Supporting Information

to: NMR 1D-Imaging of Water Infiltration into Porous Bitumen-Salt Matrices: the Effect of Salt Solubility

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S1  NMR acquisition parameters, and calibration/analysis of the measurements.

The basic scheme of an NMR experiment with a profile readout is shown in Fig. S1.

Figure S1: Acquisition scheme of a NMR experiment with profile readout, for both the RF and the gradient ($g_z$) channel.

S1.1  Calibration of the NMR profile readout.

Acquisition of a profile on a reference tube filled with pure H$_2$O (respectively pure D$_2$O) was performed in order to determine the homogeneous region of the NMR $H_1$ or RF excitation field: we found the condition $|z - z_0| < 0.5$ cm, where $z_0$ is the center of the RF coil along $z$: this defines the volume within which the profiles are undistorted. In all our experiments on BWPs, the samples could be positioned to ensure that the leached region is included within these boundaries. Measurement of the reference sample also provides a “reference signal” (corresponding to 100% water volume fraction in the tube), which we used to set the absolute vertical scale on the profiles, for instance as in main text, Fig. 3. By integration over $z$ of the reference signal, we can also obtain a “reference integral” for a known mass of water, which enables to estimate from the NMR signal the mass of each measured water population.
S1.2 \( z \)-resolved \( T_2 \) experiment.

The \( z \)-resolved \( T_2 \) experiment is simply obtained by repeating the above-mentioned profile readout for various values of the interpulse duration \( \tau \). In general, 9 values of \( \tau \) covering the 300 \( \mu s \) - 70 ms range were used. Assuming the simplest case of a monoexponential relaxation within a slice (defined as in main text, Fig. 2), the average intensity would evolve as \( \exp(-2\tau/T_2) \). In our analysis, fitting functions with 2-3 exponential relaxation components were rather used to describe BWPs, with 1-2 components describing the relaxation from liquid water, while the remaining (very short) component accounts for the relaxation from the solid part. This solid part can in principle correspond to bitumen or possibly to bound water in salt hydrates. We found out, however, that the reconstructed profile corresponding to the very short \( T_2 \) population (data not shown) was (1) homogeneous across the full sample, and, (2) unchanged between the dry and leached material - therefore, we consider that this signal represents exclusively the unaltered bitumen.\(^a\)

S1.3 \( z \)-resolved \( T_1 \) experiment

![Figure S2: Acquisition scheme for the \( z \)-resolved \( T_1 \) experiment. Same definitions as on Fig. S1.](image)

The \( z \)-resolved \( T_1 \) experiment is performed by using an inversion-recovery preparation sequence (i.e. a \( \pi \) inversion pulse followed by a recovery time \( \Delta t \)) before the profile readout,

\(^a\)In a separate study on some other model BWPs, we found that the relaxation from the dry material had also a “long” component (\( T_2 \sim 100 \) ms) in addition to the very short one. This might originate from remainders of solvents that were injected within the material during the extrusion process. Such a long component was not detected in the compounds that are described in the present work.
see Fig.S2. The typical acquisition was performed using 9 values of $\Delta t$, and $\tau = 300 \mu$s for the profile readout.

A subtraction of the signal from the bitumen ($T_1 = 350 \pm 50$ ms) was performed prior to the analysis. Both the signal from water and the signal from bitumen could be obtained from the same $z$-resolved $T_1$ experiment, as the measured region always encompassed a dry zone (see for instance in main text, Fig. 2).

In the case of monoexponential relaxation, the intensity within a slice would evolve as $I(t) = M_i + M_0(1 - \exp(-t/T_1))$. In our analysis, we used: For the I-type sample (see main text, Fig. 4): either monoexponential fitting function (45 days data) or a biexponential (1.5 year data). For the S-type sample (Fig. S4): a so-called stretched exponential function $I(t) = M_i + M_0(1 - \exp(-(t/T_1)^\beta))$ was used.

We would like to quickly discuss the values of the stretch exponent, $\beta$, we obtained for the S sample, see Fig. S4. We found $0.8 < \beta < 1$.

An analysis of the stretched exponential function in terms of a distribution of relaxation times has been performed by Johnston. In this reference, the distribution of $\log_{10}(T_1)$ corresponding to stretched exponentials with different values of $\beta$ is computed. For $\beta > 0.8$, for instance, it is found that the Full Width at Half Maximum (FWHM) of the distribution of the variable $[\log_{10}(T_1)]$ is FWHM< 0.3. This corresponds to $T_1$ values spreading over less than an order of magnitude. For our datasets fitted with the stretched exponential, we consider the FWHM calculated by Johnston, as a function of $\beta$, to be a good estimate of the inhomogeneity in the $T_1$ values. Especially, it gives FWHM= 0.3 for the minimum stretched exponent we observed, $\beta = 0.8$ - which corresponds to a factor of $10^{0.3} \simeq 2$ in the $T_1$ values between the lower and upper sides of the distribution. Such a discrepancy is plausible as it is well within the range of $T_1$ values that we observed in these compounds, whenever a discrete fit (mono or bi-exponential) was performed. It should be remarked that recovering

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For $z > 0$, i.e. within the region defined by the meniscus, the level of the bitumen signal is naturally expected to be lower compared to the region located right below the $z = 0$ mark. As subtracting the full bitumen signal might not be relevant in this case, the subtraction was performed only when it was found to improve the quality of the final fit, and was not performed otherwise.
-in addition to the FWHM- the full distribution of $T_1$ values is a more complicated problem, that supposes the use of regularization techniques.$^2$

S1.4 Evaluation of the quality of the fits.

The fits were performed using a Levenberg-Marquardt algorithm to minimize the Residual Sum of Squares ($RSS$):

$$RSS = \Sigma_i (y_{i,\text{exp}} - y_{i,\text{sim}})^2,$$

where $y_{i,\text{exp}}$ and $y_{i,\text{sim}}$ are respectively the experimental and simulated values. A good estimate of the fit quality was achieved by comparing the noise level $\sigma_n$ to the standard deviation of the fit $\sigma_{\text{fit}} = \sqrt{RSS/(n-p)}$ where $n$ and $p$ are the number of fitted points and fit parameters, respectively.

For the $z$-resolved $T_2$ or $T_1$ experiments, the fits were performed by starting with the minimum number of relaxation components, and increasing it in order to reach $\sigma_{\text{fit}}/\sigma_n < 3.5$ for every slice in a given experiment. Under our acquisition conditions (acquisition gain $rg = 16$, sampling time $dw = 2.5 \mu s$ in analog mode with HADC+ digitizer, readout gradient $G_z = 18 \text{ G} \cdot \text{cm}^{-1}$), we experimentally determined the noise obtained when calculating the average signal on a slice of size $\delta z$, to be $\sigma_n = (0.22 \%) \times (\delta z_{[\text{mm}]} \times (ns)^{-0.5}$ (where $\delta z$ is given in mm and $ns$ is the number of scans for the acquisition, that is, for $\delta z = 1 \text{ mm}$ and for our typical number of scans $ns = 32$, the noise would be within $\pm 0.22\%$ on the previously defined absolute scale).

S2 Additional data related to main text, section 3.5

($z$-resolved $T_1$)
Figure S3: Reconstructed water populations corresponding to the \( z \)-resolved \( T_2 \) and \( T_1 \) experiments on the \( \text{H}_2\text{O} \)-leached I-type sample, at 1.5 year leaching time. (A) and (B) correspond to the short and long relaxation times, respectively.

Figure S4: \( z \)-resolved \( T_1 \) experiments on the S-type \( \text{H}_2\text{O} \)-leached sample, at different leaching times, 45 days and 1 year. The data acquired at 45 days was fitted with a stretched exponential (a stretch exponent \( 0.8 < \beta < 1 \) was observed), while the data acquired at 1 year was better fitted with a biexponential.
S3 The PFG-STE diffusion experiments

S3.1 Description

![Diagram of PFG-STE experiment]

Figure S5: Definition of our basic PFG-STE experiment for measuring diffusion. On the gradient (\(g_z\)) channel, we used two identical trapezoidal gradient pulses, separated by the diffusion interval \(\Delta\).

Diffusion experiments were performed using the PFG-STE sequence from Tanner,\(^3\) see Fig. S5. We sometimes complemented the sequence with a profile readout in order to obtain the \(z\)-resolution. We used trapezoidal gradient pulses, i.e. with linear ramping during the rise and the decrease phases of the gradient. Each of these transient phases lasted a time \(p_{17} = 0.25\) ms, and, in between, the gradient was maintained at the target value \(g_z\) for a time \(p_{18}\), typically equal to 1.25 ms (we used up to \(g_z = 1200\) G·cm\(^{-1}\), that is 67% of the maximum gradient allowed by the probe). We recall that, for a trapezoidal gradient pulse, the effective gradient pulse duration (i.e. the duration of an equivalent rectangular pulse) reads \(\delta = p_{17} + p_{18}\). The minimum \(\tau\) value was \(\tau = 2.5\) ms, which ensures a minimum recovery time of 750 \(\mu\)s between the end of the first gradient pulse and the 2\(^{nd}\) 90° RF pulse.

S3.2 Analysis of the PFG-STE diffusion experiments

For an homogeneous water population obeying the Fick law with a self-diffusion coefficient (along \(z\)) \(D_{zz}\), the propagator \(P(z_0|z,\Delta)\) (where \(P(z_0|z,\Delta)\) describes the probability for a particle initially located at position \(z_0\) to have moved at position \(z\) during the time \(\Delta\)) reads:
\[ P(z_0|z, \Delta) = (4\pi D_{zz}\Delta)^{-1/2}\exp(-z - z_0)^2/4D_{zz}\Delta). \]

The resulting NMR signal intensity after the PFG-STE experiment is predicted to be of the form:\(^3\)

\[ I(q) = \frac{I_0}{2}e^{-(\Delta - \tau)/T_1 - 2\tau/T_2}e^{-q^2D_{zz}(\Delta - \delta/3)}, \quad (2) \]

where \( q = \gamma g z \delta \) is homogeneous to a wavevector, and \( I_0 \) is the signal corresponding to the full thermal magnetization of the observed nuclear spins. Because of the \( \tau \) values chosen for this experiment, only the liquid water was observed. It is important here to remark that we could evaluate \( I_0 \) independently by integration over the reconstructed profiles from the \( z \)-resolved \( T_2 \) experiment, such as the ones shown in main text, Fig. 3.

In the general case of a non-Fickian propagator (e.g. in a porous system with restricted diffusion), the intensity \( I(q) \) can be calculated from the propagator in the approximation \( \delta \ll \Delta \) (short gradient pulses).\(^4\) For clarity, we will first assume that the relaxation processes are not active \( (T_2 >> \tau \text{ and } T_1 >> \Delta) \). In this case, one has:\(^6\)

\[ I(q) = \frac{I_0}{2} \int_{z_0} P_0(z_0) \int_z P(z_0|z, \Delta) \exp(-iq(z - z_0)) dz dz_0, \quad (3) \]

where \( P_0(z_0) \) describes the distribution of observed molecules at the time of the first gradient pulse. For an homogeneous distribution of the water molecules in the pores, i.e. constant \( P_0(z_0) \) within the pores volume, one obtains, by integrating over \( z_0 \):

\[ I(q) = \frac{I_0}{2} \int_{\delta z} P_{\text{avg}}(\delta z, \Delta) \exp(-iq\delta z) d\delta z = \frac{I_0}{2} \tilde{P}_{\text{avg}}(q, \Delta), \quad (4) \]

where the averaged propagator \( P_{\text{avg}}(\delta z, \Delta) \) is obtained after defining \( \delta z = z - z_0 \) and performing integration over all the volume: \( P_{\text{avg}}(\delta z, \Delta) = \int_{z_0} P(z_0|z_0 + \delta z, \Delta) \). As seen also in Eq. 4, the attenuation \( I(q)/I_0 \) corresponds simply to \( \frac{1}{2} \tilde{P}_{\text{avg}}(q, \Delta) \), that is the Fourier transform of

\(^{6}\)The expression given by Tanner and Stejskal\(^4\) describes the attenuation for a simple Spin-Echo (SE) sequence. To adapt this description to the case of the Stimulated Echo (STE), we add a factor \( \frac{1}{2} \), which accounts for the fact that only half of the spins are refocussed in the STE sequence. See also the other work from Tanner.\(^3\)
the averaged propagator with a $\frac{1}{2}$ prefactor.$^d$

We now focus on counting the molecules that have travelled a distance $|\delta z| < a$, where $a$ is an upper boundary in the $z$ direction, during the diffusion interval. The proportion of such molecules is given by:

$$p(|\delta z| < a) = \int_{-\infty}^{\infty} w(\delta z) P_{\text{avg}}(\delta z, \Delta) d\delta z,$$

(5)

where $w(z)$ is a weight function, $w(z) = 1$ for $-a < z < a$, and $w(z) = 0$ otherwise. One can then use Eq. 4, and a property of the Fourier transformation (conservation of inner product):$^e$

$$p(|\delta z| < a) = \frac{1}{2\pi} \int_{-\infty}^{\infty} \tilde{w}(q) \tilde{P}_{\text{avg}}(q, \Delta) dq = \frac{1}{\pi} \int_{-\infty}^{\infty} \tilde{w}(q) \frac{I(q)}{I_0} dq,$$

(6)

where $\tilde{w}(q)$ is the Fourier transform of $w(\delta z)$. For the weight function defined above, we obtain $\tilde{w}(q)$ of the “sinc” form:

$$\tilde{w}(q) = 2a \times \text{sinc}(qa),$$

(7)

with $\text{sinc}(x) = \sin(x)/x$. The function is shown in Fig. S6. One issue is that the NMR experiment allows sampling of the $I(q)$ curve only up to a certain value $q_{\text{max}}$, therefore the integral from Eq. 6 cannot be calculated as such. To circumvent this problem, we define a modified function, $\tilde{w}_{\text{mod}}(q)$ by truncating the original sinc function at $q_{\text{max}}$ (see Fig. S6). By reverse Fourier transformation of $\tilde{w}_{\text{mod}}(q)$, we obtain a modified gate function in real space, $w_{\text{mod}}(z)$. As can be seen from Eq. 7, the $\tilde{w}(q)$ function is more appropriately expressed as a function of the variable $qa$. As the truncation value $q_{\text{max}}$ is fixed by the maximum $q$ attained in a given experiment, the “fidelity”, in real space, of the modified gate function ($w_{\text{mod}}(\delta z)$)

$^d$We use the convention for the Fourier transform: $\tilde{f}(q) = \int_{-\infty}^{+\infty} f(z) e^{-iqz} dz$.

$^e$Both the weight function $w(\delta z)$ and the propagator $P_{\text{avg}}(\delta z, \Delta)$ are real-valued and even functions, therefore, there is no need for complex conjugation in the integral (inner product), even after Fourier Transformation.
to the original one \(w(\delta z)\) is determined by the value of \(a\) that we choose. On the one hand, the best “fidelity” is obtained for\(^1\) \(q_{\text{max}}a >> 1\). On the other hand (in our case) a lower value of \(a\) was needed in order to extract information on the confinement properties from the integral in Eq. 6. As a compromise, we choose \(a = 2\pi/q_{\text{max}}\), so the truncation is performed at the second zero of the sinc function: this way, the resulting \(w_{\text{mod}}(\delta z)\) function does not differ very much from the original gate function, as can be seen in Fig. S6. Therefore, the integral from Eq. 6 will remain a good estimation of the probability that a molecule has travelled a distance \(|\delta z| < a\). To perform the integration, practically, interpolation of the experimental \(I(q)\) points was realized by fitting the points with a phenomenological function (typically, a sum of up to 5 gaussians with different half-widths). The \(q < 0\) part of the integration was simply computed using the \(I(q) = I(-q)\) property.

Figure S6: Weight functions for Eq. 5: ideal gate function \(w(\delta z)\) (dark dots) and modified gate function \(w_{\text{mod}}(\delta z)\) (red line).

We now turn our attention to the case in which relaxation processes are active \((T_2 \sim \tau\) and/or \(T_1 \sim \Delta)\), hence affecting the \(I(q)\) intensity. A simple way to model such relaxation processes is to consider an effective propagator, that is to replace \(P_{\text{avg}}\) by:

\[
P_{\text{avg}}^{\text{eff}} = r(\delta z, \Delta, \tau)P_{\text{avg}}, \tag{8}
\]

where \(r(\delta z, \Delta, \tau) < 1\) is an attenuation prefactor due to relaxation processes. As a result,\(^1\)This criterion ensures that a sufficient number of oscillations of the sinc function occurred before truncation. In this case, the deviation from the ideal gate function after reverse Fourier transform is minimal.
the integral from Eq. 6 will provide a lower bound, \( p(\lvert\delta z\rvert < a)_{\text{min}} \). It seems reasonable to consider that this property will remain in good approximation as we replace the ideal gate function with the modified one \( (w_{\text{mod}}) \), shown in Fig. S6.

Interestingly, we can also put an upper bound to \( p(\lvert\delta z\rvert < a) \). First, we need to notice that we can count the proportion of molecules that have travelled more than the distance \( a \), i.e. \( p(\lvert\delta z\rvert > a) \). For this, one just needs to replace, in Eq. 5, \( w_{\text{mod}}(\delta z) \) with \( 1 - w_{\text{mod}}(\delta z) \). After performing Fourier transform, this leads to replacing \( \tilde{w}_{\text{mod}}(q) \) by \( 2\pi\delta(q) - \tilde{w}_{\text{mod}}(q) \), and ultimately to a modified version of Eq. 6:

\[
p(\lvert\delta z\rvert > a) = \frac{1}{\pi I_0} \left( 2I(q = 0) - \int_{-\infty}^{\infty} \tilde{w}(q) I(q) dq \right). \tag{9}
\]

Similarly here, by considering the relaxation effects, we infer that this integral will give a lower bound, \( p(\lvert\delta z\rvert > a)_{\text{min}} \). Since it is direct that any molecule that has travelled a distance \( \lvert\delta z\rvert > a \) will not have travelled \( \lvert\delta z\rvert < a \), an upper bound for this latter population will be given just by:

\[
p(\lvert\delta z\rvert < a)_{\text{MAX}} = 1 - p(\lvert\delta z\rvert > a)_{\text{min}}. \tag{10}
\]

**S3.3 Description of the PFG-STE decay curve in model situations**

**Spherical pores**

In Fig. S7 are shown the simulated PFG-STE curves corresponding to spherical pores of defined radius, as the simplest model of confinement.

The analytical expression of the decay in this particular geometry is known.\(^5\) We here assumed the water diffuses freely (with the diffusion coefficient of bulk water at ambient conditions, \( D_0 = 2.2 \times 10^{-9} \text{ m}^2 \text{ s}^{-1} \)) inside pores of radiiueses \( r \), \( 0.2 \leq r \leq 0.6 \mu\text{m} \). Considering the values of the diffusion time \( \Delta \) in main text Fig. 6 and 7, \( \Delta \geq 50 \text{ ms} \), one gets, for the biggest value of \( r \) we consider here, \( 0.6 \mu\text{m} \), \( D_0\Delta/r^2 = 277.8 \gg 1 \).
In this regime \((D_0 \Delta / r^2 \gg 1)\), the attenuation follows the simple expression, corresponding to the structure factor of a sphere of radius \(r\):\(^5\)

\[
I(q) \propto \frac{9(qr \cos(qr) - \sin(qr))^2}{(qr)^6}.
\] (11)

As seen in Fig. S7, for radii 0.2 ≤ \(r\) ≤ 0.6 µm, the attenuation obtained with Eq. (11) is comparable with the one seen in the experimental data discussed in main text, section 3.7.1. In particular, we remark that a signal loss by 60% (as in Fig. 6) is obtained for a radius \(r = 0.43\) µm.

Figure S7: Simulated PFG-STE diffusion curves for the two models: (1) spherical pores of different radii below one micrometer (discontinuous lines) and (2) Fickian diffusion at \(\Delta = 400\) ms (continuous lines). The range of \(q\)-values is the same as in Fig. 6 and 7, in main text.

**Fickian diffusion**

Fickian diffusion can be recovered at long diffusion intervals in porous systems in which restriction of motion occurs without confinement. Such a behavior at long \(\Delta\) is sometimes also referred to as the tortuosity limit.\(^6\) In Fig. S7 are shown diffusion decay curves with different values of the diffusion coefficient \(D\) in between \(D = 2 \times 10^{-13} \text{ m}^2 \text{s}^{-1}\) and
\[ D = 2 \times 10^{-14} \text{ m}^2 \text{ s}^{-1} \]. It can be seen that these two extreme values give decays comparable to the one predicted for the 0.6 and 0.2 \( \mu \text{m} \) pores. However, these values of \( D \) are several orders of magnitude below bulk water at ambient conditions; such a discrepancy does not correspond to the typical behavior observed in porous systems, where \( D/D_0 \) is typically closer to unity.\(^6\)

**S3.4  \( z \)-resolved PFG-STE experiment on a I-type sample**

Figure S8: Profiles acquired on the “I-2” sample (2\(^{nd}\) \( \text{H}_2\text{O} \)-leached I-type sample) at 1 year leaching time, after preparation with the PFG-STE sequence. We used a diffusion interval \( \Delta = 200 \text{ ms} \) and two different values of the PFG-STE gradient. Dotted line, or “low gradient”, corresponds to diffusion wavevector \( q_0 = 1.1 \times 10^3 \text{ m}^{-1} \), while continuous line, or “high gradient”, to \( q_2 = 3.3 \times 10^6 \text{ m}^{-1} \). The gradient used for the profile readout was not changed in between the two measurements.

The aim of this section is to cross-check the analysis of the diffusion experiments with varying \( \tau \) performed on the 2\(^{nd}\) \( \text{H}_2\text{O} \)-leached I-type (“I-2”) sample (see main text, section 3.7.1). We additionally used the PFG-STE sequence in combination with a profile readout, in Fig. S8, are shown the resulting weighted profiles for two different values of the PFG-STE gradient \( g_z \). The “low gradient” (that yields \( q_0 = 1.1 \times 10^3 \text{ m}^{-1} \)) was chosen weak enough so that diffusion will not affect the obtained profile.\(^8\) In contrast, based on

\(^8\)However, on the corresponding “low gradient” profile in Fig. S8, the signal in the depth, or i.e. below the surface peak \((z < -0.2 \text{ cm})\) is strongly attenuated because of various relaxation effects occurring during the pulse sequence. This explains why the intensities are underestimated in the depth of the sample, compared to the \( z \)-resolved \( T_2 \) data in Fig. 3. We calculate indeed that, in this region, a maximum attenuation of 60% could occur only because of the relaxation effects during the sequence used in Fig. S8.
the analysis presented on Fig. 7, one can predict that the preparation with “high gradient” ($q_2 = 3.3 \times 10^6 \text{ m}^{-1}$) will lead to a profile representing only the long $T_2$ population. *We indeed verify the matching of the high gradient profile in Fig. S8 to the profile of the long $T_2$ population (Fig. 3).* Thus the findings detailed in main text for the I-2 sample are consistent with the $z$-resolved diffusion experiment.

## S4 Other information

### S4.1 Extrusion process for synthesis of the BWPs (figure)

Figure S9: Extrusion process for the synthesis of the bitumen waste product.

### S4.2 Reproducibility across samples

#### S4.2.1 Comparison of weighing data for 10 and 50 mm diameter tubes

In Fig. S10, are shown the comparison of weighing data, for each BWP, between the H$_2$O-leached NMR tubes (10 mm diameter) and a set of 5 tubes of diameter $\phi = 50$ mm. The data for the NMR tubes tend overall to lie lower, which might be a consequence of alteration (e.g. salt transport) occurring during the repeated NMR measurements, as, to perform those measurements, the water was removed sometimes for 1 or 2 days. A strong variability across the I-type samples is observed in particular, as seen for instance from the important standard
deviation for the 50 mm dataset (error bars) on Fig. S10. Such a variability might originate from different salt repartition within the material from one sample to another.

Figure S10: Comparison of the mass water uptake per unit surface, measured by weighing (gravimetric) method, between the H$_2$O-leached samples in 10 mm NMR tubes and 50 mm tubes (such as the one used for the ESEM measurement). For the 50 mm tubes, the data and error bar correspond respectively to the average and standard deviation on 5 tubes, except for the last point, at $t = 225$ days, where the sample taken for ESEM measurement is not included (for this point, the average and SD is performed on the 4 remaining tubes).

S4.2.2 Reproducibility of NMR measurements across samples

Although the $^1$H NMR measurements presented in the text relate to only one of the two H$_2$O-leached sample, I or S (except for one diffusion measurement described in 3.7.1, that was performed on the second I-type sample, I-2), all measurements described up to section 3.5 were also performed comparatively, at 1 year leaching time on samples I-2 and S-2, to check for reproducibility. For the I-type samples, the profile reconstruction allows to see that the discrepancy in water uptake between I-1 and I-2 (as evidenced in Fig. S10) is related to higher water concentration within the "surface peak" in the former case. No important difference was found within the two different H$_2$O-leached S samples.
Figure S11: Determination of the pore volume fraction (i.e. porosity) in the S-type sample, from an analysis of the ESEM picture Fig. 5.F. (A): Original picture and (B): Picture obtained after analysis, with the pore space colored in white (and the space outside the pores colored in black). The porosity, obtained as the proportion of white pixels in (B), amounts to 41%.
Figure S12: ESEM z-profiles of the different samples after 6 months leaching (H$_2$O).
References


