CFMR

approches multi-échelles en mécanique des roches

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Propriétés poroélastiques de roches calcaires oolithiques

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OUTLINE

 motivation investigated material and posed problem multiscale homogenization method

 two steps SC scheme for porous oolitic rocks (two porosities) simplified microstructure and homogenization scheme step I : microscale → mesoscale (micropores) step II : mesoscale → macroscale (mesopores + ..)

application to an oolitic limestone
 experimental results
 comparison model-experiments

conclusions & prospects

investigated materials

- porous oolitic rocks such as limestones
- studied in CO₂ geological storage research programs
- high porosity (0.2 0.3), high permeability, isotropic materials
- *granular type* structure (grains = porous oolites)
- many different facies depending on geological history

microstructure oolitic limestone (example)

Ghabezloo et al. (2009)



posed problem

interpretation of macro experimental results (scale : 1-10 cm)

mechanical tests (deviatoric compression tests, oedometer test, hydrostatic test etc.)

poromechanical tests : drained - undrained (Biot coef)

including microstructural information

mineral content (calcite)

porosity structure (pore scale distribution and pore shape)

properties of constituents (micro-indentation results)

linear poroelasticity \Leftrightarrow **linear thermoelasticity**

- porous oolitic rock type composites (limestones, iron ore etc.) = grain + matrix
- granular type rock : high volume fraction of oolites
- first approach : Hashin Composite Sphere Assemblage model (Hashin (1962)) applied to linear poroelasticity
- mathematical analogy linear poroelasticity linear thermoelasticity ⇒ theoretical background well established (Schapery (1968), Rosen and Hashin (1970), Siboni and Benveniste (1991), Hervé and Zaoui (1993), Benveniste (2008) among many others).
- many recent and similar approaches on concrete cement based materials (Bary and Béjaoui (2006), Hashin and Monteiro (2002), Heukamp et al. (2005))

Hashin Composite Sphere Assemblage (CSA)

cement paste Bary and Béjaoui (2006) (among others)



Hashin and Monteiro (2002)

composite sphere assemblage (CSA) model

two step Four Phase model for porous oolic rocks



key influence of *ITZ* between oolite-matrix, Nguyen et al. (2011)

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effective bulk modulus k_{hom}^{II}

Four Phase (step II) – DSC (step I)





effective shear coefficient $\mu_{\rm hom}^{II}$

Four Phase (step II) – DSC (step I)



effective Biot coefficient b_{hom}^{II}

Four Phase (step II) – DSC (step I)



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Lavoux limestone (oolitic facies)



microstructure of Lavoux limestone (X50)



microstructure of Lavoux limestone

- oolites : spherical grains of concentric layers, diameter range $100\mu m 1mm$
- micrite : *microcristalline calcite*, spherical grains, diameter range $1\mu m 5\mu m$
- oolites : assemblage of micropores & micrite grains
- sparite : spar calcite, diameter range $20\mu m 100\mu m$

microstructure of oolites (Grgic (2011))



mercury porosimetry : Lavoux limestone

Entrance radii of pores r (µm) 80.5459 64.9677 52.4346 52.4346 7.227.3847 7.227.3847 7.227.3847 7.22845 7.27509 7.4793 7.27509 7.4793 7.27509 7.4793 7.27935 7.27935 7.2793 7.27935 7.2793 7.27935 0.01999 0.0195757 0.0195757 0.0195757 0.0195757 0.019575757 0.019575757575 (%) 1st injection \rightarrow Total Hg porosity porosity n_{Hg,tbulk} *n_{Hg, bulk}* = 23.4 % n_{Hg,bulk} Fraction of the total Hg (%) Ω Mercury pressure (MPa)

no continuous phase : SC scheme step I



2 Steps (SCS) : 2 scales *micro/meso* pores



constituents and volume fractions

three phases at meso level (step I)

$$\Omega = \Omega_o + \Omega_b + \Omega_c \tag{1}$$

- o : porous oolite (poroelastic material)
- b : *meso* pores

 c : sparitic cement (pure calcite, elastic solid phase) volume fractions

$$f_o = \frac{\Omega_o}{\Omega} , f_b = \frac{\Omega_b}{\Omega} , f_c = \frac{\Omega_c}{\Omega}$$
$$f_o + f_b + f_c = 1$$

assumption : scale separation *micro-mesopores*

- *intra oolitic voids* of spherical shape, diameter range $0.002 \,\mu m < r < 2 \,\mu m$, referred as *micropores*, index *a*,
- *inter oolitic voids* of spherical shape, diameter range $2 \mu m < r < 50 \mu m$, referred as *mesopores*, index *b*

total pore volume Ω_p & pore volume fraction f_p

$$\Omega_p = \Omega_a + \Omega_b \tag{2}$$

$$f_p = \frac{\Omega_a + \Omega_b}{\Omega} = f_a f_o + f_b \tag{3}$$

$$f_a = \frac{\Omega_a}{\Omega_o} \quad , \quad f_b = \frac{\Omega_b}{\Omega}$$
 (4)

typical data for Lavoux Limestone

$$f_p = 0.26, \, f_o = 0.74, \, f_a f_o = 0.14$$
 , $f_a = 0.19$, $f_b = 0.12$

Step I : SC scheme (oolites)

simple formula, isotropic case (spherical distribution), SC

$$\frac{k_o^I}{k_o^s} = \frac{1 - f_a}{1 + \alpha_o^I \left(k_o^s - k_o^I\right) / k_o^I} \quad , \quad \frac{\mu_o^I}{\mu_o^s} = \frac{1 - f_a}{1 + \beta_o^I \left(\mu_o^s - \mu_o^I\right) / \mu_o^I}$$

$$\alpha_o^I = \frac{3 \, k_o^I}{3 \, k_o^I + 4 \, \mu_o^I} \quad , \quad \beta_o^I = \frac{6 \left(k_o^I + 2 \, \mu_o^I\right)}{5 \left(3 \, k_o^I + 4 \, \mu_o^I\right)}$$

unknowns : k_o^s , μ_o^s : properties of solid oolite phase (microcalcite or micrite).

 $k_o^I \& \mu_o^I$: homogenized elastic properties of porous oolite, to be compared to experimental microindentation data.

Linear poroelastic properties of oolites - mesoscale (]

isotropic poroelastic relations, oolite, step *I* (mesoscale)

$$\underline{\underline{\sigma}} = \left(3k_o^I \mathbb{J} + 2\mu_o^I \mathbb{K}\right) : \underline{\underline{\varepsilon}} - b_o^I P_a \underline{\underline{i}}$$

$$f_a - f_a^0 = b_o^I \underline{\underline{i}} : \underline{\underline{\varepsilon}} + \frac{P_a}{N_o^I}$$
(5)

micromacro compatibility relations (homogeneous solid phase)

$$b_o^I = 1 - \frac{k_o^I}{k_o^s} \quad , \quad \frac{1}{N_o^I} = \frac{b_o^I - f_a}{k_o^s}$$
 (6)

(Biot (1941), Biot (1977), Cowin (2004), Coussy (2004))

Step II : SC scheme, meso → macroscale

$$\underline{\underline{\Sigma}} = \mathbb{C}_{\mathsf{hom}}^{II} : \underline{\underline{E}} - \underline{\underline{B}}_{a}^{II} P_{a} - \underline{\underline{B}}_{b}^{II} P_{b}$$

$$\left(\phi_{a} - \phi_{a}^{0}\right)^{II} = \underline{\underline{B}}_{a}^{II} : \underline{\underline{E}} + \frac{\overline{P}_{a}}{N_{aa}^{II}} + \frac{P_{b}}{N_{ab}^{II}}$$

$$\left(\phi_{b} - \phi_{b}^{0}\right)^{II} = \underline{\underline{B}}_{b}^{II} : \underline{\underline{E}} + \frac{P_{a}}{N_{ba}^{II}} + \frac{P_{b}}{N_{bb}^{II}}$$

$$(7)$$

 ϕ_a porosity associated with Ω_o , at the scale of the rve Ω

$$\phi_a = \frac{\Omega_a}{\Omega} = \frac{\Omega_a}{\Omega_o} \frac{\Omega_o}{\Omega} = f_a f_o \quad , \quad \phi_b = \frac{\Omega_b}{\Omega} = f_b \tag{8}$$

 $\underline{\underline{\Sigma}}$ stress tensor, ϕ_a , ϕ_b porosities, $\underline{\underline{E}}$ strain tensor, P_a , P_b pore pressures, \mathbb{C}_{hom}^{II} overall drained stiffness tensor, $\underline{\underline{B}}_a^{II}$, $\underline{\underline{B}}_b^{II}$ Biot tensors, N_{ij}^{II} solid Biot moduli

Step II : isotropic case

$$\underline{\underline{\Sigma}} = \left(3k_{\text{hom}}^{II} \mathbb{J} + 2\mu_{\text{hom}}^{II} \mathbb{K}\right) : \underline{\underline{E}} - b_a^{II} P_a \underline{\underline{i}} - b_b^{II} P_b \underline{\underline{i}} \\ \left(\phi_a - \phi_a^0\right)^{II} = b_a^{II} \underline{\underline{i}} : \underline{\underline{E}} + \frac{P_a}{N_{aa}^{II}} + \frac{P_b}{N_{ab}^{II}} \\ \left(\phi_b - \phi_b^0\right)^{II} = b_b^{II} \underline{\underline{i}} : \underline{\underline{E}} + \frac{P_a}{N_{ba}^{II}} + \frac{P_b}{N_{bb}^{II}}$$
(9)

 k_{hom}^{II} , μ_{hom}^{II} overall drained bulk modulus & shear coefficient b_a^{II} , b_b^{II} Biot coefficients, N_{ij}^{II} solid Biot moduli

Average stress and strain

continuous description of stress field in the heterogeneous R.V.E. (Ulm et al. (2005), Dormieux et al. (2006))

$$\underline{\underline{\sigma}}(\underline{z}) = \mathbb{C}(\underline{z}) : \underline{\underline{\varepsilon}}(\underline{z}) + \underline{\underline{\sigma}}^p(\underline{z}) \quad , \quad \forall \underline{z} \quad \text{in } \Omega \tag{10}$$

$$\mathbb{C}(\underline{z}) = \mathbb{C}_o^I \quad \text{in } \Omega_o, \quad \mathbb{C}(\underline{z}) = \mathbb{C}_b = 0 \quad \text{in } \Omega_b, \quad \mathbb{C}(\underline{z}) = \mathbb{C}_c \quad \text{in } \Omega_b$$
(11)

and uniform prestress $\underline{\underline{\sigma}}^p(\underline{z})$ per phase

$$\underline{\underline{\sigma}}^{p}(\underline{z}) = \underline{\underline{\sigma}}^{p}_{o} = -b_{o}^{I}P_{a}\underline{\underline{i}} \quad \text{in } \Omega_{o}, \quad \underline{\underline{\sigma}}^{p}(\underline{z}) = \underline{\underline{\sigma}}^{p}_{b} = -P_{b}\underline{\underline{i}} \quad \text{in } \Omega_{b}$$

$$\underline{\underline{\sigma}}^{p}(\underline{z}) = 0 \quad \text{in } \Omega_{c}$$
(12)

Levin's theorem (see Levin (1967), Levin and Alvarez-Tostado (2006), Ulm et al. (2005)) used in linear microporoelasticity

Two subproblems : ' and "

1. first subproblem ': drained conditions (zero prestress field $\underline{\underline{\sigma}}^p = 0, P_a = P_b = 0$) loading parameter = macroscopic strain tensor $\underline{\underline{E}}$.

$$\underline{\underline{\xi}}'(\underline{z}) = \underline{\underline{E}} \cdot \underline{\underline{z}} \quad \text{on} \quad \partial \Omega$$

$$\underline{\underline{\sigma}}' = \mathbb{C}(\underline{z}) : \underline{\underline{\varepsilon}}' \qquad (13)$$

2. second subproblem " : zero-displacement boundary problem with loading defined by prestress field $\underline{\sigma}^p$

$$\underline{\underline{\xi}}''(\underline{z}) = 0 \quad \text{on } \partial\Omega$$

$$\underline{\underline{\sigma}}'' = \mathbb{C}(\underline{z}) : \underline{\underline{\varepsilon}}'' + \underline{\underline{\sigma}}^p(\underline{z})$$
(14)

Derivation of $\mathbb{C}_{hom}^{II} - b_i^{II}$: First subproblem '

first subproblem ' ($P_a = P_b = 0$), loading parameter : uniform strain tensor <u>E</u> imposed on the boundary of the *RVE*

$$\underline{\underline{\Sigma}}' = \left\langle \underline{\sigma}' \right\rangle = \mathbb{C}_{\text{hom}}^{II} : \underline{\underline{E}}$$

$$\left[\left(\phi_a - \phi_a^0 \right)^{II} \right]' = b_a^{II} \underline{\underline{i}} : \underline{\underline{E}}$$

$$\left[\left(\phi_b - \phi_b^0 \right)^{II} \right]' = b_b^{II} \underline{\underline{i}} : \underline{\underline{E}}$$
(15)

$$\begin{aligned} \mathbb{C}_{\mathsf{hom}}^{II} &= \mathbb{C}_{\mathsf{SC}} = \mathbb{C}^{0} = \left\langle \mathbb{C}_{r} : \left[\mathbb{I} + \mathbb{P}^{0} : \left(\mathbb{C}_{r} - \mathbb{C}^{0} \right) \right]^{-1} \right\rangle \\ \left\langle \underline{a} \right\rangle &= \frac{1}{\Omega} \int_{\Omega} \underline{\underline{a}} d\Omega \quad , \quad \mathbb{P}^{0} = \frac{\alpha^{0}}{3 \, k^{0}} \, \mathbb{J} + \frac{\beta^{0}}{2 \, \mu^{0}} \, \mathbb{K} \\ \alpha^{0} &= \frac{3 \, k^{0}}{3 \, k^{0} + 4 \, \mu^{0}} \quad , \quad \beta^{0} = \frac{6 \left(k^{0} + 2 \, \mu^{0} \right)}{5 \left(3 \, k^{0} + 4 \, \mu^{0} \right)} \end{aligned}$$
(16)

overall bulk modulus & shear coefficient

micro & macro isotropy, spherical distribution, ($k_{\rm hom}^{II}=k^0$, $\mu_{\rm hom}^{II}=\mu^0$)

$$\frac{k^0}{3\,k^0 + 4\,\mu^0} = f_o \frac{k_o^I}{3\,k_o^I + 4\,\mu^0} + (1 - f_o - f_b) \,\frac{k_c}{3\,k_c + 4\,\mu^0} \quad (17)$$

$$\frac{1}{5(3k^{0}+4\mu^{0})} = f_{o} \frac{\mu_{o}^{I}}{6\mu_{o}^{I}(k^{0}+2\mu^{0})+\mu^{0}(9k^{0}+8\mu^{0})} + (1-f_{o}-f_{b}) \frac{\mu_{o}^{I}(k^{0}+2\mu^{0})+\mu^{0}(9k^{0}+8\mu^{0})}{6\mu_{o}(k^{0}+2\mu^{0})+\mu^{0}(9k^{0}+8\mu^{0})}$$
(18)

 $k_o^I, \mu_o^I, k_c, \mu_c \leftrightarrow$ micro-indentation results (mesoscale) $k_{\text{hom}}^{II}, \mu_{\text{hom}}^{II} \leftrightarrow$ macro results (compression tests etc.)

overall Biot coefficients (1)

SC scheme, \mathbb{P}^0 tensor identical for all phases, strain localisation tensor (r = o, b, c)

$$A_{r} = \left[\mathbb{I} + \mathbb{P}^{0} : (\mathbb{C}_{r} - \mathbb{C}^{0}) \right]^{-1} \\ A_{r} = \frac{k^{0}}{k^{0} - \alpha^{0} (k^{0} - k_{r})} \mathbb{J} + \frac{\mu^{0}}{\mu^{0} - \beta^{0} (\mu^{0} - \mu_{r})} \mathbb{K}$$
(19)

then ($k_b = 0$, $\mu_b = 0$)

$$A_{o} = \frac{k^{0}}{k^{0} - \alpha^{0} (k^{0} - k_{o}^{I})} \mathbb{J} + \frac{\mu^{0}}{\mu^{0} - \beta^{0} (\mu^{0} - \mu_{o}^{I})} \mathbb{K}$$

$$A_{b} = \frac{1}{1 - \alpha^{0}} \mathbb{J} + \frac{1}{1 - \beta^{0}} \mathbb{K}$$

$$A_{c} = \frac{k^{0}}{k^{0} - \alpha^{0} (k^{0} - k_{c})} \mathbb{J} + \frac{\mu^{0}}{\mu^{0} - \beta^{0} (\mu^{0} - \mu_{c})} \mathbb{K}$$
(20)

overall Biot coefficients (2)

average strain tensor per phase

$$\left\langle \underline{\varepsilon}' \right\rangle^r = \underline{\varepsilon}'_r = \mathbb{A}_r : \underline{\underline{E}}$$
 (21)

change of macroporosity

$$\left[\left(\phi_b - \phi_b^0 \right)^{II} \right]' = f_b \underline{\underline{i}} : \left\langle \underline{\underline{\varepsilon}}' \right\rangle^b = f_b \underline{\underline{i}} : \mathbb{A}_b : \underline{\underline{E}} = b_b^{II} \underline{\underline{i}} : \underline{\underline{E}}$$
(22)

$$\begin{bmatrix} \left(\phi_a - \phi_a^0\right)^{II} \end{bmatrix}' = f_o \left\langle \left(f_a - f_a^0\right)' \right\rangle^o$$

$$= f_o b_o^I \underline{i} : \left\langle \underline{\varepsilon}' \right\rangle^o = f_o b_o^I \underline{i} : \mathbb{A}_o : \underline{\underline{E}} = b_a^{II} \underline{i} : \underline{\underline{E}}$$

$$(23)$$

$$\langle \underline{\underline{a}} \rangle = \frac{1}{\Omega} \int_{\Omega} \underline{\underline{a}} d\Omega \quad , \quad \langle \underline{\underline{a}} \rangle^{\alpha} = \frac{1}{\Omega^{\alpha}} \int_{\Omega^{\alpha}} \underline{\underline{a}} d\Omega$$
 (24)

overall Biot coefficients (3)

Biot coefficient associated to microporosity (intra-oolite)

$$b_a^{II}\underline{\underline{i}} = f_o b_o^I\underline{\underline{i}} : \mathbb{A}_o \tag{25}$$

$$b_a^{II} = \frac{f_o k^0 b_o^I}{k^0 - \alpha^0 \left(k^0 - k_o^I\right)}$$
(26)

Biot coefficient associated to mesoporosity (inter-oolite)

$$b_b^{II}\underline{\underline{i}} = f_b\underline{\underline{i}} : \mathbb{A}_b \tag{27}$$

$$b_b^{II} = \frac{f_b}{1 - \alpha^0} \tag{28}$$

connected porosities & $P_a = P_b = P$

overall Biot coefficient with equal pressures in *micro* & *meso* porosities

$$b^{II} = b_a^{II} + b_b^{II} \tag{29}$$

$$b^{II} = \frac{f_o k^0 b_o^I}{k^0 - \alpha^0 \left(k^0 - k_o^I\right)} + \frac{f_b}{1 - \alpha^0}$$
(30)

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data at mesoscale : microindentation

mesoscopic bulk moduli of Lavoux limestone constituents deduced from indentation data (microindentation, current work : nanoindentation, **C. Auvray**)

constituent	volume fraction	n_{mes}	k_i (GPa)	σ (GPa)	
oolite	$f_o = 0.74$	29	27.1	5.4	k_o^I
sparite grain	$f_c = 0.14$	34	68.2	6.1	k_c
meso pores	$f_b = 0.12$				$k_b = 0$

 k_i : mean value, σ : standard deviation Microindentation: Flat indenter ($D = 300 \mu m$) High-resolution camera ($25 \mu m$) fixed on indenter frame used to identify oolite and sparitic cement

reference data solid phase : pure calcite

solid phase \approx mono-mineral, $f_{calcite}^s \approx 0.98$. pure calcite mineral : trigonal anisotropic class system (Winkler (1975)) Elastic constant of calcite (GPa)

Constituent	C_{1111}	C_{3333}	C_{2323}	C_{1122}	C_{1133}	C_{1123}
Calcite ($CaCO_3$)	144.	84.0	33.5	53.9	51.1	-20.5

random distribution \Rightarrow isotropization

$$\mathbb{C}_{ca}^{\text{is}} = 3k_{ca}^{\text{is}} \mathbb{J} + 2\mu_{ca}^{\text{is}} \mathbb{K}$$
$$k_{ca}^{\text{is}} = \frac{\mathbb{C}_{ca} :: \mathbb{J}}{3} \approx 76.0 \quad \text{GPa}$$
$$\mu_{ca}^{\text{is}} = \frac{\mathbb{C}_{ca} :: \mathbb{K}}{10} \approx 36.8 \quad \text{GPa}$$

bulk moduli at mesoscale (experimental data)



mean values of sparitic cement *c* & oolite *o* compared to pure calcite : $k_c/k_{ca} \approx 0.90$ & $k_o^I/k_{ca} \approx 0.36$

model-experiment: mesocale

two unknown coefficients identified at mesoscale, $\chi_o \& \chi_c$ (solid phase in o & solid phase c) sparitic cement c: homogeneous isotropic solid phase

$$k_c = \chi_c k_{ca}$$
 , $\mu_c = \chi_c \mu_{ca}$, $\chi_c = 0.9$ (31)

porous oolite *o* : self consistent scheme, with micritic calcite grains

$$k_o^s = \chi_o k_{ca}$$
 , $\mu_o^s = \chi_o \mu_{ca}$, $\chi_o = 0.58$ (32)

effective bulk modulus at mesoscale

$$k_o^I = 23.9 \text{ GPa}$$
 , experiment : $k_o^I = 27.1 \text{ GPa}$ (33)

Table 1: Lavoux limestone

	k_s^{II} (GPa)	k_{hom}^{II} (GPa)	μ_{hom}^{II} (GPa)	b_{hom}^{II}
Ехр	47.1	18-22	10-12	0.83
Model	47.2	21.1	12.1	0.57

Effective bulk modulus of the solid phase = unjacketed compressibility K'_s

$$\underline{\underline{\Sigma}} = -P\underline{\underline{i}} \quad , \quad \underline{\underline{\sigma}}^p = \underline{\underline{\sigma}}^p(P) \tag{34}$$

$$k'_{(s)\text{hom}}^{II} = \frac{Tr(\underline{\underline{\Sigma}})}{3Tr(\underline{\underline{E}})} = \frac{k_{\text{hom}}^{II}}{1 - b_{\text{hom}}^{II}}$$
(35)

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 2-Step SC homogenization model for a specific microstructure simple homogenization scheme useful as first approach correct order of magnitude for meso-macro properties degraded *ITZ* (Interfacial Transition zone) around oolites (lower macroproperties)

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 2-Step SC homogenization model for a specific microstructure simple homogenization scheme useful as first approach correct order of magnitude for meso-macro properties degraded *ITZ* (Interfacial Transition zone) around oolites (lower macroproperties)

prospects

characterization at lower scale : nanoindentation better description of inter oolitic material & flat mesopores concave shape imperfect interfaces

Model 2 : ITZ + Three steps

Model 2 : oolite + ITZ (oi) – macro pore (b) - sparitic cement (c)

step III : self consistent model with three constituents : **porous oolite + ITZ** – inter oolite pores – sparitic cement

step I : homogenization intra oolitic pores & ITZ pores (SC scheme)

step II : homogenization oolite (o) & ITZ (i) Three Phase model (GSCS)



Step I : micropores in oolite ITZ : Self Consistent (S

step I : homogenization of micropores in oolite (o) and ITZ (i)



Step II : oolite & ITZ = oi (GSCS)

step II : three phase model (GSCS) homogenization of porous oolite (o) and porous ITZ (i)



Step III : macro level, 3 distinct phases (SC)

Step III : self consistent scheme

homogenized porous oolite + ITZ (oi)

macro pores (b)

sparitic cement (c)

Simplifying assumptions : isotropic distribution & spherical shape for all constituents



better description of inter oolitic material

polycrystalline cement paste



Pichler and Hellmich (2010)

porous granular materials - imperfect interfaces

He et al. (2013)



Hashin and Monteiro (2002)

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