# Effects of the distribution and geometry of porosity on the macroscopic poro-elastic behavior: Compacted exfoliated vermiculite

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*Abstract*— For high temperatures and high pressures applications, materials such as compacted exfoliated clays are interesting materials to substitute asbestos. Because of the thermo-mechanical loadings applied to material, it is fundamental to determine the mechanical properties of such medium. An approach is proposed in order to evaluate the poro-elastic properties of such porous material. The definition of representative elementary volume (REV) is based on scanning electron microscopy (SEM) analysis.

Moreover, an approach based on Mori-Tanaka technique, with an iterative process, is proposed because of the specific features of studied porous material: Ellipsoidal porosities with specific orientation distribution, transversely isotropic matrix and porosity volume ratio about 30%. This model is illustrated by an application with the simulation of the behavior of compacted exfoliated clays. In this paper, the elastic properties are determined, but other poroelastic properties could be calculated by the same way.

*Keywords*—poro-elasticity, homogenization, porous media, orientation, distribution

## I. INTRODUCTION

In recent years, argillaceous material have spurred considerable interest in the sealing industry partly because of their potential for large range of temperature and pressures compared to standard sealing materials, as elastomers. Some compacted exfoliated clays as vermiculite represents an interesting alternative to asbestos and to compacted exfoliated graphite for high temperature sealing applications. Indeed, because of its chemical and thermal stability, many applications, as high temperatures and high pressures sealing, are possible [1, 2]. The vermiculite has an argillaceous structure made by the stacking of two types of flakes. Moreover, it is subjected to expansion under heat or chemical effect (exfoliation). From the compaction of exfoliated vermiculite, it could results a porous material with very low permeability. However, the substitution of asbestos by compacted exfoliated clay for sealing application requires a complete characterization of its poro-thermo-elastoplastic

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behavior. Indeed, the design of a material involves an accurate knowledge of the mechanical properties. For example, the elastic recovery of such material is essential to ensure the sealing quality. However, an exhaustive experimental characterization is difficult. Moreover in order to optimize the microstructure for an improvement of the macroscopic properties, it is essential to develop predictive method.

It seems interesting to be able to establish the link between the macroscopic behavior of the studied materials and their microstructure. Considering the high cost of the experimental identification of the poro-elastic behavior, a micromechanical approach can be interesting. This approach consists, by a change of scale, to characterize the macroscopic effective behavior of the real heterogeneous material from the knowledge of the constituent's properties and from the knowledge (complete or partial) of the spatial distribution of these constituents. The heterogeneous material is so substituted, through this process, by an equivalent homogeneous material. Theses approaches, widely developed in the field of the heterogeneous materials [3], also supply a pertinent knowledge on the mechanical behavior of the porous medias [4, 5] and transport phenomena in these material [6-9]. It should be underline that most of the papers about the poroelastic properties evaluation deal with rocks. In order to facilitate the development of the use of argillaceous material for sealing applications, constitutive relationships must be developed, that predict the bulk mechanical properties of such material as a function of the microstructure.

The explicit analytical expression of equivalent behaviour was established by solving the problem of Eshelby inclusion for simplified geometries (an elastic ellipsoidal inclusion in an infinite elastic matrix). The first results of homogenization due to Voigt, Reuss and Hill [10]. These results are classified as simplified approaches. The method of dilute distributions [11], the self-consistent method [10] and variational or energy methods developed by Hashin and Shtrikman [12-15] are also included in this type of approach. Berryman [16] and Kachanov [17] discussed the approaches by the Eshelby tensor for the poro-elastic behavior. They studied the mechanical properties of materials with cracks or pores. The homogenization of the poro-elastic behavior of porous media with ellipsoidal pores was recently studied by Dormieux et al. [18, 19] and Ulm et al. [20].

The homogenization method of periodic structures has been developed by many authors, using rigorous mathematical basis

[21-24]. The microstructure is described by the periodic repetition of a basic known cell. The implementation of periodic homogenization methods often requires numerical calculations on a basis cell. Chakraborty [25] has developed a technique of homogenization based on FEM for periodic porous microstructures. Poutet et al. [26] has determined the effective mechanical properties for a periodic porous medium. The homogenization problem is solved numerically on the elementary cell to determine the macroscopic mechanical properties of the medium. Three structures were considered: deterministic, fractal and random.

Recently, the development of the numerical computing tools allows to solve the local homogenization problem on very rich representative elementary volume (REV) [27-29]. The numerical homogenization methods are based on FEM solution of the homogenization problem on REV. Grondin [28] studied the concrete by the numerical homogenization methods. Zeghadi [29] worked on metallic materials. Claquin [27] worked on the interfaces of granular media. Delenne et al. [30] proposed a model of cohesive granular materials made of aluminium rods glued. The mechanical behaviour and failure of these materials have been studied numerically and experimentally.

In present study, the objective is not to develop a new method of homogenization but to use an analytical model that provides good estimates of effective elastic properties of the porous materials studied in this article, which have the following features:

- Porosity volume ratio relatively high (30% to 40%).

- Anisotropic properties of constituents (vermiculite flakes).

Indeed, some homogenization techniques provide incorrect values when the porosity volume ratio is too high. Thus, the proposed iterative process consist of gradually increase the porosity volume ratio, so that at each step the porosity ratio added remains low [31].

In a first part, after a brief recall of the poro-elastic laws, the basic principles of the homogenization and more particularly the principles of the approach of Mori-Tanaka [32, 33] are presented. Then, an introduction of an iterative homogenization process is proposed. The purpose of this iterative process is to improve the forecast of the macroscopic behavior when porosity volume fraction is relatively high. Finally, after a convergence analysis of the iterative process, parametric studies highlight the effect of microstructure parameters (shape ratio, porosities orientation...) on the macroscopic behavior of compacted exfoliated vermiculite.

# II. MATERIAL

The clay used in this study is vermiculite and accurately is exfoliated vermiculite Granutech E from China (Yuli). The mean size of these vermiculite particles was in the range of 0.7 and 2 mm. In order to reduce the size of these particles, different treatments were tested [34]. Vermiculite powder samples were provided by the LCME (Laboratoire de Chimie Moléculaire et Environnement). The ultrasonic irradiation in  $H_2O_2$  (hydrogen peroxide) leads to reduce the particle size. After, this treatment, it was revealed by laser particle size

measurement, the average particle size is about 2.0  $\mu$ m. It is important to underline that the particle size of vermiculite, used in this study, is very different from the size of the vermiculite particles commonly encountered. In order to obtain samples with good mechanical properties, the vermiculite powder is compacted with a pressure about 80 MPa. The mechanical compaction was realized at room temperature (20 °C).

In order to study the microstructure (stacking of vermiculite flakes, porosity ...) of the compacted exfoliated Vermiculite (CEV) samples, a microscope LEO 440 Stereoscan with a tungsten filament was used. The observations were made on the lateral side of cylindrical samples and on samples with a flat surface, cut with a microtome Leica RM2165, as shown in fig. 1. Six parallelepipedic samples (4mm×5mm×4mm) were analyzed. The large size of sample allows to do several observations for each samples in order to obtain a representative average information on microstructure of CEV (Fig. 1).



Fig. 1 Samples used for SEM observation: (a) cylindrical sample (b) cutting plane (c) parallelepiped sample (d) CEV sample

The geometrical properties of the porosity network were estimated with the use of an image processing software (ImageJ [35]). The SEM images were processed to analyze the microstructure of different CEV samples and to determine the sizes of porosities. Pores appear as a very dark area allowing us to distinguish and quantify them by image analysis. The pores were identified by thresholding of the pore brightness to produce a binary image [36]. The dark area fraction was evaluated and the pore volume ratio was determined.

For the studied porous material, the SEM micrographs of vermiculite highlight that porosities result mainly from the lake of vermiculite flakes. Thus, this material can be models by the juxtaposition of vermiculite cluster whose flakes and all porosities are oriented in the same direction (Fig. 2). Each family thus constitutes a transverse anisotropic material, because the axis of revolution of porosities is perpendicular to the plan of isotropy of the matrix.



Fig. 2 SEM Image of a sample of the vermiculite

# III. GOVERNING EQUATIONS

#### A. Poro-elasticity governing equations

The study of the mechanical behavior of the porous media was formalized in the 1930's with the works of Terzaghi [37]. Then some developments were proposed by Biot [38] with the first general formulation of the reversible behavior of the porous media. The Biot's theory supposes that the medium consists of two phases: a solid phase (matrix) and a porous phase saturated by a fluid (Fig. 3). The behavior of porous media depends on the behavior of each phase, the pressure of the interstitial fluid and the exchanges or non-exchanges of fluid with exterior. There are two cases:

a- The drained configuration (fluid exchange with outside), the pressure remains constant inside the porous media.

b- The no drained configuration. There are no fluid exchanges with exterior.

The linear poroelastic law has the following form:

$$\begin{cases} \underline{\sigma} = \underline{C} : \underline{\varepsilon} - M \cdot \underline{B} \cdot \frac{m}{\rho_0^f} & \text{(no drained configuration)} \\ \underline{\sigma} = \underline{C}_0 : \underline{\varepsilon} - \underline{B} \cdot p & \text{(drained configuration)} \end{cases}$$
(1)

 $\underline{\underline{\sigma}}$ : Stress tensor,  $\underline{\underline{C}}$ : No drained rigidity tensor,  $\underline{\underline{C}}_{0}$ : Drained rigidity tensor,  $\underline{\underline{\mathcal{E}}}$ : Strain tensor, M: Biot coefficient,  $\underline{\underline{B}}$ : Biot tensor, m: fluid mass supply per unit of initial volume, p: interstitial pressure and  $\rho_{0}^{f}$ : Fluid density at initial configuration.



The subject of this paper is the mechanical behavior of the CEV media and not the fluid transport properties. All porosities open and occluded are taken into account. Moreover, in this first study, the matrix is supposed to be linear elastic.

# B. Modeling

The method employed in this study was adapted from the approach developed by Mori and Tanaka [32, 33] to determine elastic properties for porous media. The aim of the proposed approach is to predict the effect of the micro morphology of the porous media on the macroscopic behavior.

The four characteristics of the proposed study were:

a- Vermiculite, matrix of studied porous media, assumed to be anisotropic and it could to be considered as transverse isotropic [40, 41]. Indeed, this matrix issues from the stacking of vermiculite flakes. The vermiculite flakes are predominantly perpendicularly to the axis of compaction (Fig. 2).

b- Porosities are modeled as ellipsoidal porosities.

c- The porosity ratio of the studied media is relatively high (in the range of 30 % to 40%).

d- The porosities are oriented with a specific orientation distribution.

Using the Mori-Tanaka approach for a two-phase media, the effective elastic tensor, is given by the following equation:

$$\underline{\underline{C}}_{\underline{\underline{m}}} = \underline{\underline{C}}_{1} + f_2 \left[ f_1 \underline{\underline{P}}_{\underline{\underline{m}}} + \left( \underline{\underline{C}}_{\underline{\underline{m}}}_2 - \underline{\underline{C}}_{\underline{\underline{m}}}_1 \right)^{-1} \right]^{-1}$$
(2)

 $\underbrace{C}_{\equiv MT}$ : homogenized rigidity tensor,  $\underbrace{C}_{\equiv 1}$ : rigidity tensor of

the phase 1,  $\underbrace{C}_{\equiv_2}$  : rigidity tensor of the phase 2,  $f_1$  : volume

fraction of the phase 1,  $f_2$  : volume fraction of the phase

2, 
$$P$$
: Hill's polarization tensor.

For transverse isotropic constituent with ellipsoidal heterogeneities, Withers [42] proposed analytical solution for the polarization tensor. This tensor depends only on the components of the rigidity tensor of the matrix, the shape and orientation of the ellipsoidal porosity. For complementary detail on the tensor of polarization, the reader can refer to the article [42]. Withers [42], Sevostianov and al [43] and Kirilyuk and al [44] have proposed analytical solutions for different shape of heterogeneity (cylinder, sphere, ellipsoid...), in a transversely isotropic media.

### C. Iterative process

The iterative process is based on the achievement of the actual media with successive additions of heterogeneities. Thus the porosity volume ratio added at each step of homogenization remains low, in accordance with the basic assumptions of the homogenization method [31]. Thus, the achievement of the actual heterogeneous media with a porosity volume ratio  $\phi$  is obtained by successive constructions of N intermediate medias, obtained by additions of a low increment of porosity  $\Delta \phi$  until the actual porosity volume ratio  $\phi$  is reached. The principle of the iterative process is summarized on fig. 4. For the studied material, the heterogeneities are the porosities in a matrix made up of vermiculite flakes. The used homogenization technique is based on the Mori Tanaka

method previously recalled. Fig. 5 illustrates the iterative process algorithm, which was implemented on the Maple software [45].



Fig. 4 Principle of the iterative process



Fig. 5 Algorithm of the iterative process

#### IV. NUMERICAL RESULTS AND DISCUSSION

# A. Effect of the iterative process

The studied material is clay, a porous media with spherical porosities (Fig. 6). The mechanical properties of the transverse isotropic matrix are displayed on table 1 (values from [46]). Also, in this paragraph, the effective macroscopic media remains transverse isotropic because porosities are spherical and so the plan of isotropy is unchanged.

In order to validate the iterative process, a parametric study of the effect of the iteration number is proposed. The macroscopic elastic properties obtained while varying the porosity volume ratio from 0 to 100% are summarized on Fig. 7. The results are display for various numbers n of iteration: n=1 (direct scheme), n=100 and n=200. From these results (Fig. 7), it is obvious that the direct homogenization trends to overestimate the macroscopic properties. Indeed, for a porosity volume ratio about 30%, the elasticity modulus,  $E_1$ ,  $G_{13}$  and  $E_3$ obtained with the direct diagram are respectively about 24%, 26% and 42% higher than those calculated with the iterative process. Moreover, the iterative process converges very quickly. Indeed, from 100 iterations, the results obtained are very close to convergence limits. One of the main interests of this iterative process is to propose a model for materials with significant porosity (or heterogeneity) volume ratio. This method enables to take into account, in an indirect way, the interaction between porosities, or inclusions for heterogeneous materials such as composite.

For low porosity volume ratio, all homogenization methods lead approximately to the same equivalent homogenous behavior [31]. But for high porosities fraction, the scattering of results increases. Some methods, like dilute approximations [11, 47], give nonphysical results for porosity fraction above a given value. Moreover, the various homogenization methods (dilute scheme, Mori-Tanaka scheme and self-consistent method) provide different results, especially since there is an important mismatch between the elastic properties of the constituents (as for porous medium). Therefore, an iterative process of homogenization has been proposed, and applied in order to solve these problems.

It should be underline that the various homogenization approaches supply similar homogenized properties when the iterative process is used. Furthermore, some methods, such as the self-consistent technique [10], which could provide erroneous macroscopic properties for porosity volume ratio up to 30 %, provide identical values those other approaches with iterative process.

The iterative process provides the same equivalent behavior of the porous media whatever the homogenization method used and whatever the porosity fraction of the medium. This iterative process unifies the results of the classical homogenization techniques proposed in the literature, which usually agree only for low porosity volume ratios.

In the following parametric studies, the iterative process was used with 100 iterations.



Fig. 6 Modeling study material with spherical porosities.

material	$E_1 = E_2$ [MPa]	E <sub>3</sub> [MPa]	G <sub>13</sub> [MPa]	$v_{12} = v_{31}$
Clay	2	10	1	0.25

Table 1 Elastic properties of a transversely isotropic matrix [46]



Fig. 7 Effect of iteration number on the effective properties: Mori-Tanaka scheme

# *B.* Effect of shape ratio of ellipsoidal porosity on the macroscopic mechanical behavior

In order to study the influence of the microstructure of the porosity network, two parameters are studied in this article. Firstly, the effect of the shape ratio of the ellipsoidal porosity on the macroscopic elastic properties is analyzed. With the previously proposed iterative process, the properties of a porous media are calculated according to the porosity volume

ratio and for three shape ratios: (Fig. 8 and Fig. 9)  $w = \frac{c}{a}$ :

- Oblate ellipsoid porosity: w = 0.1
- Spherical porosity: w=1
- Prolate ellipsoid porosity: w = 10

The vermiculite flakes, skeleton of the studied porous material, are anisotropic and can be considered as transverse isotropic. Indeed, the matrix resulting from the compression of sheets of vermiculite. Due to the preferred orientation of these sheets perpendicular to the axis of compression, the transverse isotropy assumption appears justified. The SEM images show that the pores in CEV samples are due to the absence of vermiculite sheets, these pores were modelled by ellipsoids (Fig. 10), whose the three axes are defined on the Fig. 8. The micromechanics approach is implemented in order to evaluate the influence of the morphology of the pore on the macroscopic elastics properties of the material.

It is assumed that all ellipsoidal porosities are aligned in the matrix. The matrix properties are mentioned in the Table 1. The matrix is supposed transverse isotropic. Thus, the homogenized media is also transversely isotropic.







Fig. 9 The three types of porosity



The main results are summarized on the Fig. 11. It is checked that the porous media is transversely isotropic for all kind of inclusions. These results underline the effect of the geometry of porosities on the macroscopic behavior of porous material. For example, for a porosity ratio of 15%, the elastic module  $E_1$  and  $E_3$  vary respectively from 66% and 82% for shape ratio w=1 and w=10. For a fixed porosity volume ratio, when the



Fig. 11 Modulus  $E_1$  and  $E_3$  versus the porosity volume ratio for various shape ratios (w =0.1, 1 and 10). Mori-Tanaka scheme

# *C.* Effect of the distribution on the macroscopic poroelastic properties

Mechanical properties and anisotropy of porous material are strongly influenced by the orientation of the porosities. The SEM images shows that the pores are relatively aligned perpendicular to the compaction axis of the vermiculite powder. In order to analyze the effect of the geometric distribution of ellipsoidal porosities, a relatively simple approach is proposed. In order to obtain some information of the effect of the porosity's orientation distribution on the macroscopic properties of the porous material, three configurations are analyzed:

**a-Simplified real distribution:** The porous media is considered to be made with various families of porosity with an orientation distribution for each one these families. Each family is defined by an orientation, specified with the angle  $\beta$  (Fig. 12) and by a percentage of the total porosity proportion (Table 2 and Fig. 13). These values are obtained from the observations and analysis of SEM images and are compatible with the values in [48].

It was assumed that there is no correlation between the orientation and the spatial distribution.

The poroelastic properties of each family are obtained with the previous approach. Then in order to take into account the distribution of orientation the macroscopic elastic tensor is obtained by the following equation:

$$\underline{\underline{C}}_{\underline{\underline{m}}T} = \underline{\underline{C}}_{1} + \left[ f_1 \langle T \rangle^{-1} + \left( \underline{\underline{C}}_{\underline{\underline{m}}2} - \underline{\underline{C}}_{\underline{\underline{m}}1} \right)^{-1} \right]^{-1}$$
(3)

Where: 
$$\langle T \rangle = \sum_{i=2}^{N} f_i \overline{T_i}$$
 and  $T_i = \left[ \underbrace{P}_{\equiv i} + \left( \underbrace{C}_{\equiv i} - \underbrace{C}_{\equiv 1} \right)^{-1} \right]^{-1}$ 

 $\underbrace{\underline{C}}_{\underline{B}MT} : \text{ effective elastic tensor, } \underbrace{\underline{C}}_{\underline{B}i} : \text{ elastic tensor of the } \\ \text{phase i, } f_i : \text{volume fraction of the phase i, } \underbrace{\underline{P}}_{\underline{B}i} : \text{The Hill's (or polarization) tensor for the phase i.}$ 

In order to take into account the orientation of porosities (the change between local coordinate, axis of the ellipsoid and the structural axis of the porous media):  $\overline{T}_{ijkl} = Q_{ip}Q_{jq}Q_{kr}Q_{ls}T_{pqrs}$ 

The matrix of rotation Q is defined as follow:



Fig. 12 Ellipsoidal porosity orientation

Angle $\beta$ [degree]	$\beta = \pm 25$	$\beta = \pm 15$	$\beta = 0$
Volume fraction [%]	9	18	46

Table 2 Spatial distribution of porosity





Fig. 13 (a) SEM images of CEV and (b) orientation distribution of porosity

b- Random distribution: To describe the orientation of ellipsoidal pores, an orientation distribution function (ODF) is introduced [49]. The ODF function was used to characterize the spatial distribution of porosity. This function provides more accurate results than decomposition in family. It should be noted that the random case is a completely randomly oriented porosities (i.e. randomly oriented in three dimensions). The equivalent behaviour is calculated using the following equation:

$$\underbrace{\underline{C}}_{\underline{\underline{m}}} = \underbrace{\underline{C}}_{\underline{\underline{m}}} + \left[ f_1 \langle T \rangle^{-1} + \left( \underbrace{\underline{C}}_2 - \underbrace{\underline{C}}_{\underline{\underline{m}}} \right)^{-1} \right]^{-1}$$
(4)  
Where:  $\langle T \rangle = \sum_{i=2}^N f_i \overline{T_i}$  an

Where:

$$T_{i} = \left[ \underbrace{P}_{\equiv i} + \left( \underbrace{C}_{\equiv i} - \underbrace{C}_{\equiv 1} \right)^{-1} \right]^{-1}$$

$$\overline{\overline{T}}_{ijkl} = \frac{\int_{-\pi}^{\pi} \int_{0}^{\frac{\pi}{2}} \iint \overline{T}_{ijkl}(\alpha,\beta,\gamma)ODF(\alpha,\gamma)\sin(\beta)d\alpha d\beta d\gamma}{\int_{-\pi}^{\pi} \int_{0}^{\frac{\pi}{2}} ODF(\alpha,\gamma)\sin(\beta)d\alpha d\beta d\gamma}$$
(5)

$$ODF(\alpha, \gamma) = \exp(-s_1 \alpha^2) \exp(-s_2 \gamma^2)$$
(6)

Where  $s_1$  and  $s_2$  are factors that control the orientation distribution.

For a random orientation of inclusions s1 = s2 = 0, so ODF = 1.  $\alpha$ ,  $\beta$  and  $\gamma$  are the three Euler angles.

c- Aligned ellipses: All ellipses are aligned along the y axis.  $s_1 = s_2 = \infty$  and  $ODF(\alpha, \gamma) = \delta(\alpha)\delta(\gamma)$  where  $\delta(y)$  is Dirac's function. The aligned case corresponds to the ellipsoidal porosity perfectly aligned along the axis 2.

The simulations are carried out for porous medias having the following characteristics: a shape ratio w=0.1, and the transverse isotropic characteristics of the matrix are mentioned in Table 1.

The main results are summarized in Fig. 14. The elastic properties and anisotropy of porous material are strongly affected by the porosity distribution. For a porosity volume ratio about 25%, the Young's modulus  $E_1$  ( $E_3$  respectively) for the simplified real distribution decrease (respectively increase) by 12% (by 45%) compared to the case of aligned ellipses.



Fig. 14 Modulus  $E_1$  and  $E_3$  versus the porosity volume ratio for various spatial distributions

#### V. CONCLUSION

The proposed model is based on an iterative homogenization approach for the linear elastic behavior. The behavior of a linear porous media is obtained first of all by successive homogenizations of intermediate porous material made by repeated additions of low porosities volume ratio.

and

The approach presented in this article, proposes a tool for the evaluation of the elastic properties of a porous media with ellipsoidal porosities by taking into account the microstructure (resulting from SEM micrographs, micro tomography observations...). This method is developed for large range of porosity volume fraction, even high porosity volume ratios. The influence of the shape ratio and the spatial distribution of ellipsoidal pores were evaluated. The mechanical properties of the CEV depend significantly on the shape ratio of the porosity. The mechanical properties of the homogenized CEV are directly related to the spatial distributions of ellipsoidal pores.

It should be underlined that it is sometimes very difficult to characterize some macroscopic poroelastic characteristics, such as the Biot's coefficient and the drained rigidity coefficients for low permeability materials. The proposed model is an efficient tool to obtain the essential properties to design and optimization of a porous material. Another difficulty is to measure the properties of the matrix (without porosity). However experimental techniques, such as the micro indentation, can provide these properties. An alternative is to use the proposed approach as an inverse problem. I.e. the macroscopic properties can be measured whereas the characteristics of the constituents, here the matrix, can be evaluated numerically with the proposed prediction technique. The macroscopic properties as well as the microstructure characteristics then become the input data to evaluate the properties of the constituents of porous material. The methodology presented is of general applicability to porous media.

The perspectives, at short-term, are the confrontation between experimental measurements on compacted exfoliated vermiculite and numerical results obtained with the proposed approach.

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