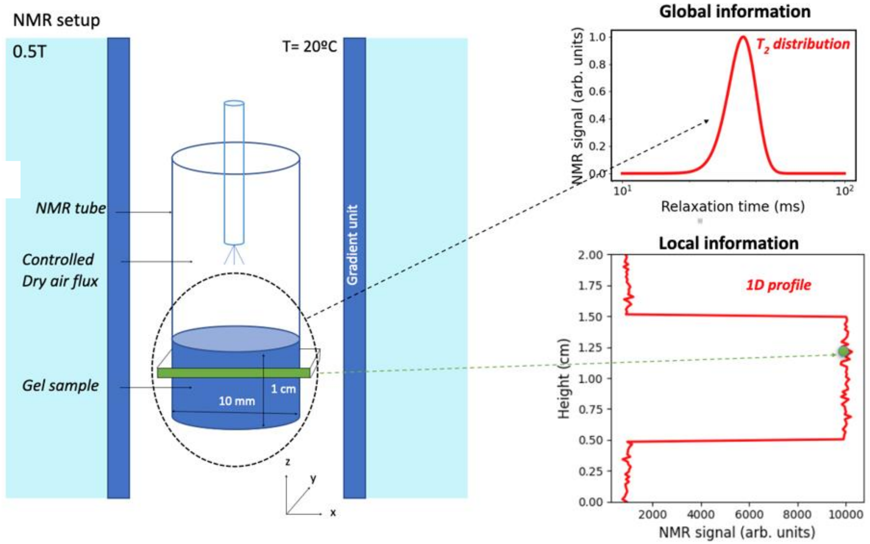


$\mathbf{T2}$: characteristic time of magnetization (transverse part) to come back at equilibrium

Minispec Brüker 0,5 teslas



CPMG sequence
Global information



Spin Echo sequence
Local information
(optional)

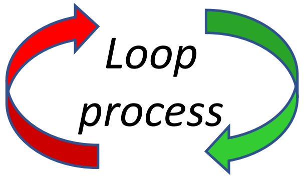
→ Transverse relaxation time
T2 probability density function
∫ A.exp(-t/T2) d(ln(T2))

V(sample) ≈ 1 cm^3 ***>> pore, particle,… size***
Duration(exp) ≈ 10 min ***<< drying duration***

↗ dry air flux + ↘ distance *[air tube – sample]*
→ « more agressive » **drying by the top surface**

→ NMR of liquid proton (**water** at 20 °C by default)

→ Direct access to NMR observable evolution



The fast exchange theory as a key concept…

→ **NMR relaxation = Surface interaction process**

→ Shift of the relaxation efficiency according to Tarr and Browstein (1979):

$$\frac{1}{\tau_2} = \frac{1}{T_{2,measured}} - \frac{1}{T_{2,\ pure\ liquid}}$$

→ **Aqueous solution :** $\tau_2 = \frac{1}{r_2} \cdot \frac{V_{water}}{n_{ion}}$

Paste, suspension, gel,… (φ: volume fraction): $\tau_2 = \frac{1}{r_2} \cdot \frac{1-\varphi}{\varphi} = \frac{1}{r_2} \cdot \frac{V_{water}}{V_{particle}}$

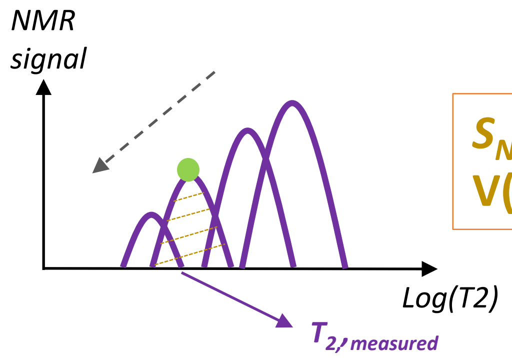
Porous medium: $\tau_2 = \frac{1}{r_2} \cdot \frac{V_{pore}}{S_{pore}}$ (saturated) or $\tau_2 = \frac{1}{r_2} \cdot \frac{V_{water}}{S_{wet}}$ (unsaturated)

→ $T_{2,pure\ water}$ = 2250 ms at 20°C (reference) > $T_{2,pure\ surf}$ and **bulk water dries first**

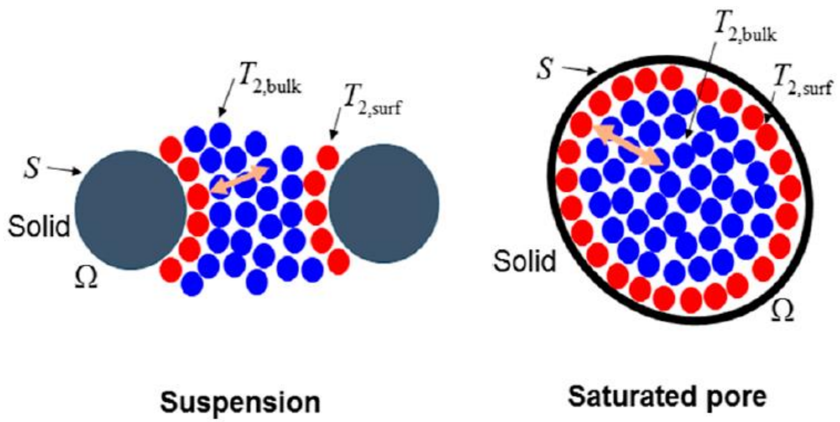
b ≈ water molecule size

r_2 : transverse relaxivity higher if the solid matrix is a stronger relaxation process for liquid water → **depends on the chemical composition**

ANR Thermo NMR, Sidi-Boulenouar (> 2026)



S_{NMR} α V(water)



Julien, Maillet, Tocquer, Aime, Coussot Langmuir, 2024

Fast exchange bulk – surface available if :

→ **1 molecule** can **explore surface and bulk** several times during T2 duration

→ **Surface residence time << Diffusion time** inside the pore

Dynamic relaxometry easier to interpret if :

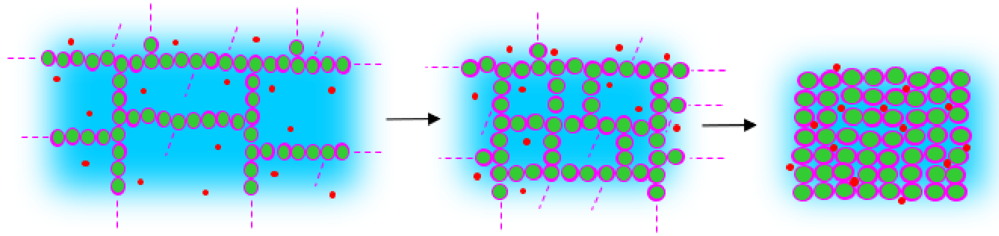
→ **$T_{2,ads.}$ or r_2 constant during the** drying if **stable roughness, chemical composition**,…

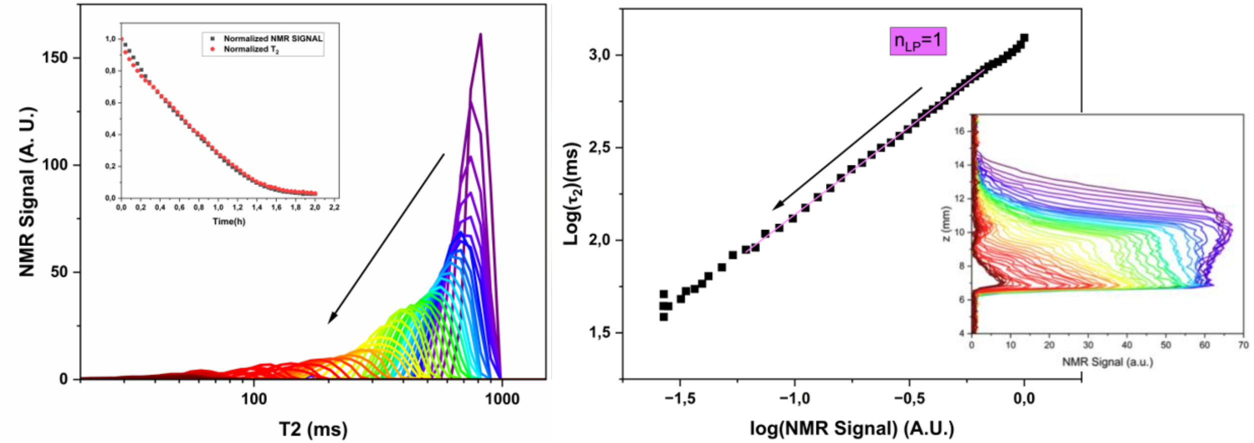
→ **No gradient** of water content, **isotropic** swelling or shrinkage,…

→ **No dewetting** of the solid matrix

The reference power law… PL(t) = 1 ! Slow drying of a stable material

*Slow drying of **Ludox suspension** 0,5 M*





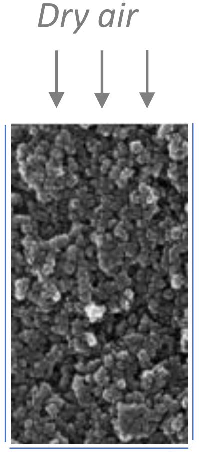
Sidi-Boulenouar, Maillet, Carouge, Ayturk, Poulesquen, Langmuir, 2026

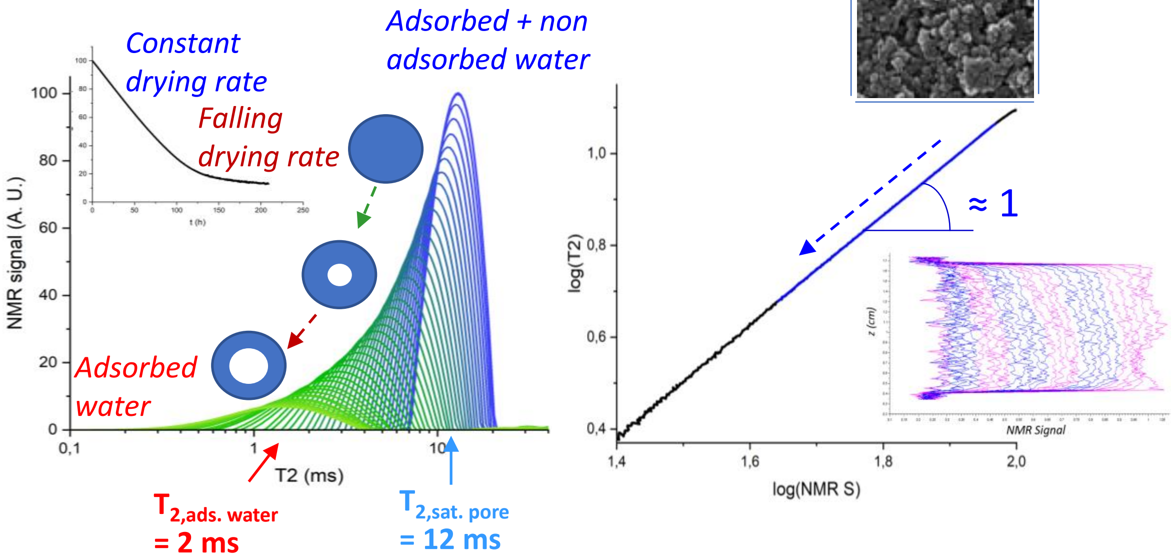
$$\tau_2 = \frac{1}{r_2} \cdot \frac{1}{V_{particle}} \cdot V_{water}$$

$\log(\tau_2) = C + 1 \cdot \log(S_{NMR})$ → **PL(t) ≈ 1**

if $V_{particle}$ **constant (robust particle), …**

Slow drying of saturated Vycor
*= pure fused **silica glass** by **3D network of interconnected** tortuous **isotropic monodisperse** pores (size ≈ 4,6 nm).*





Maillet, Sidi-Boulenouar, Coussot. Langmuir, 2022

$$\tau_2 = \frac{1}{r_2} \cdot \frac{1}{S_{wet}} \cdot V_{water}$$ (for unsaturated porous medium)

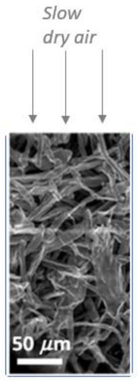
$\log(\tau_2) = C + 1 \cdot \log(S_{NMR})$ → **PL(t) ≈ 1**

if S_{wet} **constant (no dewetting), …**

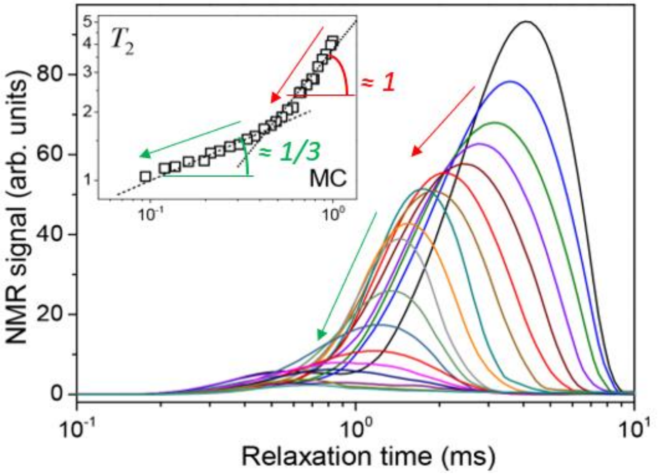
… , r_2 constant (chemical stability), fast exchange available, homogenous hydric content during the most of the drying.

Deviation of the power law PL(t) n° 1. Shrinkage with no desaturatrion (PL < or = 1).

Slow drying of
Humid cellulose



Fibre dimension :
700 x 20 µm



Maillet
Sidi-Boulenouar
Coussot. Langmuir
2022

Regime 1: LP(t) = 1/1

$$\tau_2 = \frac{1}{r_2} \cdot \frac{1}{S_{wet}} \cdot V_{water}$$

$\log(\tau_2) = C + 1 \cdot \log(S_{NMR}) \rightarrow$ **PL(t) ≈ 1**

τ_2 and V_{water} decreases by the same relative variation.

→ **Constant wetted surface**

Regime 2: LP(t) = 1/3

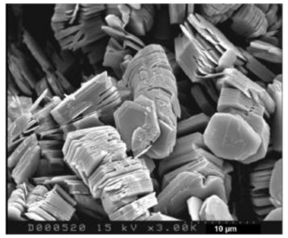
$$\tau_2 = \frac{1}{r_2} \cdot \frac{1}{S_{wet}} \cdot V_{water}$$

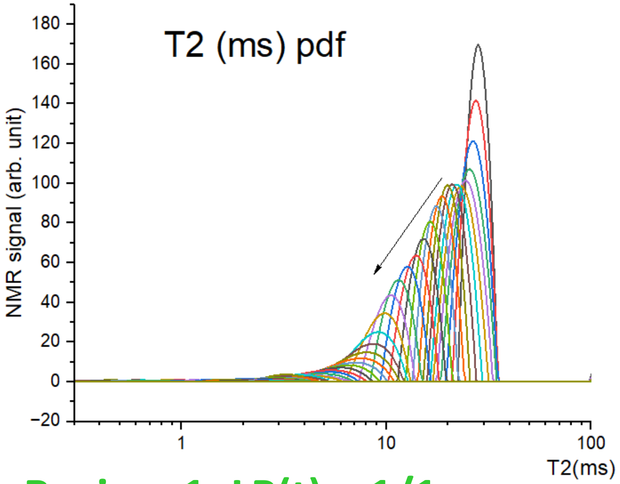
$\log(\tau_2) = C + 1/3 \cdot \log(S_{NMR}) \rightarrow$ **PL(t) ≈ 1/3**

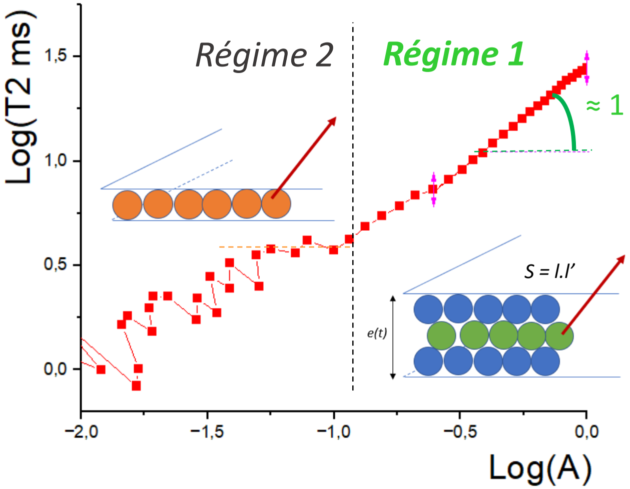
→ **Isotropic shrinkage**

if $\tau_2 \propto V_{water} / S_{wet} \propto$ [pore size]^1 and $V_{water} \propto$ [pore size]^3 , …

First part of slow drying of
kaolinite paste (φ_i = 0,27)







Maillet
Sidi-Boulenouar
Keita
2026

Regime 1: LP(t) = 1/1

$$\tau_2 = \frac{1}{r_2} \cdot \frac{1}{S_{wet}} \cdot V_{water}$$

$\log(\tau_2) = C + 1 \cdot \log(S_{NMR}) \rightarrow$ **PL(t) ≈ 1/1**

if S_{pore} = 2. l . l' is constant and V_{pore} = l . l' . e(t) decreases.

τ_2 and V_{water} decreases by the same relative variation.

→ only thickness of water e(t) decreases

« layer by layer » of water, if …

(Regime 2: LP(t) ≈ 0 → next slide…)

… , r_2 constant (chemical stability), fast exchange available, homogenous hydric content during the most of the drying.

Generalization: **PL = 1/N. N: dimentionality of the shrinkage.**

Deviation of the power law PL(t) n° 2. Desaturation with perfect dewetting (PL = 0).

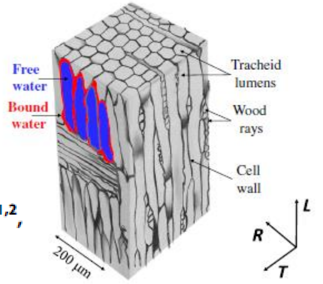
Slow drying of ***green poplar (free water)***

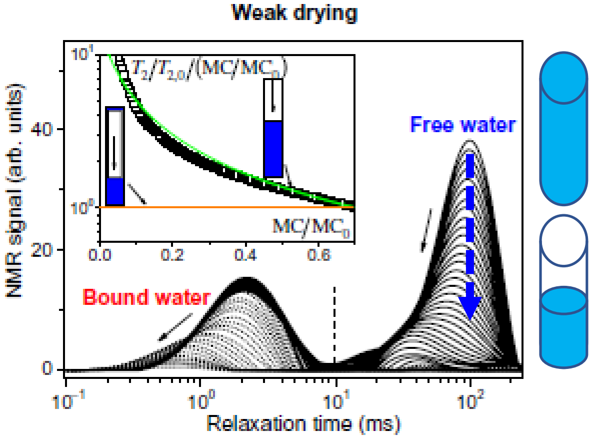
SCIENCE ADVANCES | RESEARCH ARTICLE

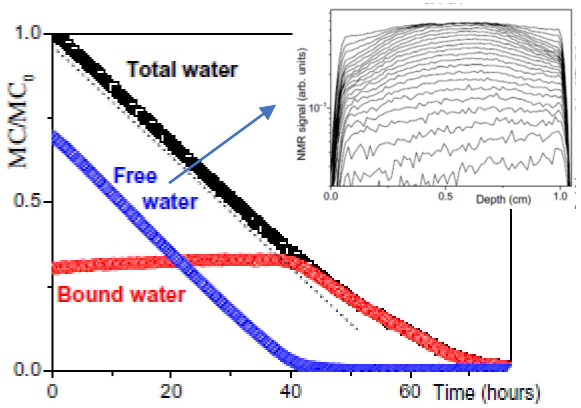
APPLIED PHYSICS

Marion Cocusse[1], Matteo Rosales[1], Benjamin Maillet[1], Rahima Sidi-Boulenouar[1], Elisa Julien[1,2], Sabine Caré[1], Philippe Coussot[1]*

Cocusse *et al.*, *Sci. Adv.* **8**, eabm7830 (2022) 13 May 2022







$$\tau_2 = \frac{1}{r_2} \cdot \frac{1}{S_{wet}} \cdot V_{water} \approx \text{cste}$$

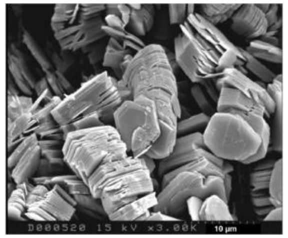
during the most of free water drying

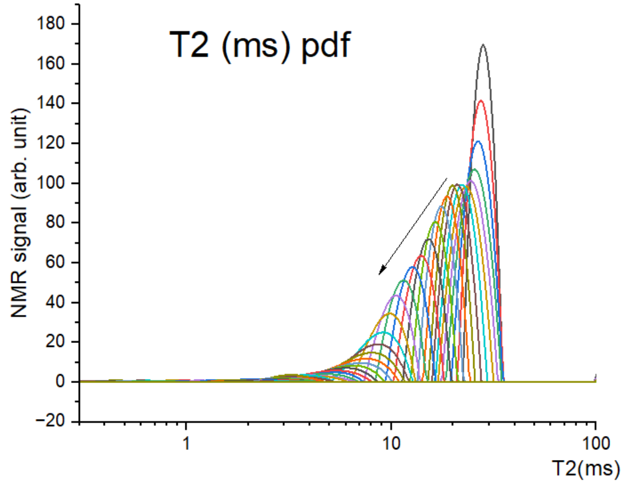
$\log(\tau_2) = C + 0 \cdot \log(S_{NMR}) \rightarrow$ **PL(t) ≈ 0**

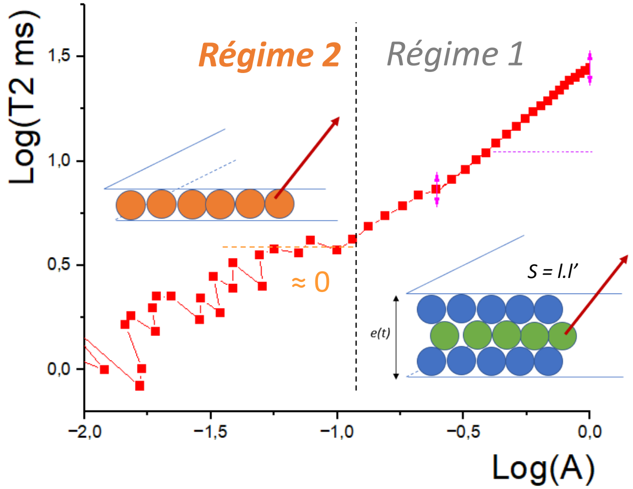
if S_{wet} and V_{water} decrease by the same relative variation

→ **Full dewetting** with the same meniscii in tracheid, …

End of slow drying of ***kaolinite paste (φ_i = 0,27)***







Regime 1: LP(t) = 1

Regime 2: LP(t) ≈ 0

$$\tau_2 = \frac{1}{r_2} \cdot \frac{1}{S_{wet}} \cdot V_{water} \approx \text{cste}$$

$\log(\tau_2) = C + 0 \cdot \log(S_{NMR}) \rightarrow$ **PL(t) ≈ 0**

if S_{wet} and V_{water} decrease by the same relative variation

→ In accordance with **dewetting during the last layer drying** between 2 clay waferers « molecule by molecule », …

(→ See **P. Coussot** presentation for more detail…)

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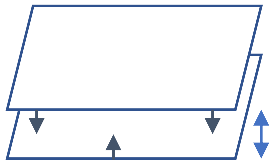
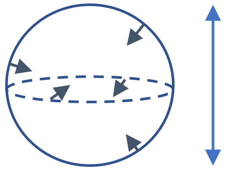
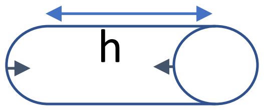
Sidi-Boulenouar
Keita
2026

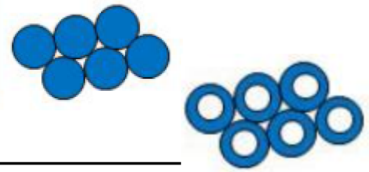
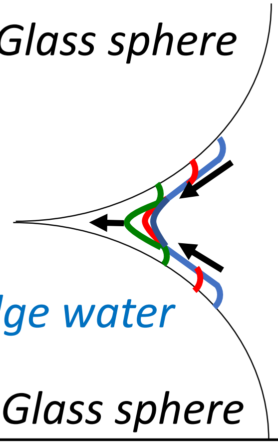
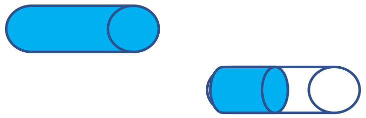
… , r_2 constant (chemical stability), fast exchange available, homogenous hydric content during the most of the drying.

Generalization of the power law if stable chemistry and homogen water content:

Shrinkage or swelling of a saturated porous medium :

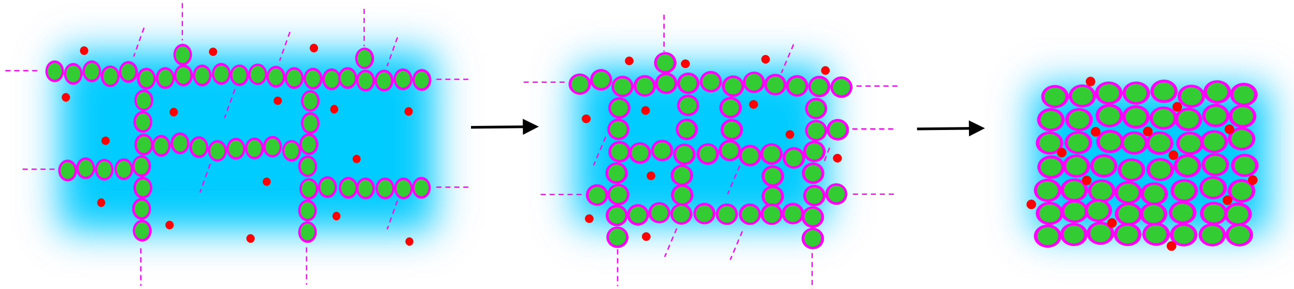
Power law	**Dimentionality of the deformation**	
1 / 1	**N = 1** Ex : Lamellar shape (only thickness changes)	
1 / 2	**N = 2** Ex :Thin cylindric shape (only diameter changes)	
1 / 3	**N = 3 Ex** : Spherical pore (only diamter changes)	
1 / ∞	Thin cylindric shape (only height changes)	
1 / N	**N : Dimenssionality of the deformation**	

Saturation or desaturation of non deformable porous medium ;

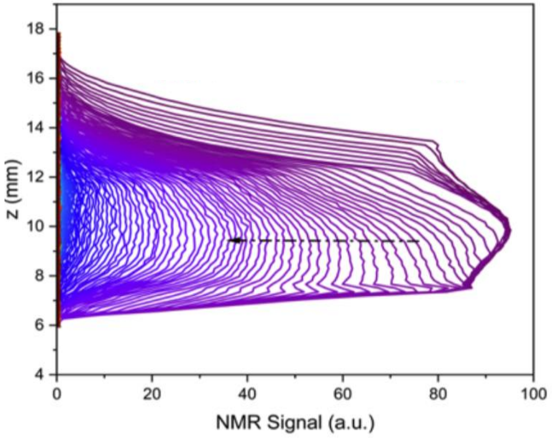
Power law	**Wetability**	
1	**Pore wall remains totaly wet** d(S(wet)/S(wet)) = 0	
]0 ; 1[	0 < d(S(wet)/S(wet)) < d(V(water)/V(water))	
0	**Stable meniscus :** d(V(water)/V(water)) = d(S(wet)/S(wet))	

Deviation of the power law PL(t) n° 3. Hydric inhomogneity development.

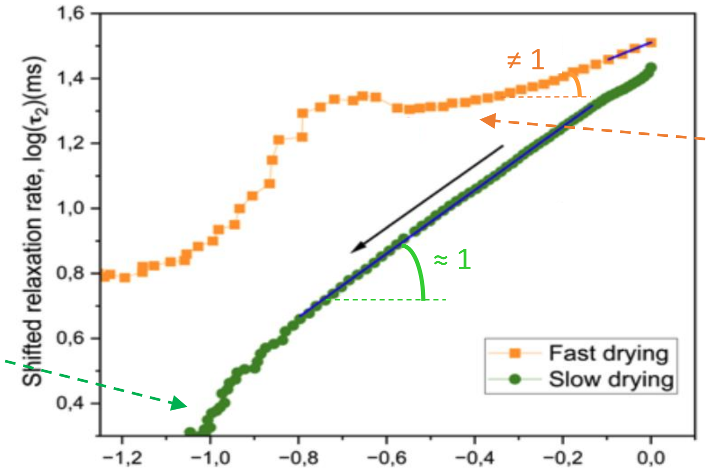
Drying of
aluminosilicate gel



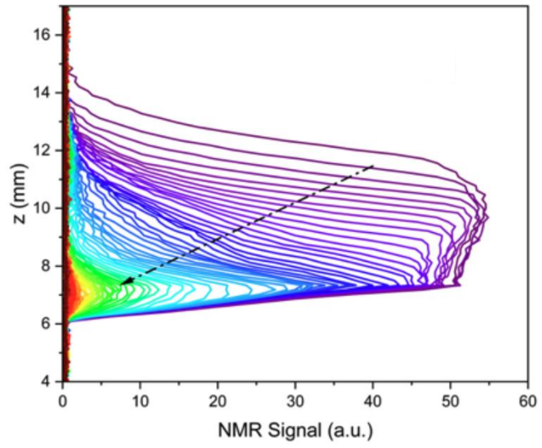
Sidi-Boulenouar, Maillet, Carouge, Ayturk, Poulesquen. Langmuir, 2026 Submitted



Hydric vertical profiles of the AlSi gel during ***slow drying***



Hydric vertical profiles of the AlSi gel during ***fast drying***



$$\tau_2 = \frac{1}{r_2} \cdot \frac{1}{V_{particle}} \cdot V_{water}$$

$\log(\tau_2) = C + 1 \cdot \log(S_{NMR})$ → **PL(t) ≈ 1** (reference value)
if $V_{particle}$ **constant (no shape or roughness modification),**
r_2 constant (chemical stability), fast exchange available,
homogenous hydric content
during the most of the drying.

$$\tau_2 = \frac{1}{r_2} \cdot \frac{1}{V_{particle}} \cdot V_{water}$$

$\log(\tau_2) = C + PL(t) \cdot \log(S_{NMR})$ → **PL(t) not constant**
… because of the **too slow diffusion of particles** that don't ensure fast redistribution of particles **during this fast drying.**
Gradient of $T_2(z,t)$ and ϕ (z,t) ↔ Deviation of PL(t)
We need to take into account **hydric gradient, contraction, reconfiguration of particle ability to predict the power law deviation**

→ **See Rahima Sidi-Boulenouar's poster for more details based on simulatuion of profiles to predict PL(t) fully !**

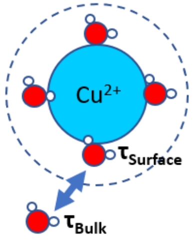
Deviation of the power law PL(t) n° 4. Chemical instability

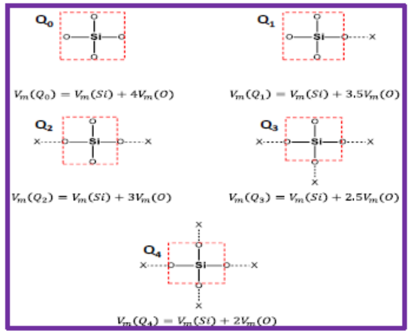
Slow drying (concentrations increase) of

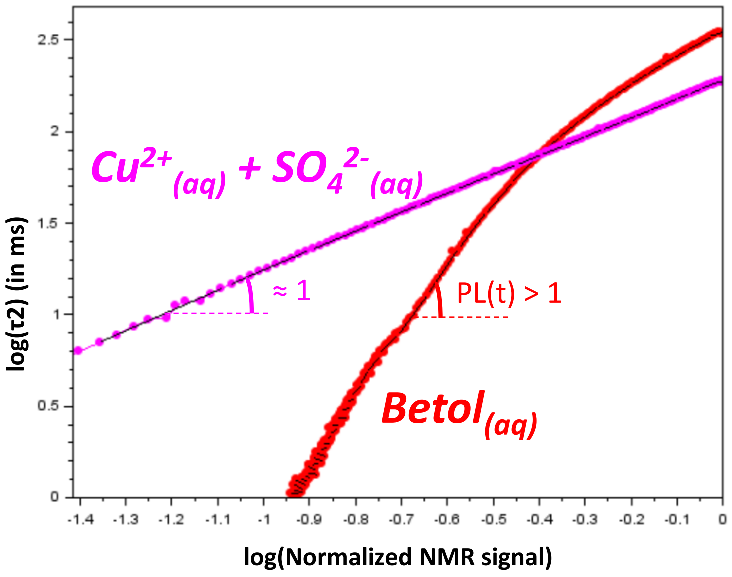
$Cu^{2+}_{(aq)} + SO_4^{2-}_{(aq)}$ solution (very stable)

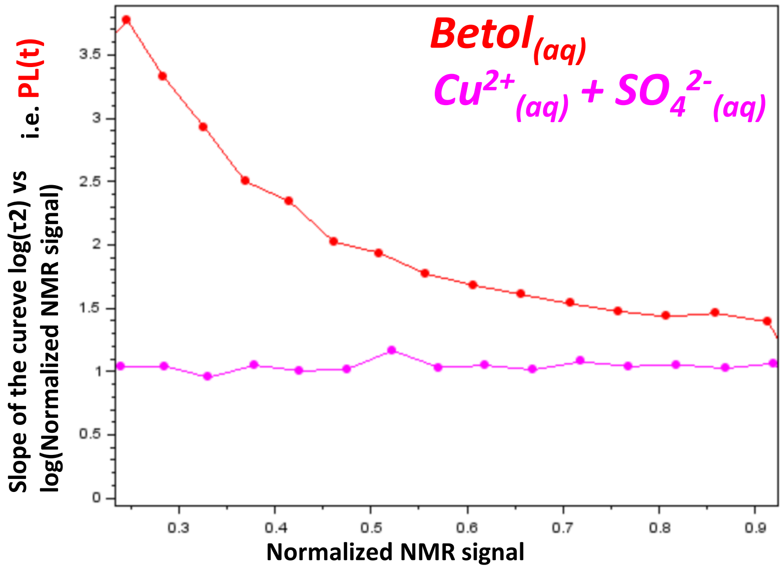
… vs … ***Betol solution (gel AlSi precursor : $Si_n O_m H_{p(aq)}$, $Na^+_{(aq)}$, $HO^-_{(aq)}$)***

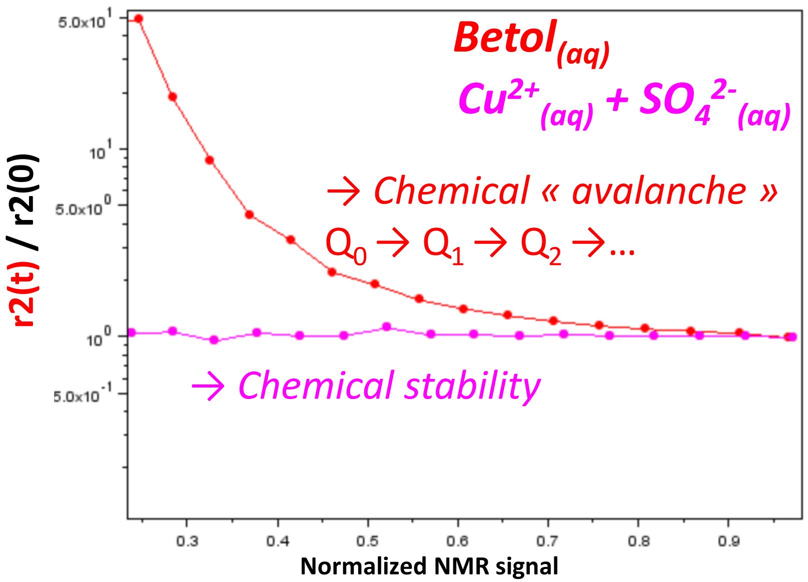
M'Homa
Chabane
Maillet
Sidi-Boulenouar
Poulesquen
2026 (in progress)











$$\tau_2 = \frac{1}{r_2} \cdot \frac{1}{n_{ion}} \cdot V_{water} \rightarrow r_2(t) = r_2(0) \cdot \left(V_{water}(t)/V_{water}(0) \right)^{1-PL(t)}$$

if **chemical instability only** and homogeneous concentration, fast exchange available.

Comprehensive structural and dynamical study of alkali silicate solutions: Influence of hydroxide concentration and alkali nature

Arnaud Poulesquen [a,*], Donatien Gomes Rodrigues [a], Rahima Sidi-boulenouar [b], Benjamin Maillet [b], Christophe Goze-bac [c], Dominique Petit [c]

Colloids and Surfaces A: Physicochemical and Engineering Aspects 736 (2026) 139622

PL(t) = 1 for *$Cu^{2+}_{(aq)} + SO_4^{2-}_{(aq)}$ solution* → **$r_2(t) = r_2(0)$** → **Cu^{2+} very stable** during all the drying.

PL(t) > 1 and increases more and more for Beteol solution → effective **$r_2(t)$ increases more and more**

→ **Betol very unstable** during all the drying in accordance **more than 2 states Q_i : $Q_0 \rightarrow Q_1 \rightarrow Q_2 \rightarrow$** … if $r_2(i)$ increases.

Genralization ?

→ **Innovative methodology to sort components (ion, particle, solide matrix,…) by degree of chemical unstabilty**.

Conclusion and perspective.

Shift of the relaxation efficinecy : $\frac{1}{\tau_2} = \frac{1}{T_{2,\text{measured}}} - \frac{1}{T_{2,\text{ pure liquid}}} = r_2 \cdot \frac{\text{cste}}{V_{\text{water}}}$ = « **Chemical term** x **Physical term** »

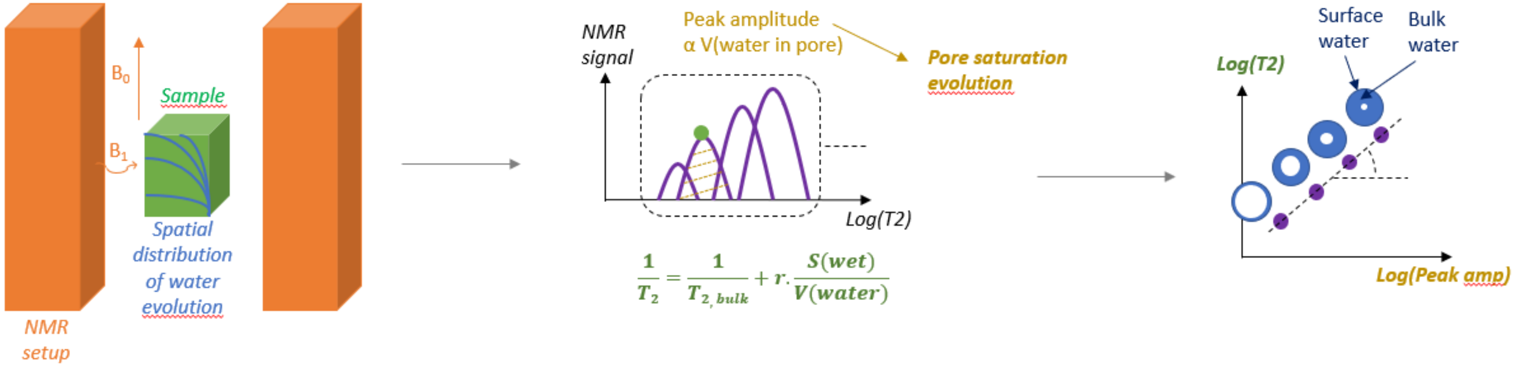
… always based on the **fast exchange** theory (surface and bulk water by diffusion and other…)

log(τ_2) vs log(NMR S) → power law evolution **PL(t)** during drying = **innovative, very generalisable representation to catch all physical and chemical instabilites by a multiscale, temprally and non invasive approach**:
dewetting of the solid matrix, **inhomogneity of local water content, chemical instability,** … and more ?

The ***Dynmaic NMR relaxometry*** can scope **all water state progressively** and is **available for hydric drying of plenty of material as porous medium, paste, suspension, gel, solution, granular media**, … and also foam, emulsion, … ?

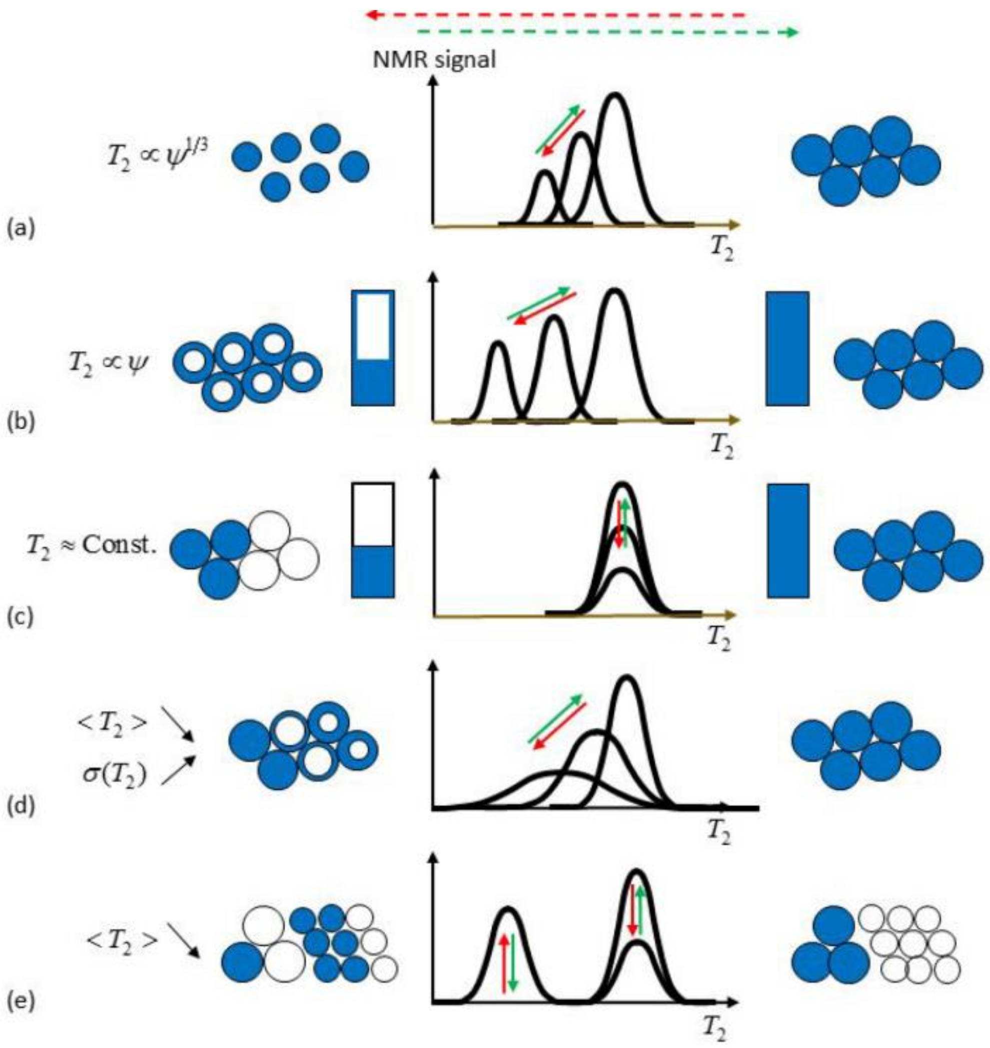
Generalizations should be possible for **all water transfer** (ex: imbibition) or **other volatil protonic liquids**,…
… or a chemicaly and physicaly unstable material (ex: setting of a fresh cement paste) ?

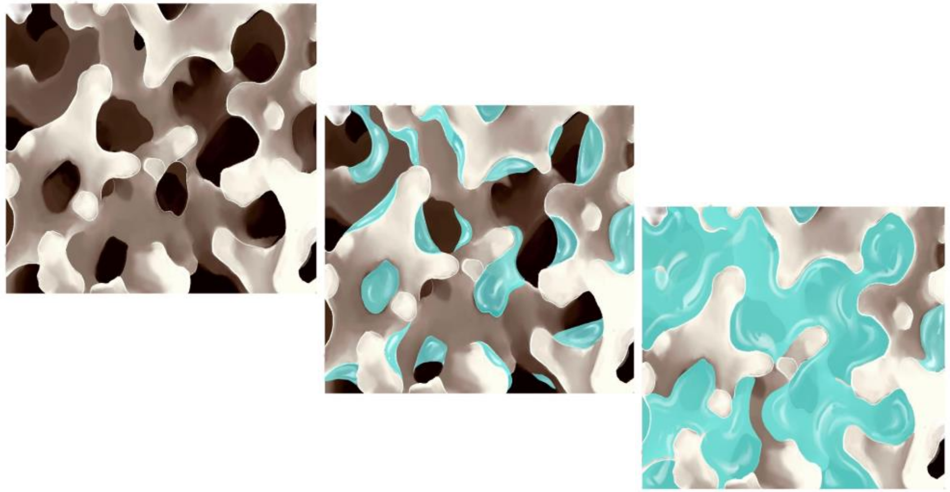
Do you want to see more NMR from Navier laboratory ?
→ 3 other presentations
(**P. Coussot, F. Gerony, E. Keita**)
→ 4 posters
(**L. Brochard, O. Hbaieb, R. Sidi-Boulenouar, A. Samba**)



Sum up of the main expected aspects of the T2 pdf

Figure 3. Evolution of the aspect of the pdf of T_2 during different ideal cases of water extraction from pores (red arrow) or the refilling of pores (green arrow). Blue areas correspond to liquid water, and white regions correspond to air: (a) homogeneous pore shrinkage or swelling; (b) homogeneous pore desaturation or saturation with perfect wetting of the solid surface; (c) heterogeneous pore emptying or filling; (d) heterogeneous pore desaturation or saturation with perfect wetting; (e) transfers from one pore size to another. In each case, some associated trend of variation of T_2 as a function of the saturation is indicated (<.> is the mean value; σ is the variance).





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Invited Feature Article

Dynamic NMR Relaxometry as a Simple Tool for Measuring Liquid Transfers and Characterizing Surface and Structure Evolution in Porous Media

Benjamin Maillet, Rahima Sidi-Boulenouar, and Philippe Coussot*

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