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# **PHYSICAL MODIFICATIONS OF CLAYS IN A DAMAGE ZONE UNDER THE INFLUENCE OF HYDRATION/DEHYDRATION: MICROSCOPIC AND MESOSCOPIC SCALES STUDY**

**Chadouli K.<sup>1</sup>, Duplay J.<sup>2</sup>, Martinez L.<sup>3</sup>, Géraud Y.<sup>1</sup>, Errais E.<sup>4</sup>, Hiver J.M.<sup>1</sup>, Gonzalez Ruiz L.E.<sup>1</sup>**

<sup>1</sup> Laboratoire Géoresources, UMR 7359 CNRS, Faculté des Sciences, Université de Lorraine, BP -239, Boulevard des Aiguillettes, 54506 Vandoeuvre Lès Nancy Cedex, France.

<sup>2</sup> LHYGES, UMR 7517, Université de Strasbourg, BP -239, 1 Rue Blessig, 67084 Strasbourg Cedex, France.

<sup>3</sup> EOST, UMR 7516 –IPGS, Université de Strasbourg, BP -239, 1 Rue Blessig, 67084 Strasbourg Cedex, France.

<sup>4</sup> Laboratoire de Physique des Matériaux Lamellaires et Nanomatériaux hybrides (LPMNMH), Faculté des Sciences de Bizerte, 7021 Zarzouna-Bizerte, Tunisia.

## **Resumen:**

La argilita Callovo-Oxfordian en la cuenca del Este de Paris (Meuse, Francia) constituyó un lugar potencialmente favorable para almacenar desechos nucleares. Sin embargo, alrededor de las secciones de los túneles que fueron perforados la morfología de la roca, rica en mineral arcilloso, las propiedades petrofísicas y la circulación de fluido podrían cambiar a largo plazo debido a ciclos de humectación y secado.

La evolución de estas propiedades es estudiada en este trabajo, usando simulaciones de hidratación/deshidratación para analizar los aspectos mecánicos y petrofísicos, acoplados con capturas ESEM, análisis de imagen, mediciones de porosimetría por inyección de mercurio y tomografía.

Los estudios se llevaron a cabo en EOST (Ecole et Observatoire des Sciences de la Terre, Strasbourg) y en el Laboratorio de Georecursos (Géoresources laboratory). El objetivo del estudio fue determinar el comportamiento de la argilita, su capacidad de adsorción/desorción, cambios petrofísicos y circulación de fluidos para definir su capacidad de limitar o retrasar la

dispersión de elementos radioactivos.

Los cambios en las condiciones de óxido reducción modifican la repartición de cargas de los elementos, modificando las propiedades de hidratación y, consecuentemente, modificando la estructura de las argilitas así como la textura de la roca. Este proceso de hidratación, a largo plazo, produce una inestabilidad cerca de las fracturas y un micro-agrietamiento progresivo con dominancia de la caolinita, pero para las argilitas hinchadas, se observó micro-agrietamiento más complejo con una multitud de aberturas y cierres de micro-fisuras y poros.

Esos análisis demostraron que la estructura de porosidad es muy dependiente de la mineralogía y que las argilitas son muy sensibles a la circulación de fluidos, los cuales modifican la geometría de los grupos de poros así como el potencial de transporte, además producen cambios en textura y porosidad

**Palabras claves:** Argilita Callovo-Oxfordian, hidratación/deshidratación, aspectos mecánicos y petrofísicos.

**Abstract:**

Callovo- Oxfordian argillite of the Bure site in the Eastern Paris Basin (Meuse, France) constituted a potential favorable to nuclear waste repository. However, around the drilled tunnel sections, the clay mineral-rich rock's morphology, petrophysical properties and fluid circulation conditions may change with time after long term wetting and drying cycles. The evolution of this properties are studied in this work, using simulations of a hydration/dehydration process to analyze mechanical and petrophysical aspects coupled with ESEM captures, images analyzes, mercury injection porosimetry measurements and tomography.

Those technics have been performed at EOST (Ecole et Observatoire des Sciences de la Terre, Strasbourg) and Géoressources laboratory. The objective of this study is to determine argillite's behavior, their water adsorption/desorption capacity, petrophysical changes and fluid circulations; in order to define their capacity to limit or delay radioactive elements dispersion.

Changes in the conditions of redox modify the repartition of elementary charges thus modifying hydration's properties and consequently modifying the structure of argillites as well as the texture of the rock. This hydration process, on a long term, procures a chemical instability near the fractural sets and a progressive micro-cracking in argillites with kaolinite dominance, but for swelling argillites this micro-cracking is more complex where we observe a multitude of opening and closing of micro-cracking and pores.

Those analyses demonstrate that porosity structure is very dependent on mineralogy, and that argillites are very sensible to fluid circulations, those last modify the geometry of pore sets as well as the transport potential and produce changes on texture and porosity.

**Keywords:** Callovo- Oxfordian argillite, hydration/dehydration, mechanical and petrophysical aspects

## **I. Introduction:**

The Callovo-Oxfordian argillites in the region south of the Meuse and north of the Haute-Marne departments (Bure, France) have been qualified by ANDRA (National Agency for Radioactive Waste Management) for the implantation of an underground laboratory to investigate the feasibility of high to medium activity nuclear waste disposal in deep geological formations.

Argillites are clay mineral-rich formations containing various amounts of other silicates and carbonates. These phyllosilicates are fine-grained minerals (micrometer scale) more or less hydrated depending on the clay mineral type. Thus smectite-rich argillites may adsorb water and increase in volume whereas illite-rich argillites are less subject to hydration and change in volume.

Argillites are subject to long-term fluctuations in environmental conditions with the effect of hydration and/or dehydration of interlayered smectite and illite/smectite and ion exchange. In addition, a change in the redox requirements modifies the distribution of elementary charges which, again, change the properties of hydration. Fluid infiltrations through joints as well as wetting of pores network can provide a chemical instability of clays around these joints.

The structure sheet as well as the composition gives them the petrophysical properties of commercial interest. That's why it's primordial to study the physico-chemical properties of each argillite for industrial application.

In the laboratory of Bure, drilled tunnel

sections and consequently the argillites are subject to long-term fluctuations in humidity, temperature and redox conditions with the effect of hydration and / or dehydration of the swelling clays and ion exchange. In addition, a change in the oxidation/reduction conditions may modify the distribution of clay surface charges which may change the hydration properties of the clay minerals. Fluid infiltrations through fissures as well as wetting of the pore network can lead to a chemical instability of clays around these fissures and possible texture changes due to alteration and progressive transformation by eventual crystallization of secondary clays.

Previous studies and observations by Environmental scanning electron microscopy (ESEM) showed progressive cracking of Callovian-Oxfordian argillites, occurring as a result of several hydration/dehydration cycles (Montes, 2002; Montes & al., 2004). This phenomenon is observed in argillites where kaolinite is the principal element; but when swelling clays dominate the cracking is more complex: there has been series of opening and closing of cracks and pores. It is also showed, as did Fouche & al. (2004) for pyrite and carbonates during compaction, that quartz grains harder than the clay matrix play a major role in the opening of cracks after several hydration/dehydration cycles.

Lot of parameters has to be studied quantitatively and qualitatively to define microscopic and mesoscopic micro-cracking and their evolution with time in terms of humidity variations, pores fluid composition, sulfurs and other minerals phases. Temperature variations are considered because they increase the mass

transfer, accelerating the alteration (Rosanne et al., 2003).

The pores network structure is strongly dependent on mineralogy; this has been proved by magnetic susceptibility analysis coupled with iron-fluid and mercury injections. Those techniques also showed the control of connectivity of pores network is controlled by phase's distribution (Esteban et al., 2007).

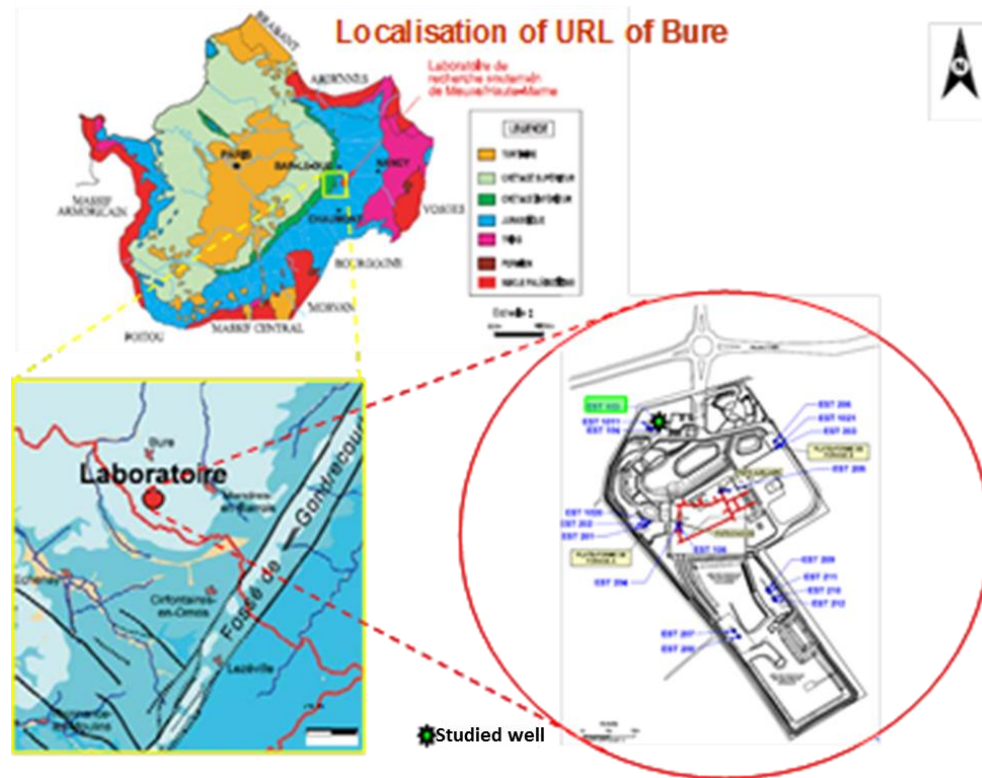
The objective is to simulate the evolution of argillite galleries or barriers during storage and after closure, under the influence of variations in the rate of Hydration, and highlight the role of mineralogy, lithology and microstructures on the possible fluid migrations. This study has a physicochemical modification approach of argillites on nanoscopic, microscopic and mesoscopic coupled with ESEM and tomography observations, kinetics of water adsorption and isotherms, petrophysical methods and quantification with image analysis.

## **II. Geologic and geographic aspects:**

The site of Bure is localized in Meurthe & Moselle region, on the eastern edge of Paris Basin (Figure 1), which is a sub-circular basin, with a maximal elongation of 600km from east to the west and a minimal elongation of 500km from the north to the south.

The substratum consists of a big depression which contains 3000 km of sediments deposited between the Triassic and the Neogene. It consists of alternation of limestone, marl and clays. These clays form the interesting layer and the studied one in this report.

Thus, through this underground laboratory, ANDRA (National Agency for Radioactive Waste Management) can observe in situ behavior of Callovo-Oxfordian clays.

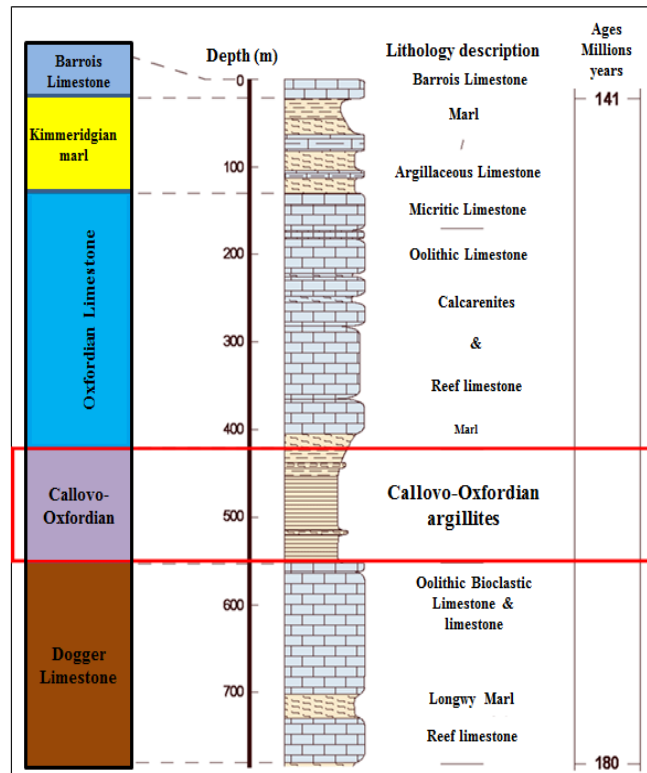


**Figure 1-Localization of Bure site in Paris basin.**

The Callovo-Oxfordian argillites are situated at a depth of 420 m to 550 m (

**Figure 2).** They are called argillites because of their clay content up to 50% and a cementation with quartz crystals and carbonates. They are considered as stable geologically and homogeneous. The mineral composition of the Callovo-Oxfordian argillites varies as a function of depth (ANDRA, 1996). Quartz and

carbonates are the dominant minerals in the first 40 m of the the Callovo-Oxfordian stratum (about 35% and 40%, respectively), whereas clay minerals become dominant below that level (about 40%). In addition, the mineralogy of the clay fraction ( $< 2 \mu\text{m}$ ) changes below 410 m depth with disappearance of illite/smectite interstratified clay minerals and sometimes the appearance of kaolinite.



**Figure 2- lithostratigraphic column of Paris basin with localization of argillites studied**

Overlying limestone and marl formations provide compact properties to the argillites due to the expulsion of pore water, great reduction of the porosity and thus increase of resistance. Moreover, the low permeability of argillite rocks may delay or limit the dispersion of radioactive elements in the environment.

### III. Material and methods:

#### III.1. Samples presentation:

Wells references	Depth (m)	Facies
EST 103 - 07563	418,14-418,25	Unit C3A: black marl with nodules intercalated with bioturbed biomicrite.
EST 103 - 07566	421,15-421,21	Unit C3A: black marl intercalated with bioturbed biomicrite with nodules.

The selected samples were taken from cores of the well “EST-103” (drilled vertically at depth of 526m), “EST-342” and FOR 1106 (**¡Error! No se encuentra el origen de la referencia.**). They have been chosen in order to test the three principal Callovo-Oxfordian facies (Silty, Argillites and Carbonated).

ES T 103 - 07575	429,53-429,59	Unit C2D: black marl and fine calcarenite in loaves and nodules.
ES T 103 - 07585	440,45-440,53	Unit C2D: alternation of black marl and bioturbated bio-calcarentie .
ES T 103 - 07591	446,3 – 446,38	Unit C2C: black silty argillite with loaves and nodules .
ES T 342 - 11521	645,74-645,84	Unit C1:black carbonated clays
ES T 25481-FO R 1106	1.25 -1.30	Unit C2B1: black silty argillites, bioclasts filled by calcite.

**Table 1- table of sample's depth and lithology**

Those Argillites have undergone cycles of Hydration/Dehydration in a chamber at controlled relative humidity and constant temperature (25°) and their porosity networks have been studied using mercury porosimeter.

### **III.2. Methods:**

#### **III.2.1. Analytic protocol:**

The The samples were mineralogically characterized by X-Ray diffraction analysis and the carbonate content was semi-quantatively estimated. The argillite's water sorption and desorption capacity was observed using environmental scanning electron microscope (ESEM), and porosity was

evaluated using mercury injection porosimetry. The images obtained from tomography and ESEM were treated using the Vislog-6 image analysis software, to reconstruct 3D view of samples and calculate porosity and permeability (Figure 3) which were compared to petrophysical results. Moreover, argillites have undergone cycles of hydration/dehydration in a chamber at controlled relative humidity and constant temperature (25°) and the amount of adsorbed/desorbed water was measured with time and with relative humidity. Adsorption isotherms and kinetic data were obtained.

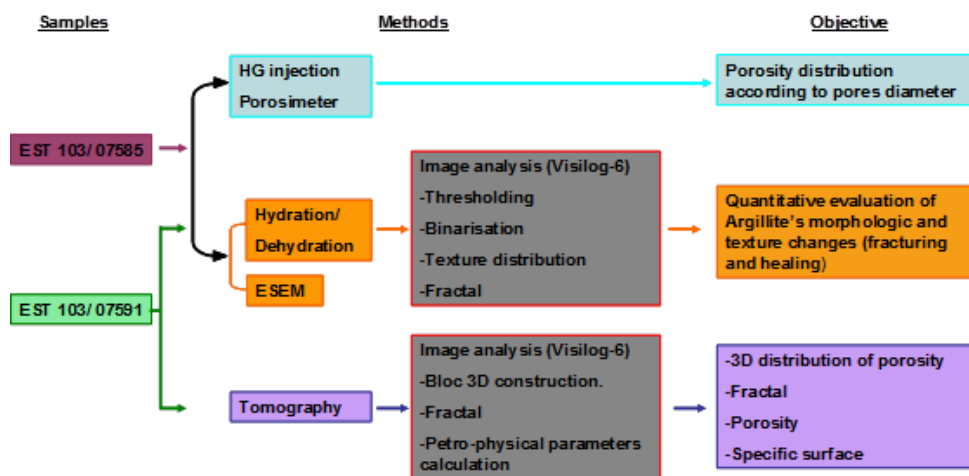


Figure 3- Different methods used to characterize Callovo-Oxfordian argillites

### III.2.2. Hydration/dehydration process and ESEM analysis:

A Philips XL30 ESEM LaB6, fitted with a gaseous secondary electron detector (GSED) to produce a surface image, was used for environmental scanning electron microscope investigations. This microscope is also equipped with a “cooling stage” to control the sample temperature. It allows dynamic procedures of hydration and dehydration of a sample under the influence of relative humidity variations (RH).

Each sample was submitted to hydration/dehydration cycles, by increase and decrease of water activity in the microscope chamber. The ESEM procedure used to simulate accelerated variations in wetting conditions and study

morphology changes of argillite consists basically of three stages: 1) drying with a chamber pressure of 2.3 Torr and temperature of 50 °C to reach a water activity of 0.025 considered here as “reference conditions”, 2) hydration at a constant temperature of 9 °C and pressure of 8.3 Torr to reach a water activity of 0.95, 3)

dehydration at a constant temperature of 9 °C and pressure of 1.1 Torr to reach a water activity of 0.125.

Samples (cubic form of 0.5 cm of edge) were submitted to these hydration/dehydration cycles (HR=95%/HR=12.5%) at fast frequency (10 à 20 min). Images at each stage of hydration were analyzed using image analyzer software to determine changes in the swelling evolution in function of the argillite nature and the number of hydration/dehydration cycles.

### III.2.3 Hydration/dehydration isotherms and water adsorption kinetics

Gravimetric measures were applied to get hydration/dehydration isotherms. The principal of the method is to measure at short or long time intervals the mass of a sample submitted to fixed relative humidity conditions in. The static method of salt solutions is the method used: the sample is placed in a sealed chamber containing a saturated salt solution. The relative humidity of the atmosphere above the salt solution is balanced to a value depending on the used salt. The salts were selected so to scan the



entire range of relative humidity between 10% and 95% at 25 ° C. Prior to the adsorption experiment, the argillite samples were dried at 105 ° C. They were then placed in an enclosure with fixed relative humidity to measure the water adsorption with time until weight stabilization. Weightings were done in short time intervals for kinetic study or in long intervals (until the variation in mass is negligible for the surrounding atmosphere balance studied) for hydration isotherms. For dehydration study, the samples were placed under dehydrating atmosphere (on silica-gel) and weighted until a new stabilization.

#### **III.2.4. Mercury injection (Petrophysical properties)**

The mercury injection porosimetry allows porosity and pore size to be estimated by measuring the volume of mercury injected in previously desiccated and de-aerated samples. The measurements of the injection pressure and of the injected mercury volume were made by steps of increasing pressure on a Micromeritics Ltd apparatus. The measurement principle is to place the sample in a cell and weigh the whole, then extract the gas from the cell, inject mercury and weigh again. The increase in pressure leads to the penetration of mercury into the pores. The measure of the volume of mercury injected permits to calculate the porosity knowing the volume of the sample and using the Laplace law.

The cumulative porosity (i.e. the injected mercury volume) is derived as a function of the pore diameter (i.e. a function of the injection pressure). It gives the total connected porosity at the maximum pressure (150 MPa) and the distribution of pore diameters.

Porosity distribution curves versus pore diameter were obtained from initial samples. They allowed analyzing different functions in the pores network and distinguish transport porosity and reaction one, the modification of volume repartition during experiments and so material reactivity.

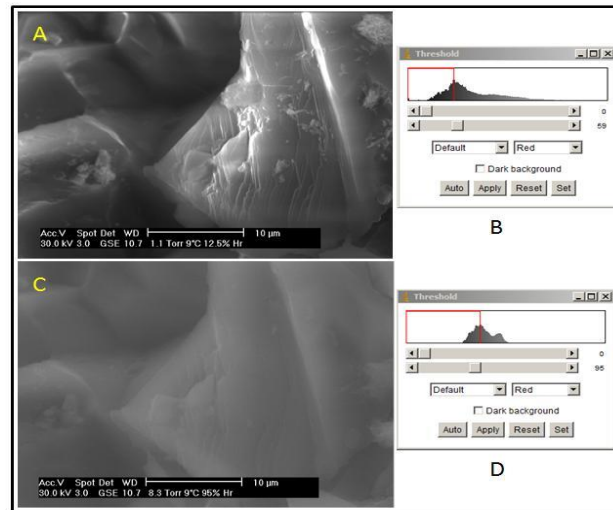
#### **III.2.5. Tomography:**

X-Ray tomo-densitometry is a non-destructive technique based on measuring the attenuation of X-ray beams, which defines a radiologic density. It allows the reconstruction of an object volume following series of scan and measures established from the outside. It permits also analyzing petrophysical properties such as porosity, pore's volume and pore's connection. The tool used is Nanofocus X-ray computed tomography scanner (phoenix nanotom m) with an X-ray detector DXR500L.

In order to observe the evolution of morphology, structure, porosity and permeability of the sample EST 103-7591 before and after hydration dehydration cycles, it has been treated in the tomograph of "l'Ecole des mines de Nancy". This sample was studied because of its strong capacity to humidification in adsorption experiments. 3D reconstruction of this sample allowed calculating porosity and permeability using pores volume and Kozeny Carman law.

#### **III.2.6. 2D Image analyzes:**

ESEM captures, at several relative humidity conditions during hydration/dehydration simulation (Figure 4), are treated using Visilog-6; in ESEM captures, a variation in the morphology of mineral structures of the samples studied as well as a variation in the electrical signal expressed in grey levels are observed.

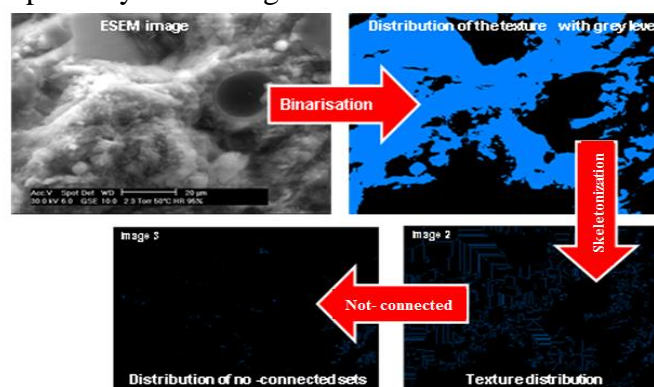


**Figure 4- Variation of ESEM images signal (Grey level)**

The Visilog-6 applications applied are ( **Figure 5**): first, a “simple threshold” which consists of putting all pixels having a grey level less than certain value at 0 and at 1 all pixels with a superior value (Figure 6). 150 images of argillites samples (that have been treated under hydration/dehydration) cycles have been treated.

Then a “skeleton” is applied, it’s an algorithm useful in shape analysis forming

a non-scalar shape analyzer tool. It consists of converting any shape to a set of curves called skeleton. Finally the “border-kill” function is used to eliminate all objects in contact with the border in order to create a new binary image of not connected- skeleton. This last application helps to separate the connected objects from the not-connected ones.



**Figure 5-Visilog-6 steps applications for image analyzes.**

A last measure is done using a fractal

dimension; it allows the study of the

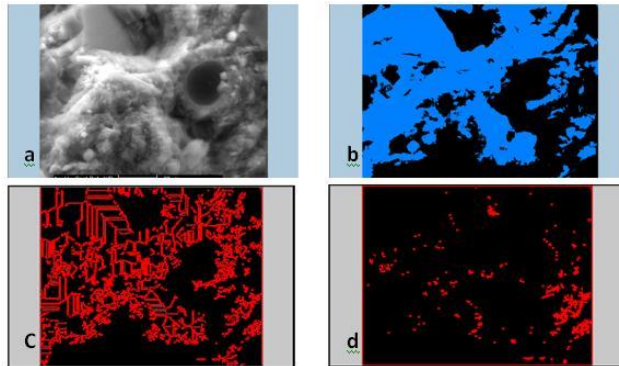
quantitative repetition and the spatial distribution of a set of objects in an image. In this case, it allows us to find correlation between connected and not-connected objects, and to analyze the texture variation with the hygrometry level used. Porosity and permeability are calculated using Kozeny-Carman law:

$$K = \frac{20\phi^5}{S_0^2(1-\phi)^2}$$

With: K: intrinsic permeability;

$S_0$  : specific surface calculated using Visilog-6 (  $m^2/m^3$ );

$\phi$ : Porosity.



**Figure 6- Threshold of an image between 100 and 254 grey's level.**

#### **IV.1. Mineralogy characterization of carbonates content in samples:**

The identification of minerals has been done by X-ray diffraction on all bulk samples and on the < 2 $\mu$ m fraction of 3 samples (7566,

7585 and 7591) for the clay mineralogy. The studied argillites are rich in calcite, dolomite and quartz (7566 and 7563 are specially dolomite rich). The 7585 and 11521 samples are richer in calcite while the 7591 sample is richer in quartz contents. Feldspars and pyrite are also present but in low amounts.

Sample 7566 contains mostly smectite and interlayered illite/smectite, 7585 contains mostly illite, smectite, interlayered illite/smectite and kaolinite. Sample 7591 contains also mostly smectite, illite, interlayered illite/smectite and kaolinite or chlorite.

Calciometry analysis showed that sample 7591 is the poorest in carbonates with a content of 18%. Samples 7566 and 7585 are constituted, respectively, of 67% and 82% of carbonates.

#### **IV.2. Porosimetry analysis:**

Porosity distribution as a function of pore diameters has been analyzed first using mercury injection porosimetry and then image treatment by the Visilog software (Figure 7).

The EST103-7566, EST103- 7585 and EST103- 7591 samples have porosity values of 6.26 %, 0.5% and 16.33% respectively. Sample 7585 is less porous and has a reduced porosity distribution regardless pores diameter unlike sample 7591 which is more porous and has a porosity network more developed but the distribution of porosity is not homogeneous.

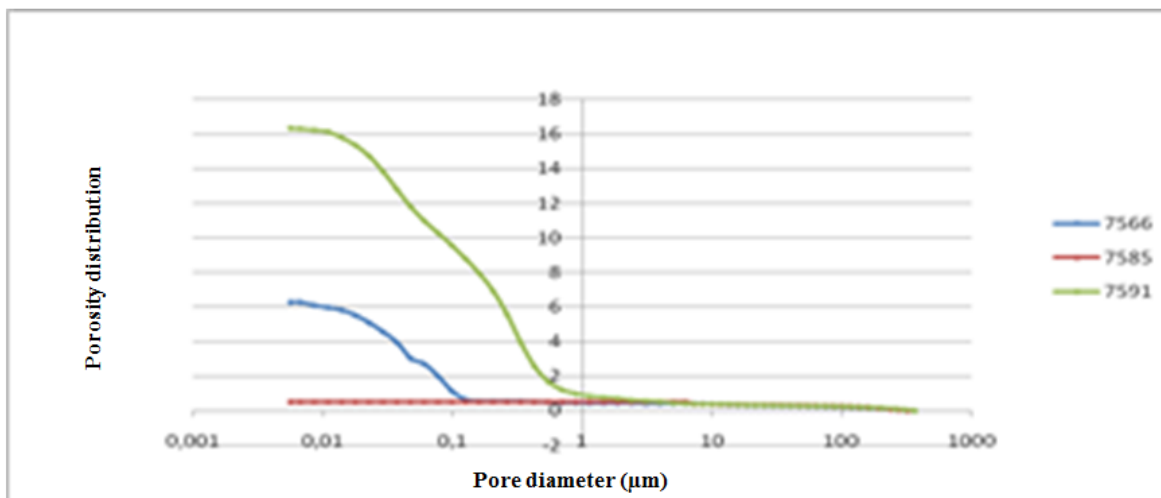


Figure 7- Porosity distribution as a function of pores diameter in samples 7566, 7585, 7591

### IV.3. Adsorption/desorption capacity and Kinetics:

Water adsorption measures of argillites were done at constant temperature 1) as a function of time and 2) as a function of relative humidity (HR). Two types of curves were obtained: hydration/dehydration kinetic curves and hydration/dehydration isotherms.

Each sample showed a different behavior for the hydration/dehydration measures (

Figure 8): samples 7585 and 11521 adsorb less water than samples 7591, 7563 and

7566. However, the amount of water adsorbed by the sample 7585 increases only at relative humidity greater than 60%, unlike the other samples where the increase is gradual since the first stages. Beyond 60% of HR this increase is more important.

Hydration kinetic curves obtained during the first cycle indicate a fast adsorption since the beginning with an amount of water adsorbed reaching a maximal value after 24h. (Figure 9).

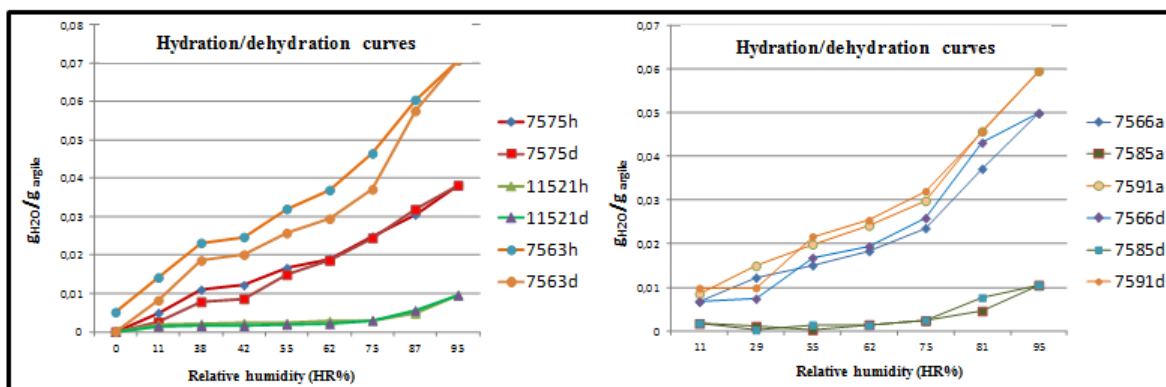


Figure 8- Hydration/dehydration Isotherms at 25°C for samples: 7575, 7563, 7566, 7585, 7591 and 11521; a, h: hydration isotherm; d: dehydration isotherm.

The evolution of water content as a function of hydration/dehydration cycles showed that the amount of water retained after dehydration at relative humidity of 0% increases significantly with cycles

progression, especially in samples of high hydration capacity (Figure 10).

However there are no significant changes in the maximal amount adsorbed at relative humidity of 95% with cycle's progression

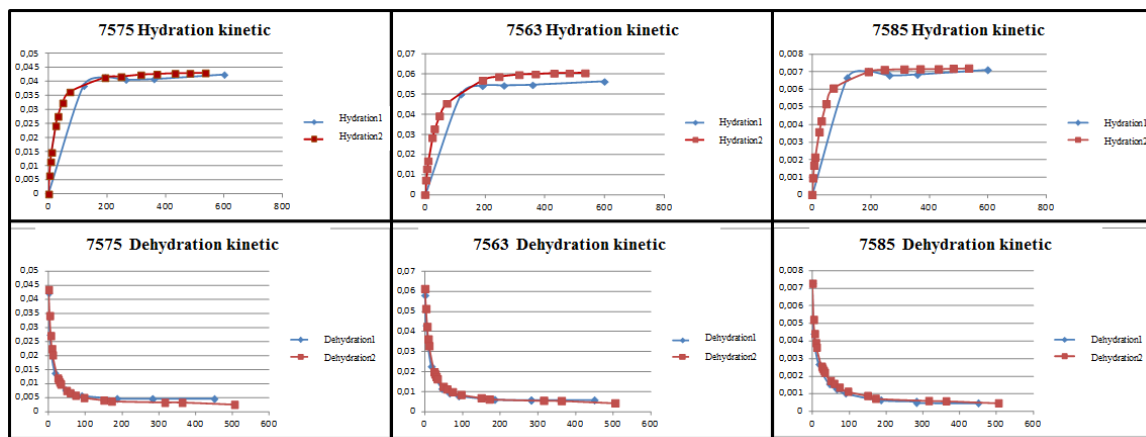


Figure 9- Hydration/dehydration kinetic at 25°C during the two cycles.

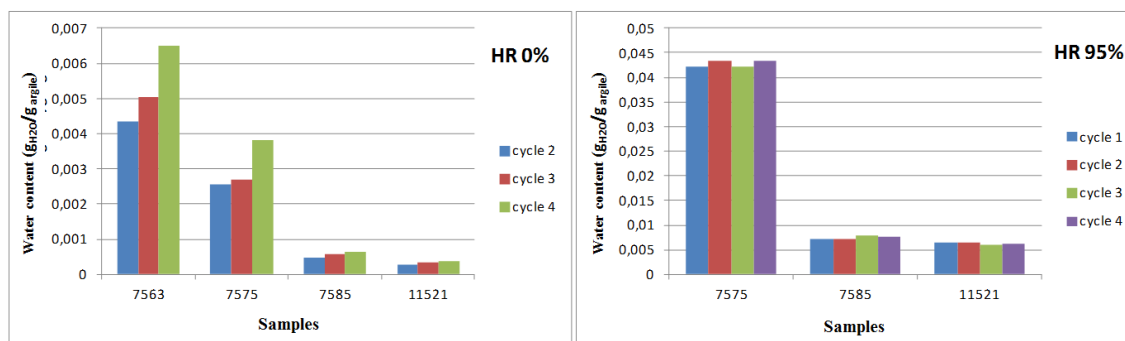
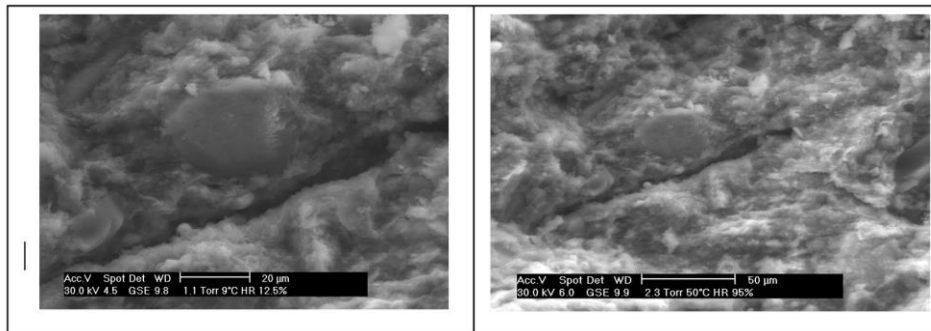


Figure 10- Wetting amount evolution relative to hydration/dehydration cycles

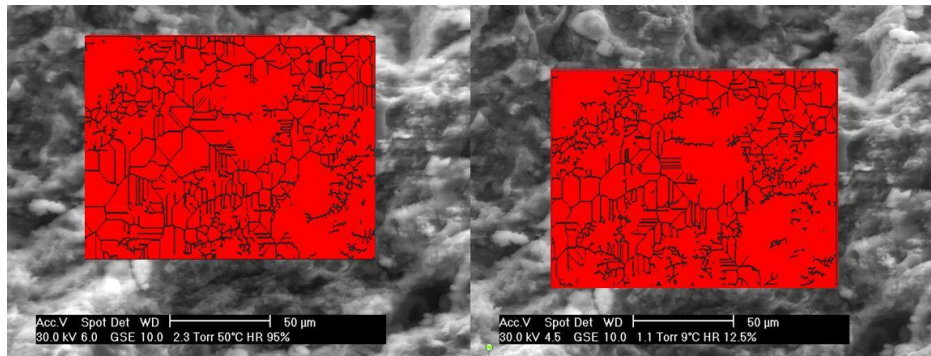
#### IV.4. Textural modification at ESEM through hydration/dehydration cycles:

ESEM observation coupled with image analysis using Visilog- 6 allowed

understanding the texture, distribution and fractal dimension of connected sets as a function of hydration/dehydration changes (HR=95% and 12.5%) (Figure 11 and 12).



**Figure 11- Two images from one argillite sample under different conditions (P, T, HR)**



**Figure 12- Connected set distribution (Sample 7591) obtained from ESEM image treatment at HR=95% (left) during first cycle and HR=12,5% (right) during the 4th cycle of hydration/dehydration.**

échantillon	P (torr)	T (°C)	% HR	Fractale (squelettes connectés)	Fractale (squelettes non connectés)
095Z	3,2	50	95	1,458	1,07
412Z	1,1	9	12,5	1,449	1,13
495Z	8,3	9	95	1,512	1,109
712Z	1,1	9	12,5	1,217	1,105
795Z	8,3	9	95	1,5	1,01
8100Z	8,3	9	95	0,981	0,876
002Z	2,3	50	2,5	1,523	1,345
112Z	1,1	9	12,5	1,243	1,155
195Z	8,9	9	95	1,48	1,263

**Table 2- Conditions (P, T, HR) during images analyzes and fractal treatment.**

The connected set distribution at high relative humidity is observed earlier than at low relative humidity; this can be explained by the pores connexion under wetting influence.

The fractal dimensions prove that the parameters influencing argillite are first relative humidity, pressure and temperature.

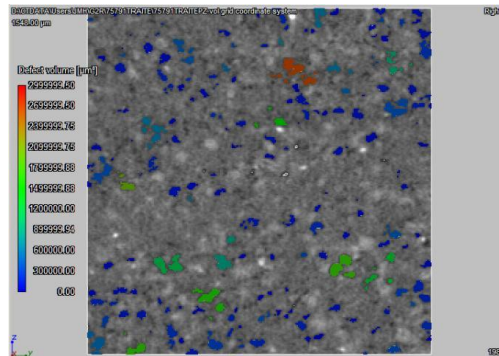


#### IV.5. Tomography:

Images analysis using tomography of 7591 and 7585 samples give 2D images of porosity distribution, then the 3D reconstruction of connected porosity and

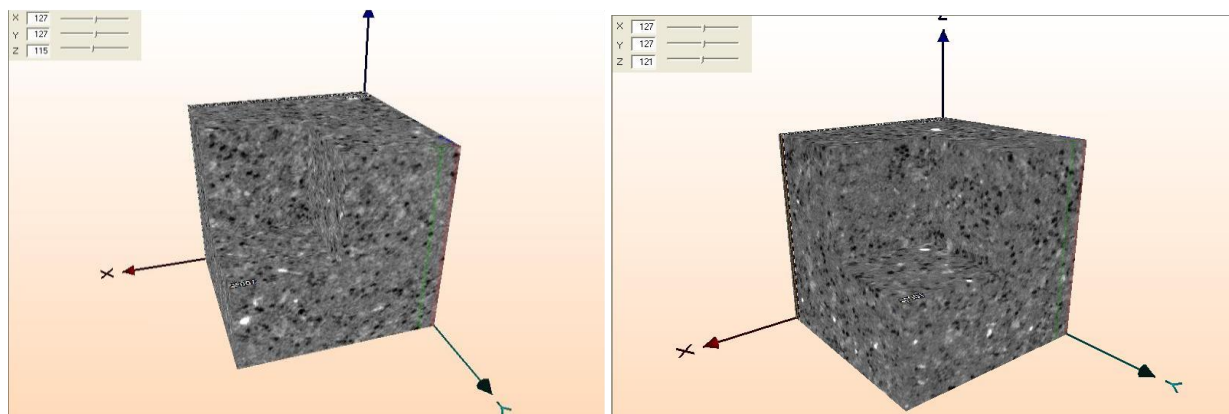
not-connected porosity, illustrate an increase of porosity values after hydration/dehydration cycles

Figure 13).

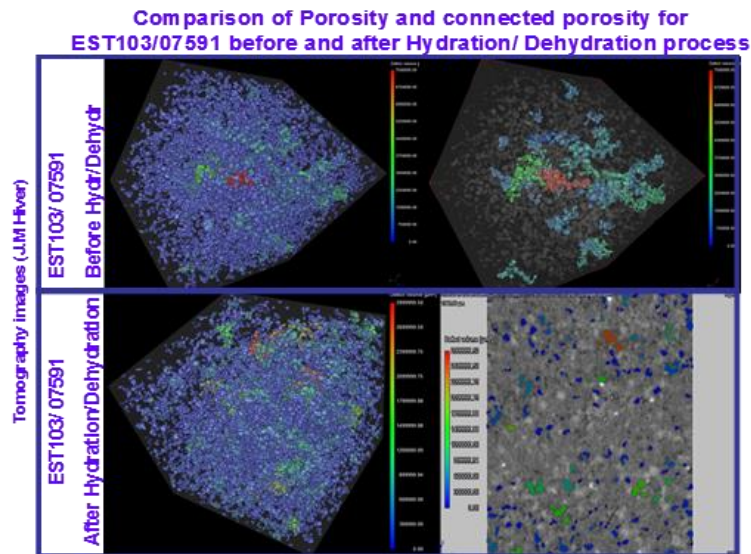


**Figure 13- Porosity distribution of 7591 sample, obtained from image analyzes at HR 12.5%, 4th hydration/dehydration.**

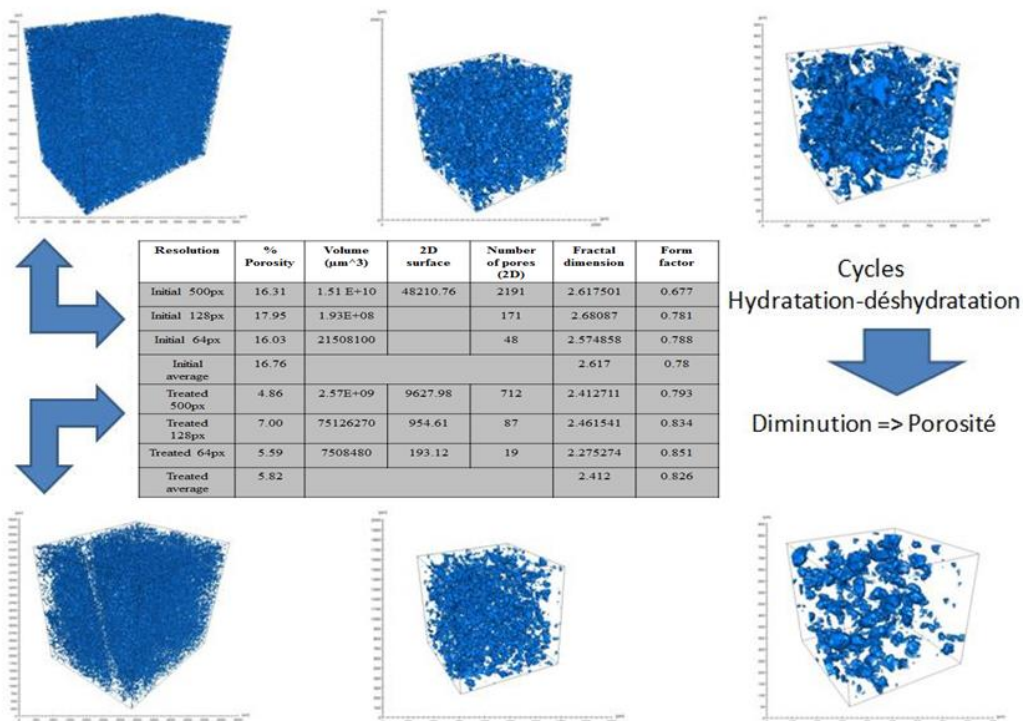
A comparison between initial sample and treated one demonstrates the evolution of porosity and the 3D bloc and in using fractal dimension on visilog-6. Results of both initial and treated sample: porosity calculation, fractal dimension, pore's volume and threshold using Visilog-6 are illustrated in the following image (Figure 14& Figure 15).



**Figure 14- Visilog-6- 3D reconstruction of tomography images of a sample before and after hydration/dehydration cycles**



**Figure 15- 3D reconstruction of tomography images of a sample before and after hydration/dehydration cycles**



**Figure 16- 3D reconstruction of a sample before and after hydration/dehydration cycles.  
Petrophysical and mechanical parameters calculated using Visilog-6**



The figures 15 & 16, illustrate decreasing in porosity, however images from hydration cycles show an increasing of the connection between pores. Hydration/dehydration cycles influence pores volume and connection.

After hydration/dehydration cycles, the fractal calculation, the sample shows changes in its petrophysical parameters, especially pores volume and number, specific surface and also their connection. This porosity decreases significantly after hydration/dehydration cycles in both clays where kaolinite dominates and where swelling clays dominates.

## **V. Discussion and Conclusion:**

The samples studied by X Ray diffraction, are rich in calcite, dolomite and quartz, and contain small amounts of feldspars and pyrite. The clay mineralogy is different from a sample to another with an abundance of smectite. Thus it can influence their water adsorption capacity.

Hydration/dehydration processes applied on those argillites, have an impact on changes in their texture, fracturing, connected elements, porosity, pores volume, hydration capacity, pores connection, morphology and capillary pressure.

The texture variation is observed in the ESEM as a function of the hygrometry level used, the texture of fractal dimension of skeleton can be explained as a variation of structure with hydration/dehydration treatment.

The difference between the initial sample and treated one is significantly observed on 2D images and 3D reconstruction. Those changes are observed and calculated on volume, proportion, shape, fractal dimension of porosity, specific surface, tortuosity and

permeability. The increase of specific surface of porosity is probably due to its volume decrease and the hydration capacity of argillites.

The porosity decreases with fractal dimension which is important in pores volume and number. This can be explained by micro-cracking through hydration/dehydration treatments.

The parameters influencing argillite's porosity with time are first the wetting capacity, pressure and temperature. This can be observed with an increase in the connection between pores and also an increase in their volume.

The porosity volume and their connection rise-up after hydration processes at high relative humidity, which can be due to micro-cracking. But since dehydration cycles are applied we observe a decrease in this porosity, confirmed by fractal dimension calculations using Visilog-6.

This study showed the importance of mineralogy, water adsorption capacity as well as the wetting process on the petrophysical quality of Bure site's potential disposal rock, and thus on its evolution through time and long term storage ability.

## **VI. References**

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