3D reconstruction of poral network at nanoscale from 2D slices.

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Résumé :

En France, les formations d'argilites sont des candidates privilégiées pour le stockage géologique de déchets haute-teneur du fait, en particulier, de leur faible perméabilité. Cependant, de façon à mieux prédire le comportement de tels milieux, la caractérisation du réseau poral est essentielle. De tels matériaux à faible perméabilité peuvent avoir une forte porosité totale mais la majorité de celle-ci est constituée de pores de taille inférieure à 100 nm et dont la distribution reste peu connue. La connectivité et la topologie de ces pores influence les propriétés du milieu poreux, par exemple liées au transport de gaz. De manière à mieux comprendre la structure du réseau poral, des techniques d'imagerie sont utilisées pour générer des images 2D et 3D, comme la microtomographie ou le FIB-MEB. Cette dernière méthode permet une résolution nanométrique (5 à 20 nm) au prix d'un faible volume imagé (en général quelques µm^3).Un tel volume est souvent inférieur au VER (volume élémentaire représentatif), en particulier en ce qui concerne les propriétés de transport. Dans ce travail, nous explorons la possibilité d'écarter les images 2D acquises et d'utiliser une méthode de reconstruction numérique pour générer un volume 3D à l'échelle du VER. Dans ce but, nous étudions un groupe de 180 coupes FIB-MEB parallèles d'une argile synthétique à porosité nanométrique. Nous comparons deux méthodes, basées sur un algorithme de simulation multi-point (MPS), pour reconstruire une configuration 3D cohérente de la structure porale d'après des sections 2D espacées de différentes distances. Les résultats de ces reconstructions sont ensuite comparées au volume de référence en ce qui concerne des caractéristiques globales, comme la porosité, des paramètres morphologiques, comme la distribution de tailles de pores ou l'indice de connectivité, ainsi que des simulations LBM (Lattice Boltzmann) d'écoulement pour évaluer la capacité des méthodes de reconstruction de générer des structures satisfaisantes. La première méthode, qui utiliser une reconstruction séquentielle des coupes, génère des résultats avec une faible sensibilité à la distance de conditionnement mais créé des effets de plan. La seconde méthode, qui agrège les valeurs dans les trois directions orthogonales, permet une meilleure reconstruction de la structure porale. Cette méthode, par contre, est plus sensible aux données de conditionnement. Les valeurs de perméabilités obtenues par simulation sur la seconde méthode sont similaires à celles obtenues sur l'image de référence pour une distance entre les coupes conditionnnantes inférieure à 11 cellules. Par *conséquent, en réduisant le nombre d'images qu'il est nécessaire d'obtenir, cette méthode ouvre la possibilité d'explorer des volumes 5 à 10 fois plus grand que ceux actuellement imagés, permettant une meilleure représentativité du milieu poreux.*

Abstract :

In France, claystone geological formations are privileged candidates for deep underground nuclear waste storage, due, in particular, to their low permeability ability. However, in order to better predict the behavior of such media, the characterization of the pore network is essential. Such low permeability materials may have a high overall porosity but the majority of this porosity is constituted by pores smaller than 100 nm and whose distribution is still poorly known. The connectivity and the topology of these pores influence the porous media properties, for instance in regards to gas transport. In order to better understand the pore network structure, imaging techniques have been used to provide 2D and 3D images, such as microtomography or FIB-SEM. The latter allows a nanometric resolution (5 to 20nm) at the expense of the total imaged volume (usually a few µm3). Such a sample size is usually below the REV (Representative elementary volume), especially in regards to transport properties. In this project, we explore the possibility to distance the imaged 2D slices and use a numeric method to reconstitute the 3D volume at the REV scale. To this end, we study a stack of 180 parallel FIB-SEM slices of a synthetic clay of nanometric porosity. We compare two methods, based on multiple-point statistic algorithm (MPS), to reconstruct a coherent 3D configuration of its porous structure from 2D sections with different sampling spaces. The results of the reconstruction based on sparse data are then compared to the initial volume in regards to global characteristics – such as porosity - , morphological parameters – such as pore size distribution and connectivity index - as well as Lattice Boltzmann flow simulations to assess the capacity of the reconstruction method to provide satisfactory results. The first method, using a slice sequential reconstitution, produces results with low sensitivity to conditioning data but induces planar effects. The second method, aggregating values of three orthogonal directions allows a better reconstruction of the pore structure. This method is, however, more sensitive to conditioning data. The permeability values resulting from flow simulations on the second method are similar to those obtained on the total image for a distance between conditioning slices up to 11 cells. Thus, by reducing the number of direct images to acquire, this method opens the possibility of exploring volumes 5 to 10 times larger than those currently analyzed, therefore leading to a better representativity of the porous medium.

Mots clés: milieu nanoporeux, reconstruction, morphologie, échelle du pore

1 Introduction

The study of fluid transport in nanoporous geomaterials is essential for various applications such as gas storage (CO2, H2), geological storage of radioactive water or non-conventional reservoirs exploitation. In order to assess the properties of a nanoporous material, a DRP (Digital Rock Physics) approach may be undertaken. Such an approach requires the acquisition of a 3D image, subsequent image treatment so as to perform a numerical upscaling of the physical properties of the medium (permeability, dispersivity, etc.). For geomaterials, a commonly used image acquisition technology is the microtomography. However, the resolution of microtomography is usually of around 1 µm, recent techniques allowing resolution of 0.3 µm. In nanoporous materials such as clayrock or shales, pore size distribution range is often below such resolution. Recently, SEM (Scanning Electron

Microscopy), which allows nanometric resolution of 2D slices, has been coupled with FIB (Focused Ion Beams) to allow a 3D description at a few nanometers resolution [1]–[3]. This technique, however, remains rather difficult and is constrained by the total size of the image (usually a few μm^3), leading to questioning in regards to the representativity of the imaged pore structure, especially when considering more complex behavior such a multi-phase flow or reactive transport.
We therefore evaluate the capacity of spacing the imaged slices and numerically reconstructing the 3D volume at a larger scale to accurately recover the desired properties of the material. To this end, we will test two different reconstruction methods ([4], [5]) on a fully-known set of FIB-SEM images of synthetic clay (from [6]). In order to assess the performance of the approaches, the reconstructed results will be compared through a variety of morphological descriptors and effective properties.

2 Materials and Method

2.1 Initial data

The sample studied is a synthetic clay, *i.e.* compacted illite powder, with an overall porosity of 32%. The sample has been studied and imaged by [6]. The image obtained from the FIB-SEM has then been segmented by a specific method as described in [6]. For the purpose of the present study, we used the segmented image of size 1096x1095x180 voxels with a resolution of 5nm provided by [6] as reference data. Considering the size of this domain and the subsequent computing time, we only considered a set of 180x180x180 voxels for development purpose within this study.

2.2 Reconstruction methods

Multiple reconstruction approaches exist and have been applied in geological context [4], [5], [7]–[9]. Most of these works were tested on microtomography images such as ones of sandstone. In the present work, we will focus on two Multiple Point Statistics (MPS) approaches based on the work of [4] (the 3DA-MPS algorithm) and [5] (the s2Dcd algorithm). Both approaches were tested using the SKUA-Gocad software.
A first step in reconstruction methods is the acquisition of training images (TI). A training image is essentially a conceptual model that should try to include all pore structure patterns for porous media. The training image can be 2D or 3D and may be obtained through imaging techniques such as SEM and FIB-SEM. For MPS approaches, a template should be defined to capture multi-point statistics. The template is used to scan the TI and collect patterns. Following [4], a 9x9 template is used in the present study.
The s2Dcd (sequential 2D conditioning data) algorithm is based on a sequential 2D-MPS simulation along a given surface (among the three possible directions) using the training image that describes heterogeneity along the direction of this surface and conditioned by the points already simulated in that surface or given as conditioning data at the beginning of the simulation.
The 3DA-MPS (3D Aggregation – Multi-Point Statistics) algorithm reconstructs the 3D volume node by node and constrained by the porosity. At first, the probability of a given node to be associated to void space is equal to porosity, then, when sufficient conditioning data becomes available, measured statistics from the TI are being used. For each node randomly picked in the domain, a data event is extracted in each direction. In each direction, a probability value is computed using a classic direct sampling.

In the present work, the reconstruction will be based on training images from the three directions and conditioning data will be selected from the full 180x180x180 images with different spacing in one direction (every 5, 11, 22 images). For each simulation method and set of parameters, 5 reconstructed samples were generated to assess the variability of the results. Computing time was around 20 minutes for s2Dcd and 40 minutes for 3DA-MPS method, though could be improved using high-performance computing.

2.3 Morphological characterization

In order to assess the performance of the reconstruction methods, a set of morphological descriptors will be used. The descriptors were chosen as they are usually considered relevant for key physical processes such as flow, multiphase flow, transport (including reactive transport), etc.

- Porosity
- Pore Size Distribution (PSD) was computed using the XLib plug-in of ImageJ [10] using both the continuous PSD and the discrete PSD approaches.
- Euler characteristic, which provides a first idea of the connectivity of the medium, was computed using the MorphoLibJ plug-in of ImageJ [11]
- Isolated pores characteristics such as volume and specific surfaces were also obtained using the MorphoLibJ package
- Percolating pores, i.e. pores connecting two opposite faces of the reconstructed images, were obtained using the Object Counter process of ImageJ
- A connectivity index is calculated as the ratio of the larger pore volume to the total pore volume.

On top of that, permeability of the reconstructed samples was computed using the LBM software Sailfish [12].

3 Results and discussion

The morphological descriptors representing D_{10}, D_{50} and D_{90} (as obtained from the PSD), I (connectivity index) as well as the porosity are summarized as ratio to the reference values in the below Table.

Algorithm	Conditioning distance (slices)	D10	D50	D90	I	ε
s2Dcd	5	74%	72%	61%	97%	97%
	11	74%	69%	55%	96%	91%
	22	74%	64%	52%	95%	83%
3DA-MPS	5	95%	100%	93%	100%	91%
	11	91%	75%	57%	98%	116%
	22	93%	67%	60%	77%	47%

For both reconstruction methods, the result is degraded with the distance of the conditioning data for most of the descriptors. For porosity and connectivity index, this degradation is more significant for the 3DA-MPS approach than for the s2Dcd algorithm. On the contrary, the PSD for 3DA-MPS is closer to the reference PSD than s2Dcd. When the spacing between conditioning slices is larger than the template size (9x9), some larger pores are no longer recovered.

The ratio of permeability of the reconstructed medium to the permeability of the real medium is studied for the 3DA-MPS data. For a spacing between conditional data of 5 voxels, this ratio is close to one in each flow directions. At a spacing of 11 cells, this ratio decreases to an average of 0.6, the reconstructed permeability being lower than the real permeability. The result is even more degraded for higher distances between conditioning data. Indeed, the pores tend to be disconnected under such conditions, leading to a significantly lower permeability. These results therefore validate the 3DA-MPS method of reconstructing the porous medium for well-conditioned simulations: distances between conditioning slices being less than the template simulation size, here set at 9 cells.

References

[1] L. Holzer, B. Münch, M. Rizzi, R. Wepf, P. Marschall, and T. Graule, "3D-microstructure analysis of hydrated bentonite with cryo-stabilized pore water," *Appl. Clay Sci.*, vol. 47, no. 3–4, pp. 330–342, 2010.

[2] S. Hemes, G. Desbois, J. L. Urai, B. Schröppel, and J. O. Schwarz, "Multi-scale characterization of pore microstructures in Boom Clay (HADES, Mol, Belgium) using a combination of μ-CT, BIB-SEM and serial FIB-SEM techniques," *Microporous Mesoporous Mater.*, vol. 208, pp. 1–20, 2014.

[3] Y. Song, C. a. Davy, D. Troadec, A.-M. Blanchenet, F. Skoczylas, J. Talandier, and J. C. Robinet, "Multi-scale pore structure of COx claystone: Towards the prediction of fluid transport," *Mar. Pet. Geol.*, vol. 65, pp. 63–82, 2015.

[4] H. Okabe and M. J. Blunt, "Pore space reconstruction of vuggy carbonates using microtomography and multiple-point statistics," in *Water Resources Research*, 2007.

[5] A. Comunian, P. Renard, and J. Straubhaar, "3D multiple-point statistics simulation using 2D training images," *Comput. Geosci.*, 2012.

[6] S. Gaboreau, J. C. Robinet, and D. Prêt, "Optimization of pore-network characterization of a compacted clay material by TEM and FIB/SEM imaging," *Microporous Mesoporous Mater.*, vol. 224, no. March, pp. 116–128, 2016.

[7] Y. Wu, C. Lin, L. Ren, W. Yan, S. An, B. Chen, Y. Wang, X. Zhang, C. You, and Y. Zhang, "Reconstruction of 3D porous media using multiple-point statistics based on a 3D training image," *J. Nat. Gas Sci. Eng.*, 2018.

[8] M. Gao, X. He, Q. Teng, C. Zuo, and D. Chen, "Reconstruction of three-dimensional porous media from a single two-dimensional image using three-step sampling," *Phys. Rev. E - Stat. Nonlinear, Soft Matter Phys.*, 2015.

[9] L. Chen, L. Zhang, Q. Kang, H. S. Viswanathan, J. Yao, and W. Tao, "Nanoscale simulation of shale transport properties using the lattice Boltzmann method: permeability and diffusivity," *Sci. Rep.*, vol. 5, p. 8089, 2015.

[10] B. Münch and L. Holzer, "Contradicting geometrical concepts in pore size analysis attained with electron microscopy and mercury intrusion," *J. Am. Ceram. Soc.*, vol. 91, no. 12, pp. 4059–4067, 2008.

[11] D. Legland, I. Arganda-Carreras, and P. Andrey, "MorphoLibJ: Integrated library and plugins for mathematical morphology with ImageJ," *Bioinformatics*, vol. 32, no. 22, pp. 3532–3534, 2016.

[12] M. Januszewski and M. Kostur, "Sailfish: A flexible multi-GPU implementation of the lattice Boltzmann method," *Comput. Phys. Commun.*, vol. 185, no. 9, pp. 2350–2368, 2014.

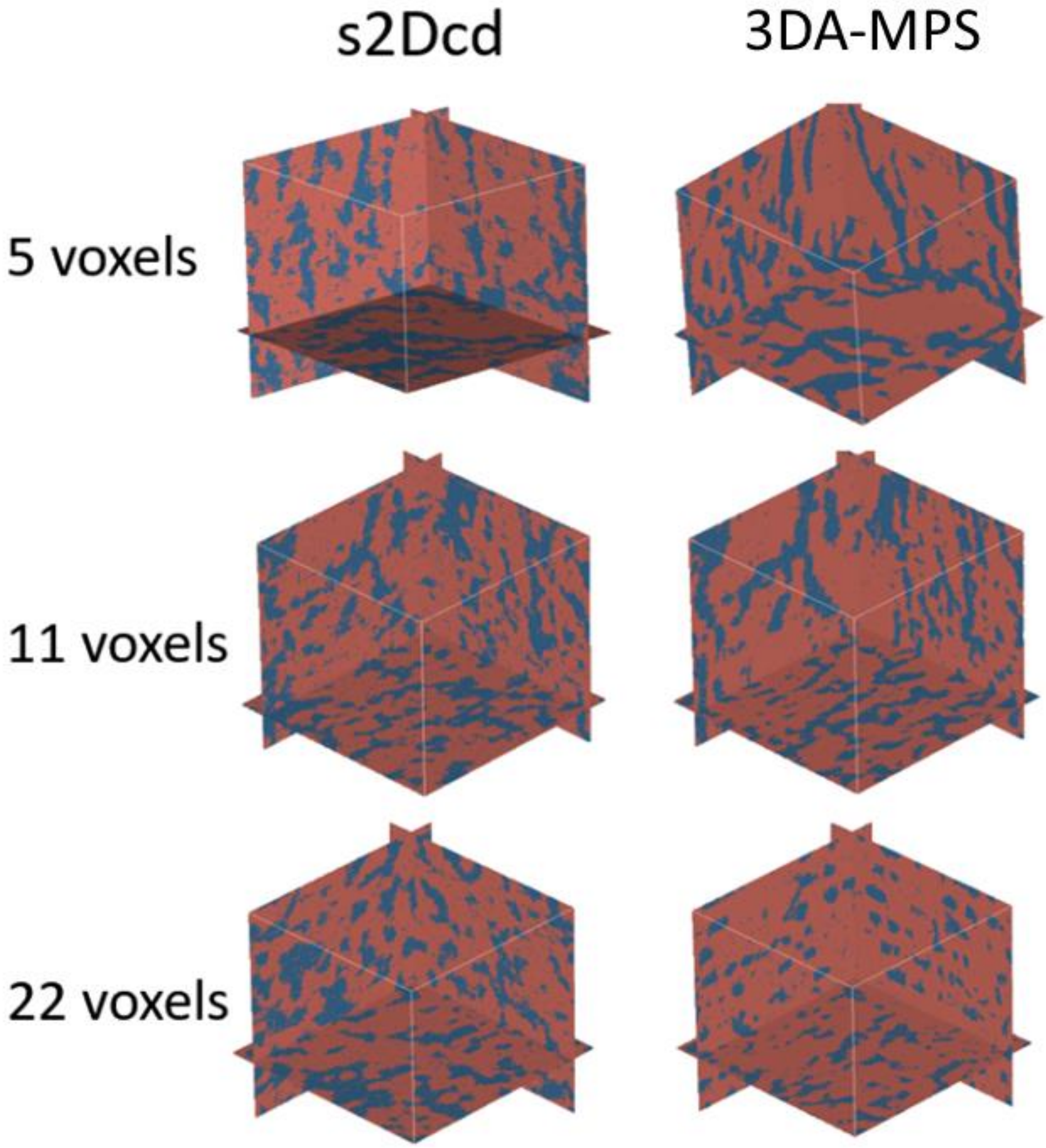

Figure 1 – Reconstructed samples with each algorithms and for different spacing between conditioning data. (blue – pore space, red – solid)