



**HAL**  
open science

## Multiscale approach to characterize effective mechanical, hydraulic and acoustic properties of a new bio-based porous material

Q.-B. Nguyen, V.-H. Nguyen, C. Perrot, A. Rios de Anda, E. Renard, S. Naili

### ► To cite this version:

Q.-B. Nguyen, V.-H. Nguyen, C. Perrot, A. Rios de Anda, E. Renard, et al.. Multiscale approach to characterize effective mechanical, hydraulic and acoustic properties of a new bio-based porous material. *Materials Today Communications*, 2021, 26, pp.101938. 10.1016/j.mtcomm.2020.101938 . hal-03252293

**HAL Id: hal-03252293**

**<https://hal.science/hal-03252293>**

Submitted on 7 Jun 2021

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

# Multiscale approach to characterize effective mechanical, hydraulic and acoustic properties of a new bio-based porous material

Q.-B. Nguyen<sup>a,b,c</sup>, V.-H. Nguyen<sup>a,b</sup>, C. Perrot<sup>d</sup>, A. Rios de Anda<sup>c</sup>, E. Renard<sup>c</sup>, S. Naili<sup>a,b,\*</sup>

<sup>a</sup>Univ Paris Est Creteil, CNRS, MSME, F-94010 Creteil, France

<sup>b</sup>Univ Gustave Eiffel, MSME, F-77447 Marne-la-Vallée, France

<sup>c</sup>Univ Paris Est Creteil, CNRS, ICMPE, F-94010 Creteil, France

<sup>d</sup>MSME, Univ Gustave Eiffel, UPEC, CNRS, F-77454, Marne-la-Vallée, France

---

## Abstract

1 This paper is a multi-aspect study which has undertaken essential numerical characteriza-  
2 tions of not only mechanical and hydraulic properties but also acoustic behaviour of a new  
3 bio-based porous epoxy resin obtained by a “green” adapted combination of the cationic  
4 photopolymerization and the porogen leaching technique. This new kind of material gener-  
5 ally possesses interconnected fillet-edge cubic pores which lead to more complex morphology  
6 than in the case of spherical or cylindrical pores. In order to characterize the effective prop-  
7 erties of the material, a multiscale approach using the asymptotic homogenization method  
8 has been applied. Such a method has induced cell problems whose the resolutions have  
9 been conducted on the geometrical configuration defined from the experimental data of the  
10 samples by using Scanning Electron Microscopy (SEM) and Mercury Intrusion Porosime-  
11 try (MIP). The sound absorption behaviour of a plate made of the studied material has  
12 been subsequently characterized by solving a normal incidence acoustic problem with an  
13 assumption of rigid impervious backing. The mechanical model has been validated by bal-  
14 ancing numerical results of the sound absorption coefficient, reflection coefficient, surface  
15 impedance, equivalent dynamic density and equivalent dynamic bulk modulus with cor-  
16 responding experimental results obtained by conducting the three-microphone impedance  
17 tube testing. As a consequence, the mechanical model has been applied to investigate the  
18 influence of microstructural characteristics on effective properties and acoustic performance  
19 of the material. To the authors’ knowledge, the features of the microstructure obtained from  
20 an elaboration process using the porogen leaching technique have rarely been studied in the  
21 literature. Four types of ordered pore arrangements together with systematic variations of  
22 the porosity and the pore size have been taken under consideration. Based on the results  
23 of these investigations, the subtle relation between microstructures and properties has been  
24 established. The processing parameters of material elaboration could be adjusted so that  
25 the obtained porous material would possess the best sound absorption performance.

*Keywords:* asymptotic homogenization, effective properties, sound absorption, finite element method, microstructures, bio-based porous epoxy resin, photopolymerization, porogen leaching technique

---

26 **1. Introduction**

27 Porous materials are used in a large number of industries such as automobile, aeronau-  
28 tic and construction due to their light weight, low production cost and flexible mechanical  
29 properties. Most of porous materials have petroleum-based polymeric matrices that come  
30 from processes using solvents and amine-type hardeners which are highly toxic to the envi-  
31 ronment. As an attempt to reduce the consumption of petroleum-based materials, as well  
32 as to avoid harmful substances, a novel porous bio-based epoxy resin has been developed  
33 by using cationic photopolymerization in conjunction with the porogen leaching technique  
34 adapted from [1] and [2]. Unlike the other methods in fabrication of porous materials such  
35 as temperature-induced phase separation [3], high internal phase emulsions [4] or gas expan-  
36 sion [5], the porogen leaching technique consists in adding porogen particles to a monomer  
37 solution, then in removing the porogen with a corresponding solvent/diluent after the poly-  
38 merization, thereby obtaining a porous scaffold. This technique includes easy operational  
39 steps and permits accurate control of pore size and porosity [6, 7]. It is also relevant to  
40 the cationic photopolymerization because this kind of polymerization is relatively solvent-  
41 sensitive. Therefore, the presence of a solvent during the polymerization step is not required  
42 [8].

43 The determination of the effective mechanical, hydraulic and acoustic characteristics of  
44 these materials is essential, in particular, during the elaboration phase of the material. In  
45 order to characterize the effective properties of these materials, experimental methods could  
46 be used to determine, for instance: (i) the effective elastic modulus *via* uniaxial compres-  
47 sion tests; (ii) the effective static permeability *via* the application of Darcy’s law under  
48 steady state conditions; (iii) acoustic properties *via* impedance tube experiments associated  
49 with the transfer function (or transfer matrix methods). Most of these methods require  
50 specific facilities and huge effort in conducting experiments that are usually related to com-  
51 plicated characterization techniques. Particularly, it remains challenging for measurements  
52 of frequency-dependent properties such as dynamic permeability or dynamic compressibility.  
53 In addition, only a few methods establish a useful relation between material microstructures  
54 and effective properties.

55 Among the methods for modelling porous materials, the average-field theory and the  
56 asymptotic homogenization are the most convenient and commonly accepted ones, since  
57 they enable the estimation of all mechanical properties of a poroelastic medium. One of the  
58 major differences between them is the choice of the representative volume element (RVE), or  
59 a “representative cell” for performing calculations related to each of these techniques: while  
60 the RVE of a statistically homogeneous body is employed in the average-field theory, the  
61 asymptotic homogenization theory is based on the representative (unit) periodic cell. The

---

\*Corresponding author  
Email address: naili@u-pec.fr (S. Naili)

62 homogenization theory starts at the microscopic scale with the detailed microstructure of the  
63 pores, the linearized equations of elasticity and the linearized equations of fluid dynamics  
64 (see for instance [9] for a mechanical point of view and [10] for a mathematical point of  
65 view). As a consequence, the effective properties in the conservation equations (continuity  
66 and momentum equations) and in the constitutive relations at the macroscopic scale are  
67 obtained according to the geometrical and mechanical properties of the microstructure.  
68 This multiscale approach based on the asymptotic homogenization theory has been used  
69 with success in several recent papers to characterize new materials [11, 12].

70 It is well-known that the effective properties and acoustic performance of porous materials  
71 strongly depend on the mechanical properties of the solid phase, the pore size, the porosity  
72 and the pore arrangement. Published research works in the open literature have mainly  
73 focused on the microstructures with idealized geometries such as spherical, cylindrical, cross-  
74 type or sometimes polyhedral pores [13, 14]. To the best of our knowledge, few studies have  
75 been carried out on the influence of microstructural parameters related to the case of fillet-  
76 edge cubic pores which mimic the pores of the above-mentioned novel porous bio-based  
77 material and result in more complex morphology than the ones cited above. Furthermore,  
78 these studies focused on only either effective mechanical properties or acoustic characteristics  
79 of materials and none of them simultaneously investigated both aspects.

80 The main objective of this study is to perform numerical characterizations of effective  
81 mechanical, hydraulic and acoustic properties of the novel bio-inspired porous epoxy resin,  
82 which is synthesized through a relevant combination of the cationic photopolymerization  
83 and the porogen leaching technique. The obtained results may allow us to make essential  
84 suggestions for improving the elaborated material. Although the above-mentioned works are  
85 related to the same topic, the present paper contributes to the modelling of the porous media  
86 in several aspects. Indeed, the paper supplies original understandings of the influence of the  
87 microstructural features, namely the pore arrangement, the porosity, and finally, the pore  
88 size on the effective properties and acoustic performance of the porous material possessing  
89 interconnected pores with the specific fillet-edge cubic shape. To the authors' knowledge,  
90 the features of this microstructure which is obtained from an elaboration process using the  
91 porogen leaching technique have been rarely studied in the literature. By considering simul-  
92 taneously the effective mechanical properties, the effective permeability and compressibility  
93 as well as the acoustic behaviour of the material, this multi-aspect study provides a relevant  
94 insight into the microstructures-properties relation, creating the foundations of a potential  
95 development for studied porous epoxy resins in a wide range of applications.

96 In order to characterize the elaborated porous material, a multiscale approach based on  
97 the asymptotic homogenization has been used. The resolution of the asymptotic homoge-  
98 nization problems, called the cell problems, has been carried out by considering idealized  
99 cell structures obtained from the imaging of sample microstructures which allows to deter-  
100 mine, in particular, the geometrical properties of microstructures. Using homogenization  
101 results, the sound absorption problem of a porous plate made of the studied material has  
102 been studied. The validation of the mechanical model has been confirmed by comparing  
103 the numerical results of the effective acoustic properties and the measurements obtained  
104 from the three-microphone impedance tube testing. The multiscale approach has been then

105 used to investigate the effect of microstructural characteristics on the effective properties  
 106 and the acoustic behaviour of the material. For this purpose, four different ordered pore  
 107 arrangements as well as systematic variations of the porosity and the pore size have been  
 108 taken into account.

109 The paper is organized as follows. The elaboration and experimental pore characteriza-  
 110 tion of the porous material is introduced in Section 2. We present in Section 3 the principles  
 111 and main formulations of the asymptotic homogenization procedure. The construction of  
 112 idealized representative volume elements is presented in Section 4. The description of the  
 113 normal incidence sound absorption problem in case of a structural poroelastic plate is pro-  
 114 vided in Section 5. The numerical results and a discussion are proposed in the Section 6. In  
 115 this section, an experimental validation of the mechanical model, as well as the influence of  
 116 microstructural characteristics on the effective properties and sound absorption performance  
 117 of the material are discussed. Section 7 completes the paper with main conclusions.

## 118 2. Material elaboration and characterization

### 119 2.1. Elaboration process using porogen leaching technique

120 The porous polymeric material was elaborated using the cationic photopolymerization  
 121 in conjunction with the porogen leaching technique, which involved the presence of chloride  
 122 sodium (NaCl) templates. The key idea of introducing NaCl particles in the process is  
 123 that the removal of these particles after the polymerization will leave the empty space,  
 124 creating porosity in the material (see Fig. 1). NaCl templates with a cylindrical shape of  
 125 circular cross-section, whose the diameter and height are respectively equal to 5.1 cm and  
 126 0.5 cm, were produced by the Spark Plasma Sintering (SPS) technique using NaCl cubic  
 127 particles whose the size varied between 250 and 400  $\mu\text{m}$ . This sintering technique induces  
 128 simultaneously a variation of heat and pressure on NaCl particles, carrying out the contacts  
 129 among NaCl particles, thereby ensuring the pore interconnection of the obtained porous  
 130 material [15]. From NaCl mass and the dimensions of the templates, the NaCl volume  
 131 fraction is about 82%.

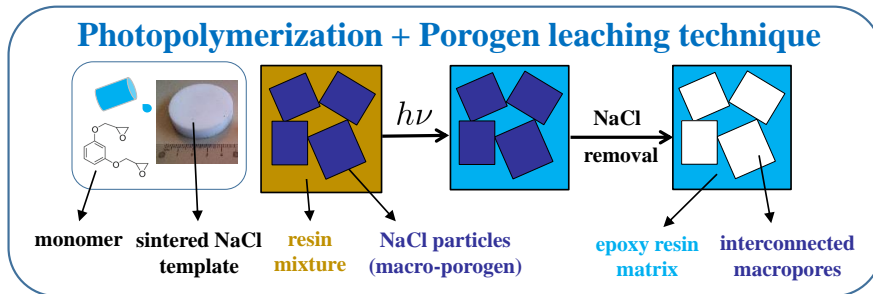


Figure 1: Schema of porogen leaching technique using NaCl particles as macro-porogen.

132 The resin was composed of resorcinol diglycidyl ether as monomer, bis (dodecylphenyl)  
 133 iodonium hexafluoro-antimonate (herein called Iod) as photoinitiator and benzophenone as  
 134 photosensitizer. The masses of Iod and benzophenone were 5% and 1% of the monomer mass,

135 respectively. After being melted at a temperature of 120°C, the liquid-state resin mixture was  
136 introduced into the NaCl template which was isolated under vacuum beforehand. According  
137 to our rheological study, the considered mixture reaches its lowest dynamic viscosity at a  
138 temperature greater than or equal to 120°C. The combination of melting the resin mixture at  
139 this temperature and the vacuum-state of the template helps the mixture deeply penetrate  
140 into the template and fill up the space between NaCl particles. The new mixture (resin  
141 mixture - NaCl template) was then isolated under vacuum for 30 min to ensure the resin's  
142 penetration. After that, this new mixture was irradiated by a polychromatic spot with  
143 a UV wavelength from 250 to 450 nm and an intensity of 220 W/m<sup>2</sup>. The irradiation  
144 was carried out on each circular face of the template for 30 min. Subsequently, the new  
145 mixture was placed in an oven at 120°C for 1.5 h to finish the polymerization. Finally,  
146 NaCl particles were removed by immersing this mixture in water for 72 h. The water was  
147 changed every 12 h. The wet sample was then placed under vacuum for 24 h to obtain  
148 the final dry porous sample. The complete NaCl removal was verified by the gravimetric  
149 method. Note that the interconnected porous network after the removal of sintered NaCl  
150 particles has been confirmed by SEM images. Besides, the fusion of NaCl cubic particles has  
151 many contact/fusion modes, for instance, "face to face", "side to side" and "point to point"  
152 modes, but the "face to face" mode has been more frequently found in SEM micrographs.  
153 This contact mode has resulted in the large entrance towards a pore as determined by MIP  
154 measurements.

155 With the proposed technique, the porosity can be adjusted to obtain materials with  
156 larger or smaller porosities. Indeed, the porosity of the resulting material can easily be  
157 altered by adjusting the pressure and the temperature during template preparation by the  
158 SPS process. Raising the pressure and the temperature during the SPS process induces a  
159 sintered porogen template with more porogen constituents in a volume, which means a final  
160 material with more porosity. On the contrary, the process with lower pressure and lower  
161 temperature leads to a material with a smaller porosity.

## 162 2.2. Experimental pore characterization

163 Pore features of the obtained porous material was then characterized by Mercury In-  
164 trusion Porosimetry (MIP) and Scanning Electron Microscopy (SEM). According to MIP  
165 results, the distribution of pore size is centered at about 180  $\mu\text{m}$ . This value is smaller than  
166 the size of NaCl particles used in the fabrication of templates (*i.e.*, 250-400  $\mu\text{m}$ ) due to the  
167 fact that during the tests by MIP, at each level of mercury pressure, one measures the largest  
168 entrance towards a pore, but not the actual inner size of a pore [16]. The dimension of this  
169 entrance is smaller than the inner pore size which is similar to particle size. The analysis of  
170 SEM micrographs (see Fig. 2) gives the average value of entrance dimension which is about  
171 192  $\mu\text{m}$ , showing a good agreement with the profile obtained by the MIP.

172 The tests of MIP also provided a porosity value of about 43%. It is noted that this value  
173 is much smaller than the NaCl volume fraction of 82%, which would be the value of porosity  
174 in case of perfect penetration of the resin mixture into the template. This is explained by  
175 the fact that the use of NaCl particles, whose the size varies between 250 and 400  $\mu\text{m}$ , lead  
176 to the existence of pore sizes that were beyond the pore size range (0.003-360  $\mu\text{m}$ ) that

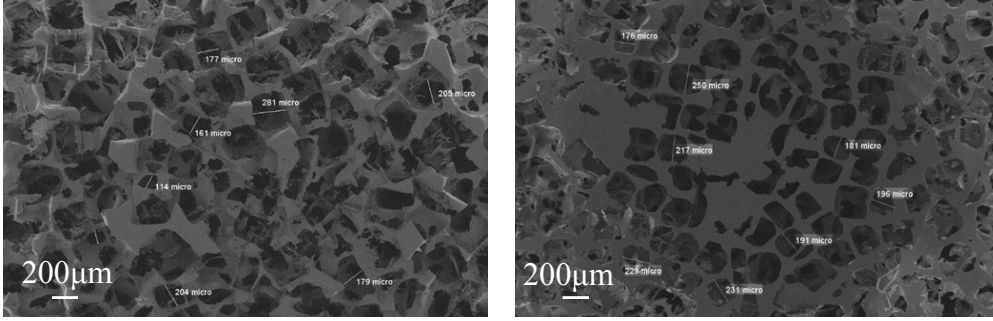


Figure 2: SEM micrographs: (left) on cross-section; (right) on external surface

177 the experimental setup can afford. The porosity was also determined by another approach.  
 178 By taking a piece of the porous material, weighing and measuring its dimensions to obtain  
 179 its mass  $m$  and volume  $V$ , the volumetric density of the porous material is determined as  
 180  $\rho = m/V$ . Based on the density of the matrix phase  $\rho^s$  which was identified when working  
 181 with the bulk material, we can find the porosity by the relation  $\phi = 1 - \rho/\rho^s$ . This approach  
 182 gave a porosity of approximately 83% which correlates with the NaCl volume fraction.  
 183 The fact that this value is slightly greater than the NaCl volume fraction is explained by  
 184 considering that the resin mixture did not perfectly penetrate and did not fill in all of the  
 185 spaces among NaCl particles. Therefore, the value of 83% was considered as the porosity of  
 186 the obtained porous material.

### 187 3. Homogenization and effective properties

188 In what follows, the Cartesian reference system is used in which the orthonormal basis  
 189 is denoted by  $(\mathbf{e}_1, \mathbf{e}_2, \mathbf{e}_3)$ . The coordinates of a point are specified by  $(x_1, x_2, x_3)$ . The  
 190 time coordinate is denoted by  $t$ . The time derivative is denoted by a superimposed dot.  
 191 Einstein's summation convention about the repeated indices is used. The imaginary pure  
 192 number denoted by  $i$  is defined by  $i^2 = -1$ . All fields are written in the frequency domain,  
 193 considering a harmonic regime with functions proportional to  $\exp(i\omega t)$  where  $\omega$  is the angular  
 194 frequency.

#### 195 3.1. Two-scale asymptotic homogenization procedure

196 The details of the two-scale asymptotic homogenization procedure, the derivation of cell  
 197 problems and macroscopic state equations can be found, for instance, in [17] and [18]. The  
 198 following parts present the principle and the main results obtained by this method.

We consider a deformable porous medium saturated by a fluid. It is assumed that the constitutive relations are the one of a linearly elastic solid for the skeleton and the one of a Newtonian compressible fluid whose dynamic viscosity is  $\eta$  for the fluid phase. The domain of a representative volume element is denoted by  $Y \subset \mathbb{R}^3$  at the microscopic scale and is decomposed into the matrix (the skeleton which composes the solid phase) and the pore subdomains. These subdomains are respectively denoted by  $Y^s$  and  $Y^f$ , and their common

boundary is designated by  $\Gamma^{sf}$  (see Fig. 3):

$$Y = Y^s \cup Y^f \cup \Gamma^{sf}, \quad Y^s \cap Y^f = \emptyset, \quad \Gamma^{sf} = \partial Y^s \cap \partial Y^f. \quad (1)$$

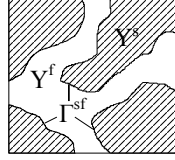


Figure 3: Sketch of the studied microscopic domains.

199 The pores of the medium are assumed to be interconnected throughout the whole domain.

200 *3.1.1. Microscopic governing equations*

In the solid phase, the momentum and constitutive equations read:

$$\frac{\partial \sigma_{ij}^s}{\partial x_j} = -\rho^s \omega^2 u_i^s \quad \text{within } Y^s, \quad (2a)$$

$$\sigma_{ij}^s = c_{ijkl}^s \varepsilon_{kl}^s, \quad (2b)$$

where  $\rho^s$ ,  $u_i^s$  and  $\sigma_{ij}^s$  are respectively the density, the components of the displacement vector and stress tensor. The quantities  $c_{ijkl}^s$  are the components of the fourth-order elasticity tensor. The components  $\varepsilon_{kl}^s$  are the components of the infinitesimal strain which read:

$$\varepsilon_{kl}^s = \frac{1}{2} \left( \frac{\partial u_k^s}{\partial x_l} + \frac{\partial u_l^s}{\partial x_k} \right) \quad (3)$$

In the fluid phase, the governing equations are the law of mass conservation and the state equation of gas:

$$\rho^f \frac{\partial v_i^f}{\partial x_i} + i\omega \delta^f = 0 \quad \text{within } Y^f, \quad (4a)$$

$$\frac{\delta^f}{\rho^f} + \frac{\tau^f}{T^f} = \frac{p^f}{P^f} \quad \text{within } Y^f, \quad (4b)$$

201 where  $p^f$  and  $v_i^f$  are respectively the pressure and the components of the velocity of the fluid  
 202 phase;  $P^f$ ,  $\rho^f$  and  $T^f$  are respectively the pressure, the density and the temperature of the  
 203 fluid phase in the rest state;  $\delta^f$  and  $\tau^f$  are respectively the perturbations of the density and  
 204 the temperature.

Due to the fact that infinitesimal harmonic motions are supposed to be in the fluid phase, the Navier-Stokes equation for the compressible viscous fluid can be linearized and written as:

$$\frac{\partial \sigma_{ij}^f}{\partial x_j} = \rho^f i\omega v_i^f \quad \text{within } Y^f, \quad (5)$$



where the constitutive equation of compressible viscous fluid and the infinitesimal strain rate are given by:

$$\sigma_{ij}^f = 2\eta\dot{\epsilon}_{ij}^f - \frac{2}{3}\eta\dot{\epsilon}_{kk}^f\delta_{ij} - p^f\delta_{ij}, \quad (6a)$$

$$\dot{\epsilon}_{ij}^f = \frac{1}{2}\left(\frac{\partial v_i^f}{\partial x_j} + \frac{\partial v_j^f}{\partial x_i}\right), \quad (6b)$$

205 where  $\delta_{ij}$  is the Kronecker symbol.

As regards the thermal field, the solid phase can maintain isothermal conditions when the specific heat capacity of the solid phase is much larger than that of the fluid phase. By denoting  $q_i^f$  and  $\kappa_{ij}^f$  respectively the components of the thermal flux and thermal conductivity from the energy conservation law associated with the specific heat under constant volume and the universal gas constant denoted respectively by  $C_v^f$  and  $R_g$ , the set of governing equations for the thermal field in the fluid phase is expressed as:

$$-\frac{\partial q_i^f}{\partial x_i} = i\omega\rho^f C_V^f \tau^f + (i\omega\rho^f R_g \tau^f - i\omega\rho^f) \quad \text{within } Y^f, \quad (7a)$$

$$q_i^f = -\kappa_{ij}^f \frac{\partial \tau^f}{\partial x_j} \quad \text{within } Y^f. \quad (7b)$$

206 For an ideal gas, Mayer's relation is valid and is given by the relation  $C_P^f - C_V^f = R_g$ ,  
 207 where  $C_P^f$  is the specific heat under constant pressure. It is also convenient to introduce the  
 208 specific heat ratio of the fluid  $\gamma^f = C_P^f/C_V^f$  which will be used in the next sections.

The continuity conditions at the common boundary  $\Gamma^{sf}$  of the solid and fluid phases for the normal stress, the velocity and the temperature are given as follows:

$$\sigma_{ij}^s n_j^s + \sigma_{ij}^f n_j^f = 0 \quad \text{over } \Gamma^{sf}, \quad (8a)$$

$$i\omega u_i^s = v_i^f \quad \text{over } \Gamma^{sf}, \quad (8b)$$

$$\tau^f = 0 \quad \text{over } \Gamma^{sf}, \quad (8c)$$

209 where  $n_j^s$  and  $n_j^f$  are the components of outward normal unit vectors to the boundary  $\Gamma^{sf}$ ,  
 210 corresponding to the solid and fluid phases, respectively.

### 211 3.1.2. Scaling and asymptotic expansions

212 A poroelastic medium that has a geometrically periodic microstructure is considered.  
 213 The unit length of the periodicity is denoted by  $l$  and the macroscopic characteristic length  
 214 by  $L$ . It is assumed that the scale ratio  $\epsilon = l/L$  is much smaller than 1, that is, the two  
 215 scales  $l$  and  $L$  differ significantly from each other. The position vector at the macroscopic  
 216 scale is denoted by  $\mathbf{x}$  (whose components are given by  $x_i$ ), and the position vector at the  
 217 microscopic scale by  $\mathbf{y} = \mathbf{x}/\epsilon$  (the components are  $y_i = x_i/\epsilon$ ).

Sound absorption is generally attributed to viscous damping and thermal dissipation which occur in the vicinity of the solid-fluid interface. Furthermore, in the air, the heat conductivity is isotropic and is defined by a tensor whose components are  $\kappa_{ij}^f = \kappa_0^f \delta_{ij}$  where  $\kappa_0^f$  is the characteristic thermal conductivity of the air. So, the thicknesses of the region where these energy exchanges take place are respectively called the viscous skin depth  $\delta_v = (2\eta/(\rho^f \omega))^{1/2}$  and the thermal skin depth  $\delta_t = (\kappa_0^f/(\rho^f \omega C_P^f))^{1/2}$ . When a poroelastic material that is saturated with the air is capable of absorbing sounds, both thicknesses are on the same order as that of the pore scale [18], leading to the relation  $O(\delta_{v,t}^2/L^2) = O(\epsilon^2)$  where  $O(*)$  is big-oh notation, which is used to describe the asymptotic behaviour of the function. Consequently, equations (6) and (7b) can respectively be rewritten as:

$$\sigma_{ij}^f = 2\epsilon^2 \eta \dot{\epsilon}_{ij}^f - \frac{2}{3} \epsilon^2 \eta \dot{\epsilon}_{kk}^f \delta_{ij} - p^f \delta_{ij}, \quad (9)$$

$$q_i^f = -\epsilon^2 \kappa_{ij}^f \frac{\partial \tau^f}{\partial x_j}. \quad (10)$$

We assume a solution in the asymptotically expanded form for physical variables  $u_i^s$ ,  $v_i^f$ ,  $p^f$ ,  $\tau^f$  and  $\delta^f$  [19]:

$$\begin{aligned} u_i^s &= u_i^{s(0)}(\mathbf{x}, \mathbf{y}) + \epsilon u_i^{s(1)}(\mathbf{x}, \mathbf{y}) + \epsilon^2 u_i^{s(2)}(\mathbf{x}, \mathbf{y}) + \dots, \\ v_i^f &= v_i^{f(0)}(\mathbf{x}, \mathbf{y}) + \epsilon v_i^{f(1)}(\mathbf{x}, \mathbf{y}) + \epsilon^2 v_i^{f(2)}(\mathbf{x}, \mathbf{y}) + \dots, \\ p^f &= p^{f(0)}(\mathbf{x}, \mathbf{y}) + \epsilon p^{f(1)}(\mathbf{x}, \mathbf{y}) + \epsilon^2 p^{f(2)}(\mathbf{x}, \mathbf{y}) + \dots, \\ \tau^f &= \tau^{f(0)}(\mathbf{x}, \mathbf{y}) + \epsilon \tau^{f(1)}(\mathbf{x}, \mathbf{y}) + \epsilon^2 \tau^{f(2)}(\mathbf{x}, \mathbf{y}) + \dots, \\ \delta^f &= \delta^{f(0)}(\mathbf{x}, \mathbf{y}) + \epsilon \delta^{f(1)}(\mathbf{x}, \mathbf{y}) + \epsilon^2 \delta^{f(2)}(\mathbf{x}, \mathbf{y}) + \dots, \end{aligned} \quad (11)$$

218 where all the functions on the right-hand side of Eqs.(11) are Y-periodic, *i.e.*, periodic in  
 219 terms of  $\mathbf{y}$  with the period of a representative volume element  $Y$ , and the superscripts ( $n$ )  
 220 represent explicitly the terms of the order of  $\epsilon^n$ . All of these expansions are then substituted  
 221 into Eqs. (2)-(10).

### 222 3.1.3. Cell problems

223 By plugging the asymptotic expansions of Eq. (11) in Eqs.(2)-(10), one can deduce  
 224 four cell problems which will be used for the determination of effective properties of the  
 225 poroelastic medium. By introducing the functional spaces  $\mathcal{C}_\#(Y^{s,f})$  of Y-periodic functions  
 226 which are sufficiently differential on  $Y^{s,f}$ , cell problems can be written in their weak forms  
 227 as follows:

**Cell problem 1:** Find the function  $\chi_p^{kl}(\mathbf{y}) \in \mathcal{C}_\#(Y^s)$  such as:

$$\int_{Y^s} \left( c_{ijkl}^s - c_{ijpq}^s \frac{\partial \chi_p^{kl}(\mathbf{y})}{\partial y_q} \right) \frac{\partial \delta \chi_i^{kl}(\mathbf{y})}{\partial y_j} dV = 0, \quad \forall \delta \chi_i^{kl}(\mathbf{y}) \in \mathcal{C}_\#(Y^s), \quad (12a)$$

$$\int_{Y^s} \chi_p^{kl}(\mathbf{y}) dV = 0. \quad (12b)$$

**Cell problem 2:** Find  $\beta_k(\mathbf{y}) \in \mathcal{C}_\#(Y^s)$  such as:

$$\int_{Y^s} c_{ijkl}^s \frac{\partial \beta_k(\mathbf{y})}{\partial y_l} \frac{\partial \delta \beta_i(\mathbf{y})}{\partial y_j} dV = \int_{\Gamma^{sf}} \delta_{ij} n_j^s \delta \beta_i(\mathbf{y}) d\Gamma, \quad \forall \delta \beta_i(\mathbf{y}) \in \mathcal{C}_\#(Y^s), \quad (13a)$$

$$\int_{Y^s} \beta_k(\mathbf{y}) dV = 0. \quad (13b)$$

**Cell problem 3:** Find the functions  $\xi_i^k(\mathbf{y})$  and  $\pi^k(\mathbf{y}) \in \mathcal{C}_\#(Y^f)$  such as:

$$\begin{aligned} \int_{Y^f} \rho^f i\omega \xi_i^k(\mathbf{y}) \delta \xi_i^k(\mathbf{y}) dV + \int_{Y^f} \eta \frac{\partial \xi_i^k(\mathbf{y})}{\partial y_j} \frac{\partial \delta \xi_i^k(\mathbf{y})}{\partial y_j} dV \\ - \int_{Y^f} \frac{\partial \delta \xi_i^k(\mathbf{y})}{\partial y_i} \pi^k(\mathbf{y}) dV = \int_{Y^f} \delta \xi_i^k(\mathbf{y}) dV, \quad \forall \delta \xi_i^k(\mathbf{y}) \in \mathcal{C}_\#(Y^f), \end{aligned} \quad (14a)$$

$$\int_{Y^f} \frac{\partial \xi_i^k(\mathbf{y})}{\partial y_i} \delta \pi^k(\mathbf{y}) dV = 0, \quad \forall \delta \pi^k(\mathbf{y}) \in \mathcal{C}_\#(Y^f), \quad (14b)$$

$$\xi_i^k(\mathbf{y}) = 0, \quad \text{on } \Gamma^{sf}, \quad (14c)$$

$$\int_{Y^f} \pi^k(\mathbf{y}) dV = 0. \quad (14d)$$

**Cell problem 4:** Find the function  $\zeta(\mathbf{y}) \in \mathcal{C}_\#(Y^f)$  such as:

$$\begin{aligned} \int_{Y^f} \frac{1}{i\omega \rho^f C_p^f} \kappa_{ij}^f \frac{\partial \zeta(\mathbf{y})}{\partial y_j} \frac{\partial \delta \zeta(\mathbf{y})}{\partial y_i} dV + \int_{Y^f} \zeta(\mathbf{y}) \delta \zeta(\mathbf{y}) dV \\ = \int_{Y^f} \delta \zeta(\mathbf{y}) dV, \quad \forall \delta \zeta(\mathbf{y}) \in \mathcal{C}_\#(Y^f) \end{aligned} \quad (15a)$$

$$\zeta(\mathbf{y}) = 0 \quad \text{on } \Gamma^{sf}. \quad (15b)$$

228 The equations (12b), (13b) and (14d) play the role of constraint conditions suppressing  
229 rigid modes and assuring the uniqueness of solutions of corresponding cell problems.

#### 230 3.1.4. Effective properties and macroscopic governing equations

The homogenization method provides effective constitutive relations for averaged fields at the order of  $\epsilon^0$ . These equations lead to Biot's model of poroelasticity as follows with the disappearance of the superscript <sup>(0)</sup> for the sake of convenience:

$$\sigma_{ij} = C_{ijkl}^* \varepsilon_{kl}^s(\mathbf{x}) - \alpha_{ij} p^f(\mathbf{x}), \quad (16a)$$

$$p^f(\mathbf{x}) = -M \left[ \frac{1}{i\omega} \frac{\partial w_i}{\partial x_i} + \alpha_{ij} \varepsilon_{ij}^s(\mathbf{x}) \right] \quad (16b)$$

where  $\sigma_{ij}$  are the components of the total stress tensor,  $w_i = \phi(v_i^f - \dot{u}_i^s)$  with  $\phi$  the porosity,  $C_{ijkl}^*$  are the components of the fourth-order drained homogenized stiffness tensor,  $\alpha_{ij}$  are

the components of Biot's second-order tensor of effective stress coefficients coupling the solid and fluid phases and  $M$  is known as Biot's modulus. These effective properties are determined by means of the solutions of cell problems (see Section 3.1.3) as follows:

$$C_{ijkl}^* = \left\langle c_{ijpq}^s \left( \frac{\partial \chi_p^{kl}(\mathbf{y})}{\partial y_q} + \delta_{pk} \delta_{ql} \right) \right\rangle, \quad (17)$$

$$\alpha_{ij} = \phi \delta_{ij} + B_{ij}, \quad B_{ij} = \left\langle c_{ijkl}^s \frac{\partial \beta_k(\mathbf{y})}{\partial y_l} \right\rangle = - \left\langle \frac{\partial \chi_k^{ij}(\mathbf{y})}{\partial y_k} \right\rangle, \quad (18)$$

$$M = (\phi c + S)^{-1}, \quad S = \left\langle \frac{\partial \beta_k(\mathbf{y})}{\partial y_k} \right\rangle \quad (19)$$

where the symbol  $\langle * \rangle$  denotes the volume average of  $*$  over a representative volume element  $Y$

$$\langle * \rangle = \frac{1}{|Y|} \int_Y * \, dV \quad (20)$$

with  $|Y|$  being the volume of representative volume element and  $c = c(\omega)$  is the effective fluid compressibility which is a frequency-dependent function due to the thermal perturbation. This quantity is identified through the solution of the cell problem presented in Eq. (15) as:

$$c = \frac{\gamma^f - (\gamma^f - 1)\phi^{-1} \langle \zeta(\mathbf{y}) \rangle}{\gamma^f P^f}. \quad (21)$$

The macroscopic governing equations can be written as:

$$\frac{\partial \sigma_{ij}}{\partial x_j} = -\bar{\rho} \omega^2 u_i^s + \rho^f i \omega w_i, \quad (22a)$$

$$w_i = -H_{ik} \left( \frac{\partial p^f}{\partial x_k} - \rho^f \omega^2 u_k^s \right), \quad (22b)$$

where  $\bar{\rho} = (1 - \phi)\rho^s + \phi\rho^f$ . Equation (22b) depicts Darcy's generalized law and  $H_{ik} = H_{ik}(\omega)$  denotes the components of the effective generalized Darcy second-order tensor [20]. This frequency-dependent quantity is determined through the solution of the cell problem presented in Eq. (14) by the following relation:

$$H_{ik} = \langle \xi_i^k(\mathbf{y}) \rangle. \quad (23)$$

The effective dynamic permeability second-order tensor is deduced from the generalized Darcy coefficient through the following relation [21]:

$$k_{ik} = \eta H_{ik}. \quad (24)$$

At the limit  $K^f/K^s \rightarrow 0$  where  $K^s$  and  $K^f$  are respectively the static bulk modulus of the solid and fluid phases, the solid phase can be considered as motionless and the poroelastic material can be regarded as a perfect fluid of the equivalent dynamic mass density  $\boldsymbol{\rho}_{eq}$  and the equivalent dynamic bulk modulus  $K_{eq}$  [18, 22]. Note that  $\boldsymbol{\rho}_{eq}$  is a second-order tensor. These two equivalent parameters can respectively be expressed in terms of the effective dynamic mass density  $\boldsymbol{\rho}_{eff}^f$  [23] and the effective dynamic bulk modulus  $K_{eff}^f$  [24] of the fluid phase as follows:

$$\boldsymbol{\rho}_{eq} = \frac{1}{\phi} \boldsymbol{\rho}_{eff}^f, \quad K_{eq} = \frac{K_{eff}^f}{\phi}. \quad (25)$$

The effective dynamic bulk modulus  $K_{eff}^f$  of the fluid phase is the inverse of the effective fluid compressibility  $c$  while the effective dynamic mass density  $\boldsymbol{\rho}_{eff}^f$  of the fluid phase can be calculated through the characteristic function  $\xi(\mathbf{y})$  as:

$$\rho_{eff,ik}^f = \frac{\phi}{i\omega} \langle \xi_i^k(\mathbf{y}) \rangle^{-1}. \quad (26)$$

### 231 3.2. Numerical implementation

232 Cell problems were solved by using the finite element method and simulations were carried  
 233 out using the Comsol Multiphysics software [25]. Cell problems 1 and 2 corresponding to  
 234 Eqs. (12) and (13) are frequency-independent, whereas cell problems 3 and 4 represented  
 235 by Eqs. (14) and Eqs. (15) are frequency-dependent. These last two problems need to be  
 236 solved at each frequency of interest.

237 After the resolution of cell problems, the characteristic functions corresponding to these  
 238 problems are determined and the effective properties of the homogenized material can be  
 239 attained by substituting these functions into Eqs. (17)-(19), (21), (23) and (26).

240 Cell problems 1 and 2 are solved on the geometry corresponding to the solid phase,  
 241 whereas the resolution of the two remaining cell problems is conducted on the geometry  
 242 representing the fluid phase. These geometries are meshed *via* unstructured meshes of  
 243 quadratic Lagrange tetrahedral elements by respecting periodic constraints between opposite  
 244 boundaries. For an elementary cube whose the side length is  $305 \mu\text{m}$ , the numbers of degrees  
 245 of freedom of the solid and fluid phases are about  $342 \times 10^3$  and  $451 \times 10^3$ , respectively.

## 246 4. Construction of idealized representative volume elements

### 247 4.1. Construction of idealized elementary pore and matrix

248 The construction of a unit cell is based on three factors: pore shape, pore entrance  
 249 dimension and porosity. Because the porous material was obtained after NaCl extraction, or  
 250 in other words, NaCl particles left the void space constituting the porosity of the material, the  
 251 pore shape correlates strictly with the shape of NaCl particles. NaCl is an ionic compound  
 252 made of sodium and chloride atoms. When a number of these molecules get together to  
 253 form a crystal, they tend to arrange themselves in a cubic pattern. As a result, NaCl  
 254 crystals are also cubic and this pattern is confirmed by the SEM image of NaCl particles

255 in Fig. 4a. Considering this fact, the shape of a pore corresponding to a NaCl particle is  
 256 regarded as cubic; more precisely we have a cubic pore with fillet external edges (see Fig. 4c).  
 257 It is useful to remember that the fusion of NaCl cubic particles has many contact/fusion  
 258 modes, namely “face to face”, “side to side”, “point to point” modes, etc., [26], but the “face  
 259 to face” mode has been more frequently found in SEM micrographs (see Figs. 2 and 4b);  
 260 therefore, this mode was chosen to construct the unit cell, leading to square-shaped pore  
 261 entrances. From the MIP profile, these square entrances are considered to have the side  
 262 dimension  $l_t = 180 \mu\text{m}$  (see Fig. 4d). Because of the value of porosity 83% identified by  
 263 determination of the mass and the volume of a piece of the porous material as described  
 264 above with the NaCl volume fraction 82%, the unit cell is constructed so that its porosity  
 265 is 83%. To make the fillet-edge cubic pore, an elementary cube with side length  $l = 305 \mu\text{m}$   
 266 and three orthogonal cylinders with a radius of  $177 \mu\text{m}$  and a length of  $305 \mu\text{m}$  are created  
 267 with the same center point. The elementary pore is then the intersection of the elementary  
 268 cube and the three cylinders. By calculating the difference between the elementary cube  
 269 and the elementary pore, the elementary matrix is obtained (see Fig. 4d). Void space in  
 270 porous media is described as a network of pores and throats where larger spaces are referred  
 271 to as pores, which are connected by smaller spaces (constrictions) referred to as throats.  
 272 Therefore, hereafter, the term “pore size” is used to refer to the length of the elementary  
 273 cube’ side.

#### 274 4.2. Construction of representative volume elements

275 In this section, the effect of the unit cell arrangements in the three-dimensional space  
 276 is introduced in order to study their impact on the effective mechanical, hydraulic and  
 277 acoustic properties. Based on the geometric configuration of a unit cell made of an idealized  
 278 elementary pore and matrix constituting the fluid and solid phase presented in section 4.1,  
 279 four different ordered arrangements of this elementary cell are presented in Fig. 5. These  
 280 four arrangements result in four distinct representative volume elements described as follows:

- 281 • Arrangement 1: the elementary cells are vertically and horizontally aligned;
- 282 • Arrangement 2: the elementary cells in the second layer are horizontally offset with to  
283 those of the first layer, by a distance  $l/2$  along of the sets of rows;
- 284 • Arrangement 3: the elementary cells in the second layer are horizontally and vertically  
285 offset with to those of the first layer, by a distance  $l/2$  along of the sets of rows;
- 286 • Arrangement 4: each elementary cell is horizontally offset with to the next cell, by a  
287 distance  $l/2$  along of the sets of rows.

288 The choice of these arrangements is, in particular, due to the fact that it maintains the  
 289 porosity at the same value of 83%, which corresponds to the porosity of the porous material  
 290 obtained by the elaboration process. Moreover, because the use of the elementary cube as a  
 291 representative volume element is enough to express arrangement 1 and the symmetry of the  
 292 cube ensures the periodicity in three directions, the representative volume element in this

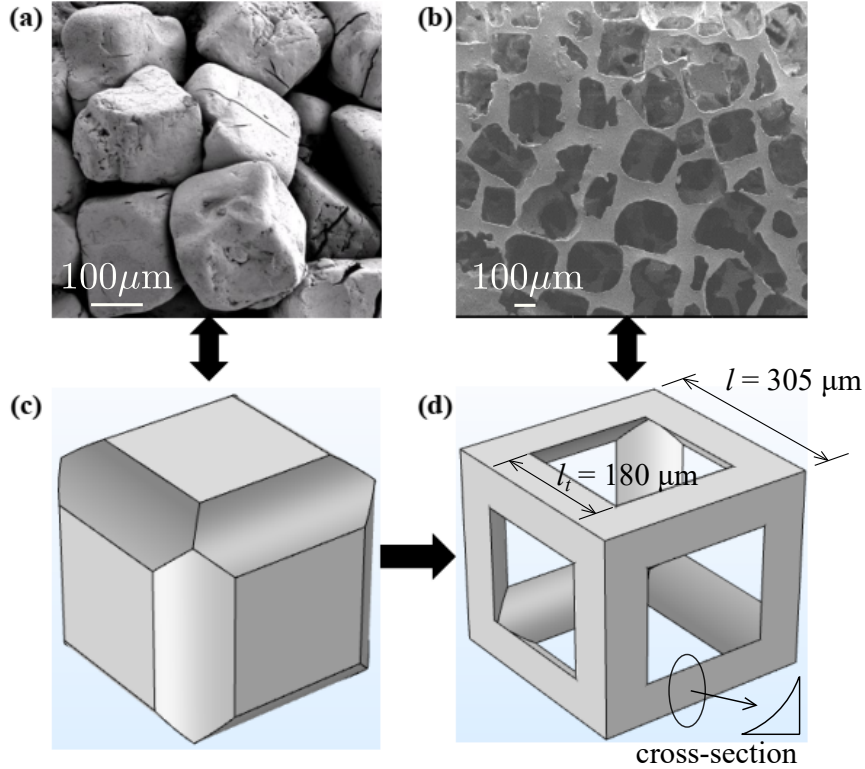


Figure 4: Construction of the elementary pore and matrix by the idealized periodic microstructure. (a) SEM image of sintered NaCl particles; (b) SEM image of the present porous material; (c) Elementary cubic pore; (d) Elementary cubic matrix.

293 case has a side length of  $l$ . For the other arrangements, the corresponding representative vol-  
 294 ume elements are cubic volumes with a side length of  $2l$  and are constructed in a manner so  
 295 that each arrangement is well represented and the periodic conditions are satisfied. All con-  
 296 sidered representative volume elements possess the orthotropic symmetry, in which there are  
 297 3 mutually orthogonal planes of reflection symmetry; especially, the representative volume  
 298 element for arrangement 1 refers to the cubic symmetry with also  $\pi/2$  rotation symmetry  
 299 with respect to those planes while the representative volume element for arrangements 3 and  
 300 4 involves the transversely isotropy with additional axial symmetry about an axis shown in  
 301 Fig. 5. It is the axis orthogonal to the square-marked surface for arrangement 3 and to the  
 302 star-marked surface for arrangement 4.

## 303 5. Sound absorption of a poroelastic plate

In order to assess the sound absorption capacity of the elaborated porous material, a normal incidence acoustic problem is considered for a plate of thickness  $d$  made of this material with a rigid impervious backing at one side and surrounded by the air from another (see Fig. 6). The problem is described in the Cartesian coordinate system with an orthogonal basis  $(\mathbf{e}_1, \mathbf{e}_2, \mathbf{e}_3)$  and the position vector  $\mathbf{x} = (x_1, x_2, x_3)$ . Because the study focuses on the

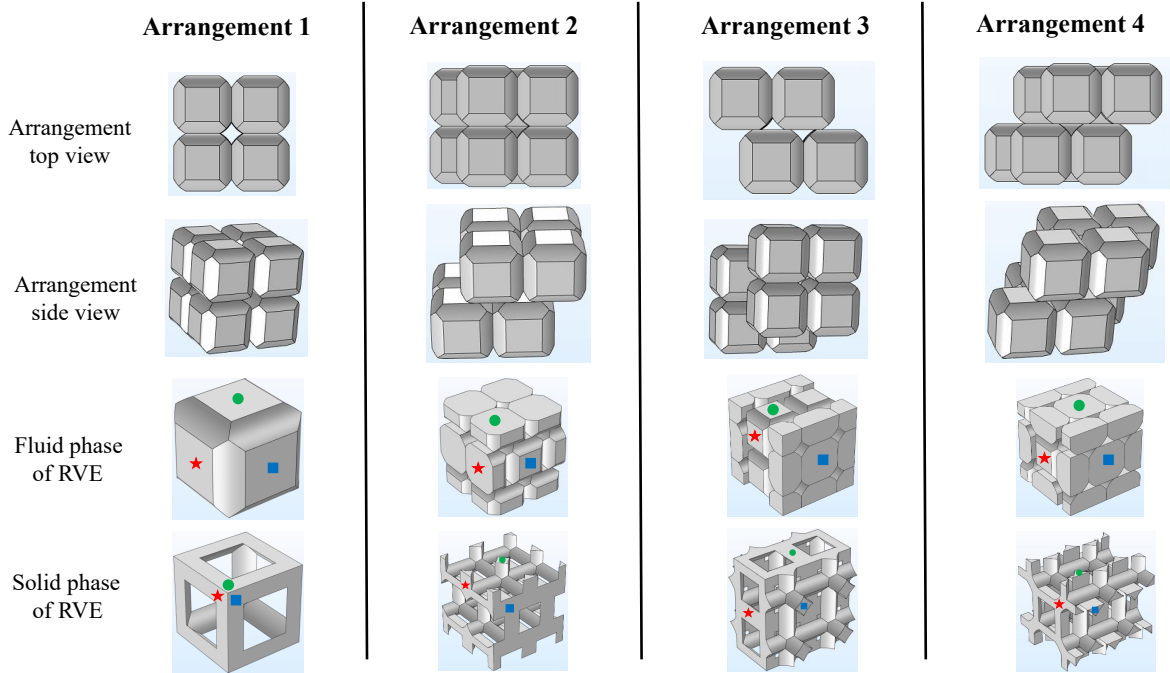


Figure 5: Representative volume elements of the four pore arrangements. The star-, square- and circle-marked surfaces correspond to the surface orthogonal to the incidence direction of the plane pressure described in section 5.

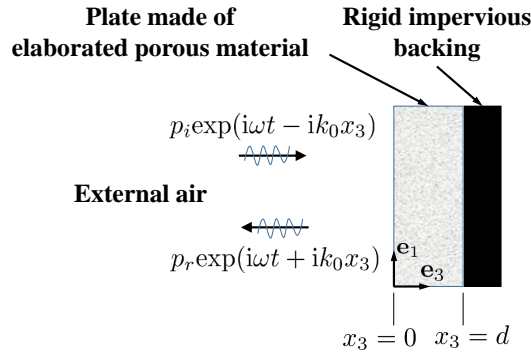


Figure 6: Normal incidence acoustic problem for a plate of thickness  $d$  made of the elaborated porous material.

normal incidence, a one-direction problem is considered and the schema in Fig. 6 shows the case of  $x_3$ -direction normal incidence. The analytical solution of one-dimensional wave propagation in an anisotropic poroelastic layer coupled with fluids has been derived in [27, 28]. The incident plane pressure wave, depicted by  $p_i \exp(i\omega t - ik_0 x_3)$  with the amplitude  $p_i$  and the wavenumber  $k_0$ , strikes the porous plate and is then reflected. The reflected wave field is denoted by  $p_r \exp(i\omega t + ik_0 x_3)$  where  $p_r$  is the amplitude. This problem is described in



detail in [29, 28]. The boundary conditions associated with this problem are the followings:

$$\sigma_{33} = -p^{(n)}, p^f = p^{(n)}, i\omega u_3^s + w_3 = v^{air} \quad \text{at } x_3 = 0, \quad (27)$$

$$u_3^s = 0, w_3 = 0 \quad \text{at } x_3 = d, \quad (28)$$

where  $p^{(n)} = p_i \exp(i\omega t - ik_0 x_3) + p_r \exp(i\omega t + ik_0 x_3)$  and  $v^{air}$  are respectively the pressure field and the velocity of the external air. By introducing the surface impedance  $Z_s$  defined by  $Z_s = p^{(n)}(0)/v^{air}(0)$  at  $x_3 = 0$  and applying the boundary conditions (27) and (28) to the macroscopic governing equations (22), the surface impedance  $Z_s$  can be determined. In a normal incidence problem, the reflection coefficient  $R$  which is generally defined as the ratio  $p_r/p_i$  can be calculated from the surface impedance through the following relation [30]:

$$R(\omega) = \frac{Z_s - Z_0}{Z_s + Z_0}, \quad (29)$$

where  $Z_0 = \rho_0 c_0$  is the characteristic impedance of the air with  $\rho_0$  the density of the air at rest and  $c_0$  the sound velocity in the air. Subsequently, the absorption coefficient  $\alpha_a$  is determined by [30]:

$$\alpha_a(\omega) = 1 - |R|^2. \quad (30)$$

## 304 6. Numerical results and discussion

305 In this section, the effect of pore arrangements, porosity and pore size on effective me-  
 306 chanical, hydraulic and acoustic properties are presented and discussed in order to improve  
 307 the performances of this new bio-based porous material. By conducting three-microphone  
 308 impedance tube experiments on the porous material, the measured effective acoustic prop-  
 309 erties are discussed in comparison with the modeling results.

### 310 6.1. Effect of pore arrangements on effective properties

311 The epoxy matrix is isotropic and its tensor  $c_{ijkl}^s$  of elastic properties is characterized  
 312 by two independent coefficients, Young's modulus  $E^s = 4.5 \times 10^9$  Pa and Poisson's ratio  
 313  $\nu^s = 0.34$ . Its density is given by  $\rho^s = 1318$  kg/m<sup>3</sup>. The fluid phase is taken to be the air with  
 314 following properties:  $\rho^f = 1.225$  kg/m<sup>3</sup>,  $\eta = 1.81 \times 10^{-5}$  Pa.s,  $\kappa_0^f = 2.41 \times 10^{-2}$  W/(m.K),  
 315  $\gamma^f = 1.4$  at  $P^f = 101325$  Pa. The sound velocity in the air is given as  $c_0 = 331.6$  m/s.

316 Note that with these data and with a frequency ranging from 4 to 4500 Hz, the associated  
 317 wavelength  $\lambda$  varies in the range given by [0.638, 7.981] (in meter) in the poroelastic domain.  
 318 These values show that the scale separation condition necessary for the relevance of the  
 319 homogenization-based modelling which requires  $\lambda \gg \ell_R$ , where  $\ell_R \sim 0.60 \times 10^{-3}$  m is the  
 320 representative volume element size, is well respected.

In the case of arrangement 1, the numerical results of effective elasticity tensor  $\mathbf{C}^*$  in the Voigt notations and Biot's tensor  $\boldsymbol{\alpha}$  are as follows:

$$\mathbf{C}^* = \begin{bmatrix} 332.9 & 34.4 & 34.4 & 1.6 \times 10^{-3} & -5.9 \times 10^{-3} & -34.3 \times 10^{-3} \\ & 332.9 & 34.4 & -13.4 \times 10^{-3} & 4.5 \times 10^{-3} & -31.1 \times 10^{-3} \\ & & 332.9 & -10.1 \times 10^{-3} & -10.0 \times 10^{-3} & 9.5 \times 10^{-3} \\ & & & 21.0 & -3.3 \times 10^{-3} & -1.6 \times 10^{-3} \\ \text{sym.} & & & & 21.0 & -1.9 \times 10^{-3} \\ & & & & & 21.0 \end{bmatrix} \text{ (MPa)}$$

$$\boldsymbol{\alpha} = \begin{bmatrix} 0.969 & 43.63 \times 10^{-7} & 8.94 \times 10^{-7} \\ & 0.969 & 17.07 \times 10^{-7} \\ \text{sym.} & & 0.969 \end{bmatrix}$$

322 Regarding frequency-dependent properties, namely Biot's modulus  $M$ , the effective com-  
 323 pressibility  $c$  and the effective dynamic permeability tensor  $\mathbf{k}$ , their values at frequency of  
 324 10 Hz read:

$$M = (0.12 + i4.08 \times 10^{-4}) \times 10^6 \text{ (Pa)} \quad (31)$$

$$c = (9.87 - i 0.03) \times 10^{-6} \text{ (Pa}^{-1}\text{)} \quad (32)$$

$$\mathbf{k} = \begin{bmatrix} 1.05 \times 10^{-9} & 2.06 \times 10^{-13} & 8.71 \times 10^{-13} \\ & 1.05 \times 10^{-9} & 9.68 \times 10^{-13} \\ \text{sym.} & & 1.05 \times 10^{-9} \end{bmatrix} \quad (33)$$

$$-i \begin{bmatrix} 1.01 \times 10^{-11} & 2.42 \times 10^{-15} & 1.25 \times 10^{-14} \\ & 1.01 \times 10^{-11} & 1.39 \times 10^{-14} \\ \text{sym.} & & 1.01 \times 10^{-11} \end{bmatrix} \text{ (m}^2\text{)} \quad (34)$$

325 It can be seen that the form of tensors  $\mathbf{C}^*$ ,  $\boldsymbol{\alpha}$  and  $\mathbf{k}$  reflects well the cubic symmetry of  
 326 the representative volume element in the case of arrangement 1 for which the tensor  $\mathbf{C}^*$  has  
 327 3 independent coefficients while two tensors  $\boldsymbol{\alpha}$  and  $\mathbf{k}$  are diagonal with  $\alpha_{11} = \alpha_{22} = \alpha_{33}$  and  
 328  $k_{11} = k_{22} = k_{33}$ .

329 More generally, it is assumed that the representative volume elements associated with 4  
 330 arrangements are taken so that their star-, square- and circle-marked surfaces are orthogonal  
 331 to  $\mathbf{e}_1$ -,  $\mathbf{e}_2$ - and  $\mathbf{e}_3$ -direction, respectively, and the indices for components of effective tensors  
 332 are determined accordingly. In a general orthotropic case, the effective elasticity tensor  $\mathbf{C}^*$   
 333 has 9 independent components and Biot's tensor  $\boldsymbol{\alpha}$  has a diagonal form with 3 independent  
 334 components. Table 1 shows 9 components of the effective elasticity tensor and the diagonal  
 335 components of Biot's tensor for the 4 cases of pore arrangements. It is easily seen that the  
 336 results reflect well the form of the elasticity tensor and Biot's tensor resulting from the ar-  
 337 rangements' symmetry. While the elasticity tensor possesses 9 independent components and  
 338 Biot's tensor has 3 independent components for arrangement 2, corresponding to the general  
 339 orthotropic symmetry, the elasticity and Biot's tensors for the arrangements 3 and 4 reflect  
 340 the transversely isotropy with 5 and 2 independent components, respectively. Particularly,  
 341 in Tab. 1, while the homogenized materials are generally more resistant to tension than to  
 342 shear and most of modulus components as well as components of Biot's tensor do not vary  
 343 strongly according to pore arrangements, a noticeable drop in tension modulus  $C_{22}^*$  along  
 344 with a sharp rise in shear modulus  $C_{55}^*$  in case of arrangement 3 implies a degradation of  
 345 tension resistance and an improvement in shear resistance of the material related to the axis  
 346 of transverse isotropy corresponding to arrangement 3.

Table 1: Non-zero components of effective elasticity tensor  $\mathbf{C}^*$  in the Voigt notations and Biot's tensor  $\boldsymbol{\alpha}$  in different cases of pore arrangements.

		Pore arrangements			
		1	2	3	4
Components of tensor $\mathbf{C}^*$ ( $10^6$ Pa)	$C_{11}^*$	332.9	341.9	381.4	346.6
	$C_{22}^*$	332.9	364.3	141.9	324.0
	$C_{33}^*$	332.9	214.6	381.0	324.0
	$C_{44}^*$	21.0	15.1	23.8	11.4
	$C_{55}^*$	21.0	33.9	90.7	37.5
	$C_{66}^*$	21.0	38.0	23.8	37.5
	$C_{12}^*$	34.4	45.0	59.1	49.7
	$C_{13}^*$	34.4	56.2	86.1	49.7
	$C_{23}^*$	34.4	25.3	59.1	62.0
	Symmetry	cubic	orthotropic	orthotropic	orthotropic
Components of tensor $\boldsymbol{\alpha}$	$\alpha_{11}$	0.969	0.965	0.959	0.965
	$\alpha_{22}$	0.969	0.966	0.980	0.966
	$\alpha_{33}$	0.969	0.977	0.959	0.966

347 In Figs. 7 and 8, we present respectively results of the effective dynamic permeability and  
348 effective dynamic compressibility. The absorption characteristics of the considered porous  
349 materials are quantified using the complex values of these two parameters. The presence  
350 of components of the permeability tensor in Fig. 7 indicates the properties of this tensor  
351 and reflects the symmetry type corresponding to each arrangement. For instance, in the  
352 case of arrangement 1, the permeability tensor is spherical for which  $k_{11} = k_{22} = k_{33}$ ,  
353 implying the cubic symmetry, whereas for arrangement 4, the tensor has a diagonal form  
354 with 2 independent components, representing the transversely isotropy. Comparing the  
355 permeability results for arrangement 1 with those for other arrangements, one can notice  
356 that while arrangements 2, 3 and 4 have the permeability component *versus* frequency  
357 curves with similar corresponding patterns, the curves for arrangement 1 follow their own  
358 specific patterns and there is a difference of at least 10 times between the permeabilities  
359 for arrangement 1 and those for the others. In addition, regarding the two arrangements  
360 corresponding to the transverse isotropy, an attention is also paid to the fact that the  
361 permeability component related to the axis of rotation symmetry has the lowest absolute  
362 value compared with two other components in case of arrangement 3 and inversely has the  
363 highest absolute value in case of arrangement 4. Regarding compressibility, Fig. 8 shows that  
364 the results for the three arrangements 2, 3 and 4 prove to be similar, while being slightly  
365 different from the compressibility for arrangement 1. This reveals that the arrangements  
366 have not much influence on the compressibility of the materials.

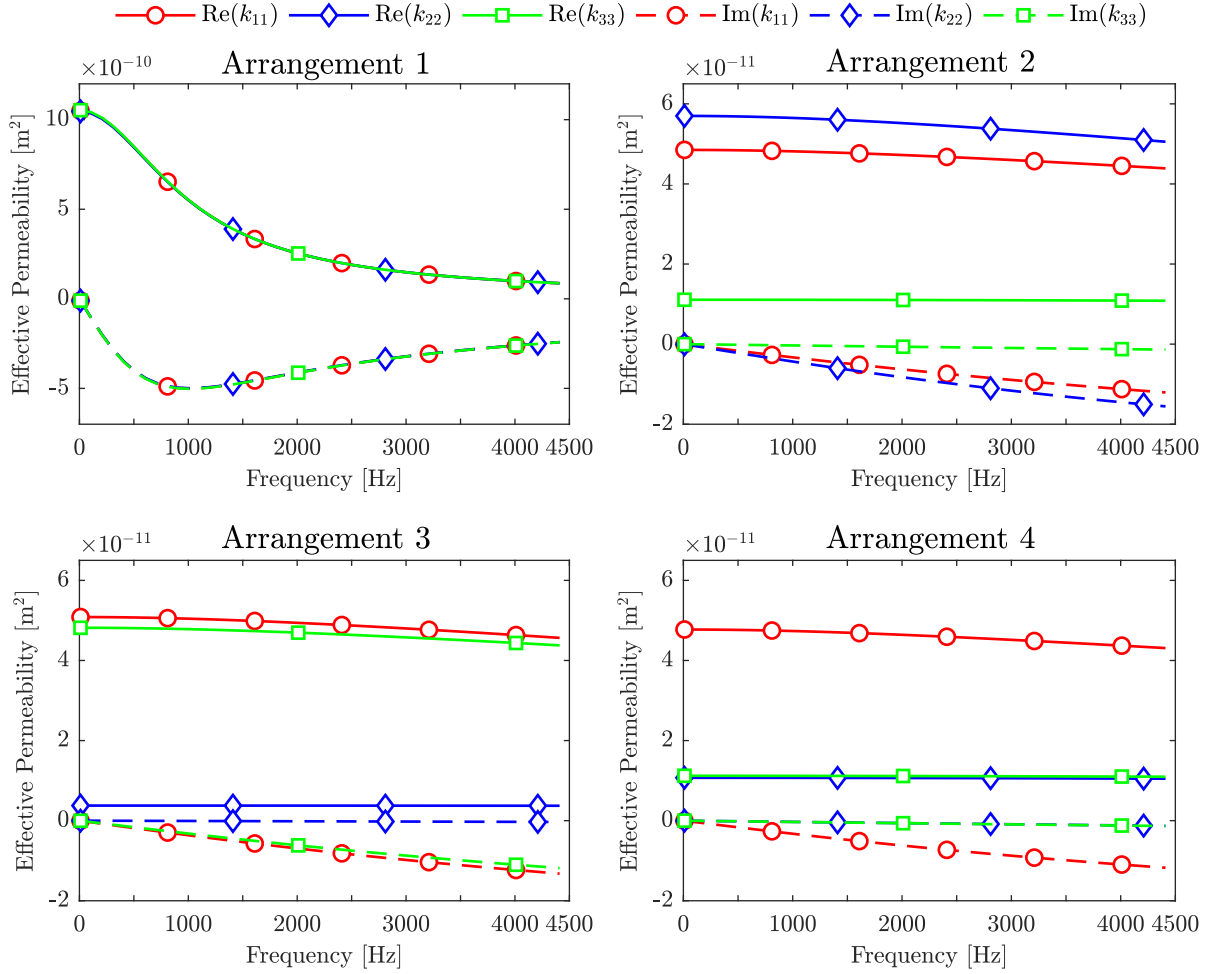


Figure 7: Dependence of non-zero components of the effective dynamic permeability tensor on different pore arrangements.

### 367 6.2. Effect of pore arrangements on sound absorption of a poroelastic plate

368 The dependence of sound absorption on pore arrangements is illustrated in Fig. 9. In  
369 this figure, the sound absorption coefficient has been calculated for different orientations  
370 of the incident plane pressure wave, which is orthogonal to the star-, square- and circle-  
371 marked surfaces defined in Fig. 5. From Fig. 9, it can be noted again that the symmetry  
372 characteristics are well represented. Arrangement 1 with cubic symmetry gives the mate-  
373 rial with the same sound absorption coefficients for all three directions. Two arrange-  
374 ments with transverse isotropy result in materials of which sound absorption coefficients are the  
375 same for 2 directions orthogonal to the axis of rotation symmetry. Surprisingly, in case  
376 of arrangement 2, despite the difference in components related to  $\mathbf{e}_1$  and  $\mathbf{e}_2$  directions of  
377 the elasticity tensor, Biot's tensor as well as the permeability tensor, the sound absorption  
378 behaviours corresponding to these directions are relatively close to each other. Concerning  
379 arrangements 2, 3 and 4, one can also observe that the sound absorption is the best at

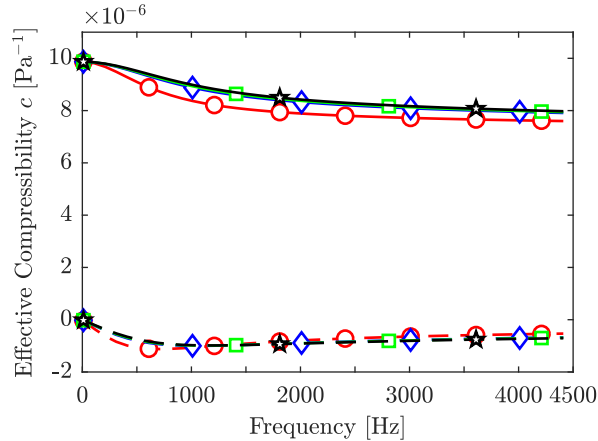


Figure 8: Dependence of the effective dynamic compressibility on different pore arrangements. Solid lines with circular, diamond, square and star markers represent respectively real parts of compressibility in case of arrangements 1, 2, 3 and 4. Dashed lines with circular, diamond, square and star markers represent respectively imaginary parts of compressibility in case of arrangements 1, 2, 3 and 4.

low frequencies in directions corresponding to a permeability smaller than those in other directions; inversely, the dominance of sound absorption is recorded at high frequencies in directions corresponding to a larger permeability. The fact that the compressibility values are similar for these three cases implies that the permeability, not the compressibility, is the key parameter resulting in the different sound absorption behaviours related to different placements of unit cells. Regarding arrangement 1 and in the frequency range of interest, one may notice the smallness of the sound absorption coefficient in this case, compared with those in other arrangements in spite of the fact that the permeability for this arrangement is much higher than those for other configurations. Normally, the increase in the permeability implies the entrance of more sound waves and hence the possibility of more attenuated sound energy. This result has to be relativized since it is generally valid for relatively large thicknesses. Nonetheless, besides higher permeability, the microstructure of the material resulting from arrangement 1 has also a larger pore entrance dimension compared to those for other configurations. This leads to the decrease of viscous damping and thermal dissipation, and therefore, results in the less effective sound absorption. This phenomenon is also observed in some parametric studies carried out in two dimensions for fibrous media [31] as well as in three dimensions for foams [14]. Because the pore arrangement duplicates the arrangement of porogen particles, we can link the porogen arrangement and the sound absorption. Although the porogen arrangement is not controlled yet in the present elaboration processing, the results of this section suggest that the arrangement of porogen particles should fall into types 2, 3 or 4 for the possibly highest sound absorption.

### 6.3. Experimental validation

#### 6.3.1. A comparison of acoustic properties

The validation of the model is performed by using three-microphone impedance tube experiments on the porous material for which the acoustic properties are measured. The

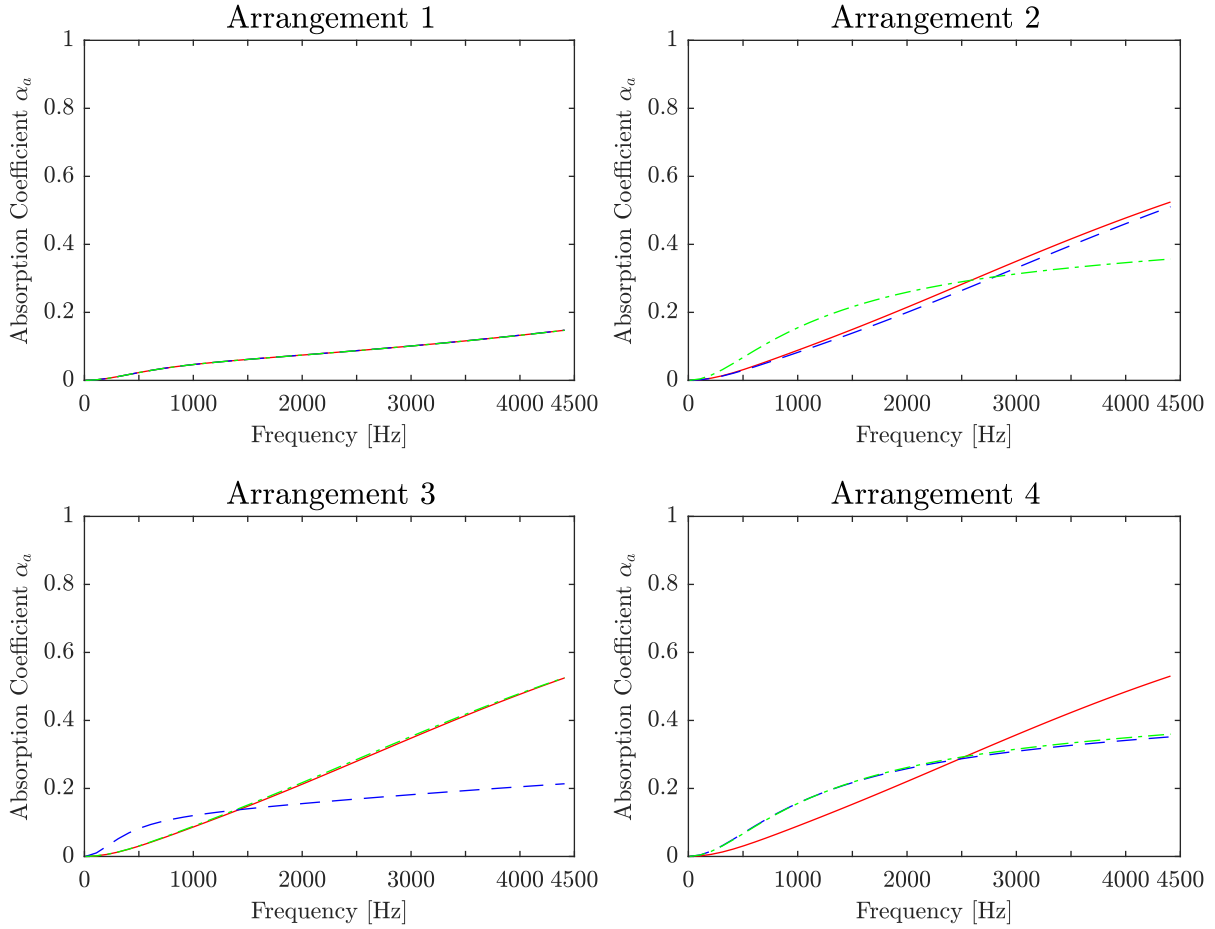


Figure 9: Dependence of sound absorption coefficients on different pore arrangements. The plate thickness is 0.5 cm. Solid, dashed and dashed-dot lines correspond to cases in which star-, square- and circle-marked surfaces of representative volume elements are orthogonal to the normal incidence direction, respectively.

405 configuration of the experiments is shown in Fig. 10. Two samples having the cylindrical  
 406 shapes with a diameter of 4 cm and a thickness of 0.5 cm were tested. For each sample,  
 407 two experiments were conducted for two opposite incidence surfaces. The tests were carried  
 408 out with a frequency ranging from 4 to 4500 Hz, under rigid impervious backing conditions  
 409 and normal incidence with acoustic plane waves. Three microphones measure the acoustic  
 410 pressures at their positions and the data are transferred to the computer equipped with  
 411 the TubeCell software developed by Matelys Resesarch Lab. This software analyses the  
 412 pressure data and computes acoustic properties such as the sound absorption coefficient, the  
 413 reflection coefficient, the surface impedance, the equivalent dynamic mass density and the  
 414 equivalent dynamic bulk modulus [32].

415 The experimental results are plotted in Figs. 11 to 13. For comparison purposes, these  
 416 figures also show the results obtained *via* the model described in this paper by using the  
 417 arrangement 1 which allows a best approximation to the effective parameters of interest.

418 In these figures, the experimental results are represented by their mean values alongside

419 the corresponding error bars at each frequency. The green and cyan colours are used for the  
 420 standard deviation.

421 A standardized measure of the dispersion is given by the coefficient of variation which  
 422 is also called relative standard deviation. The measure is expressed as a percentage and is  
 423 defined as the ratio of the standard deviation to the mean. This measure of the dispersion  
 424 is used in what follows for the acoustic properties.

425 In Fig. 11 (left), for real and imaginary parts of  $Z_s$ , the maximum values of relative  
 426 standard deviation are about 55% and 13% respectively.

427 In Fig. 11 (right), for real and imaginary parts of  $R$ , the maximum values of relative  
 428 standard deviation are about 6% and 4.5% respectively.

429 In Fig. 12, the maximum value of relative standard deviation of  $\alpha_a$  is about 15.9%.

430 In Fig. 13 (left), for real and imaginary parts of  $\rho_{eq,33}$ , the maximum values of relative  
 431 standard deviation are about 61.6% and 57.7% respectively.

432 In Fig. 13 (right), for real and imaginary parts of  $K_{eq}$ , the maximum values of relative  
 433 standard deviation are about 13.2% and 22.7% respectively.

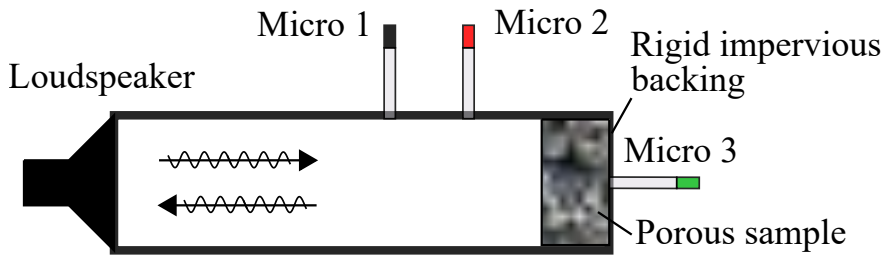


Figure 10: Sketch of experimental setup of three-microphone impedance tube experiment.

434 In Figs. 11 and 12, it can be seen that the model slightly underestimates the values  
 435 of absorption coefficient, corresponding to a little overestimation of the real part of the  
 436 values of reflection coefficient. The discrepancy is greater for higher frequencies. On the  
 437 contrary, a good match between model and experimental results is captured for frequencies  
 438 higher than 100 Hz for the values of surface impedance, whereas for lower frequencies, the  
 439 experimental values of surface impedance exhibit a fluctuation, and accordingly, a mismatch  
 440 between model and experimental results. From both model and experimental results, we  
 441 can observe that the absorption coefficient tends to increase when the frequency increases.  
 442 This observation is in accordance with the decrease in the module of the reflection coefficient  
 443 and the surface impedance with respect to the increase of the frequency.

444 The comparison is also carried out in terms of the equivalent dynamic density and the  
 445 equivalent dynamic bulk modulus (see Fig. 13). A good agreement between model and  
 446 experimental results is figured out for these two parameters in the frequency range higher  
 447 than 100 Hz. When the frequency is lower than this value, there are fluctuations in the ex-  
 448 perimental values of both parameters and accordingly the model's results deviate from the  
 449 experimental ones. The divergences observed for these two equivalent parameters, but also  
 450 for the surface impedance, may be attributed to a lack of accuracy due to edge constraints  
 451 on small samples. Additionally, the mismatch in the frequency range lower than 100 Hz may

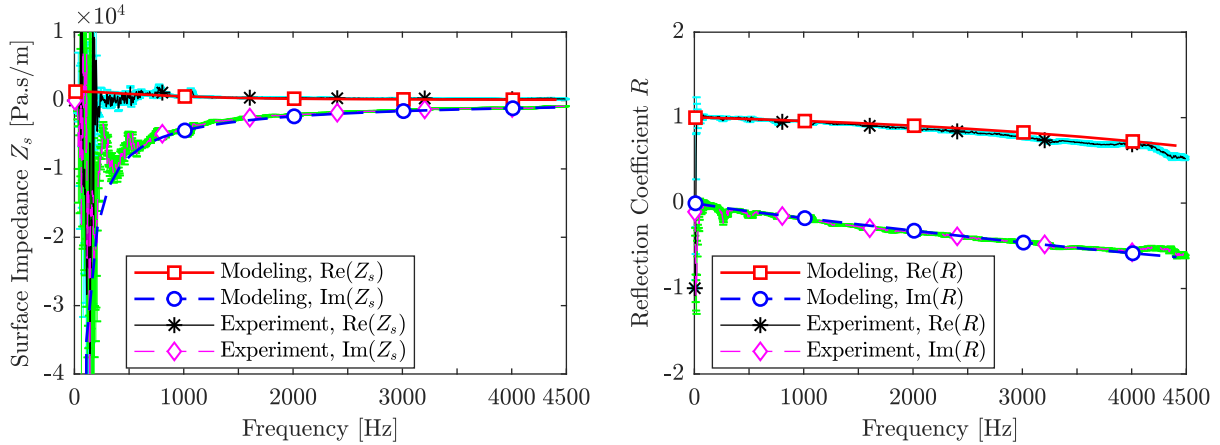


Figure 11: Comparison of model and experimental results of surface impedance  $Z_s$  (left) and normal incidence reflection coefficient  $R$  (right). The sample thickness is 0.5 cm. The green and cyan colours are used for the standard deviation.

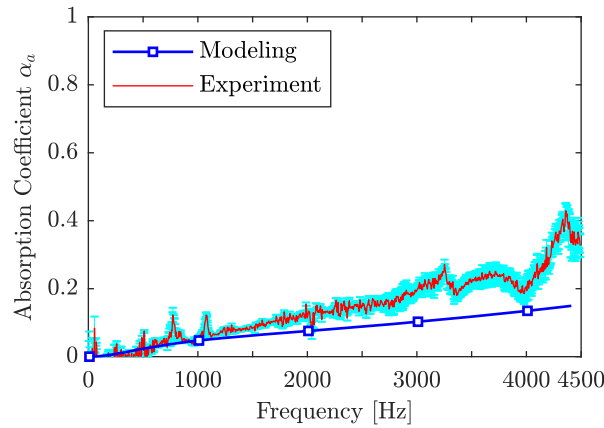


Figure 12: Comparison of model and experimental results of normal incidence absorption coefficient  $\alpha_a$  (see eq. (30)). The sample thickness is 0.5 cm. The green colour is used for the standard deviation.

452 be explained by the dependence of the three-microphone impedance tube testing on the up-  
 453 stream microphones inter-spacing, which becomes too small compared to large wavelengths  
 454 and degrades the accuracy of the testing method [32].

### 455 6.3.2. Interpretation of the differences between experimental and modeling results

456 In order to further investigate the observed discrepancies between model and experimen-  
 457 tal results of the absorption coefficient in the high frequency range, an important step is to  
 458 understand how transport properties depend on the details of the porous microstructure.  
 459 From the periodic unit cell describing the idealized pore structure of the porous material (see  
 460 Fig. 4d), we first proceed to numerically solve the appropriate transport equations (Stokes  
 461 hydrodynamic equations for viscous fluid flow, potential equations for non-viscous fluid flow,



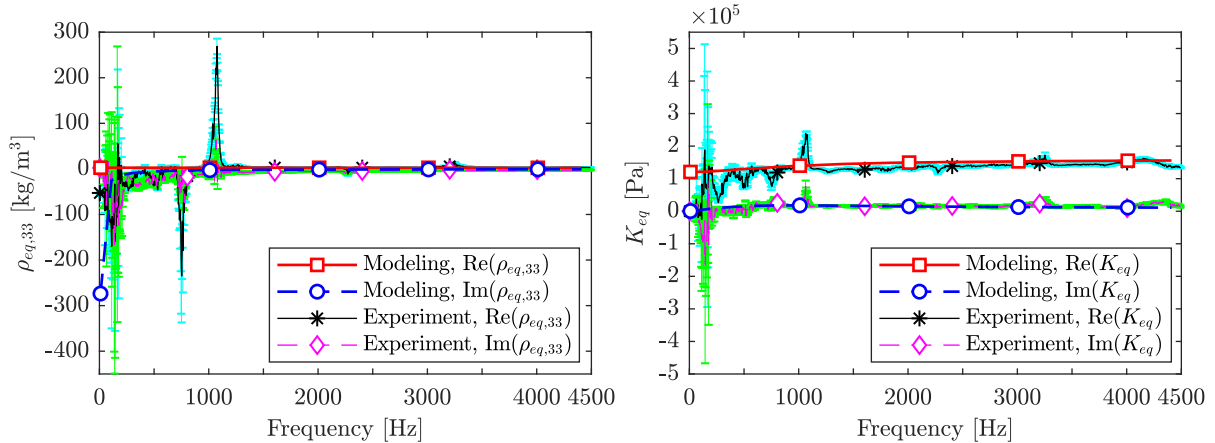


Figure 13: Comparison of numerical and experimental results of equivalent dynamic density component  $\rho_{eq,33}$  (left) and equivalent dynamic bulk modulus  $K_{eq}$  (right). The sample thickness is 0.5 cm. The green and cyan colours are used for the standard deviation.

462 Poisson equations for heat diffusion) in order to provide a description of the transport pro-  
 463 cesses in the modeled porous material [31, 14]. Then, an experimental characterization of the  
 464 transport parameters is also obtained from acoustic measurements [23, 24]. Table 2 shows  
 465 a comparison between experimentally and numerically determined transport parameters.

466 It should be noted that the viscous characteristic length  $\Lambda$  [33] is a pore-volume-to-surface  
 467 ratio of the pore-solid interface in which each area or volume element is weighted according  
 to the local value of the non-viscous fluid flow field.

Table 2: Effective properties including purely geometrical and transport properties determined numerically from the arrangement 1 and compared with the experimental results.

	$\phi$	$\Lambda$ ( $\mu\text{m}$ )	$k$ ( $\text{m}^2$ )
Simulation	0.83	112	$10.1 \times 10^{-10}$
Experiment	$0.83 \pm 0.02$	$36 \pm 23$	$(10.1 \pm 3.0) \times 10^{-10}$
	$\alpha_\infty$	$\Lambda'$ ( $\mu\text{m}$ )	$k'$ ( $\text{m}^2$ )
Simulation	1.23	203	$22.6 \times 10^{-10}$
Experiment	$1.23 \pm 0.57$	$84 \pm 24$	$(11.0 \pm 6) \times 10^{-10}$

468  
 469 Consequently, the quantity  $\Lambda$  has the dimension of length and gives a good estimate of  
 470 the smallest interconnections between pores, because of a weighting procedure implied in  
 471 its definition. The experimental value of  $\Lambda$  is about a third of the numerical one obtained  
 472 with the idealized unit cell, revealing that the observable disorder in Figs. 4a) and 4b) re-  
 473 duces significantly the interconnection between open pores. The experimental value of the  
 474 tortuosity  $\alpha_\infty$  [34] is in agreement with the one actually computed. The thermal charac-  
 475 teristic length  $\Lambda'$  [35] is another factor like  $\Lambda$  above, but without weighting and is therefore

476 a simple length scale that characterizes, in some way, the pore diameter. Comparing the  
 477 numerical and experimental values of the thermal characteristic length  $\Lambda'$ , it can be seen  
 478 that the experimental value of the effective pore diameter is lower than the one given by the  
 479 idealized model. Figures 4a) and 4b) show a surface roughness increasing the surface area of  
 480 the pore-solid interface, which has the effect of decreasing the overall pore-volume-to-surface  
 481 ratio. We note that the other measured macroscopic parameters, namely the porosity  $\phi$  and  
 482 the static viscous permeability  $k$ , are in agreement with the computed ones. Also worth  
 483 being noted, the characterized static thermal permeability  $k'$  [36], which should satisfy the  
 484 inequality  $k' \geq k$ , is close to the value of the static viscous one. Johnson *et al.* [34] and  
 485 Lafarge *et al.* [36] showed that the equivalent dynamic density  $\rho_{eq,33}(\omega)$  and the equivalent  
 486 dynamic bulk modulus  $K_{eq}(\omega)$  can be adequately described by approximate but robust  
 487 semi-phenomenological models based on these macroscopic parameters  $\phi, \Lambda', k, \alpha_\infty, \Lambda$  and  
 488  $k'$ . The better agreement between the numerically characterized and measured behaviour  
 489 of the sound absorption is indicative of the fact that the periodic unit cell captures most  
 490 of the physics of the visco-thermal exchanges, but that a more accurate representation of  
 491 the high frequency behaviour could eventually be obtained by introducing a disorder in the  
 492 pore space.

#### 493 6.4. Effect of porosity

494 In order to investigate the influence of porosity on effective properties and acoustic  
 495 behaviour of the material, three cases of porosity, namely 69%, 83% and 96%, are under  
 496 consideration in the context of arrangement 1 in which the elementary pore and matrix play  
 497 the role of the unit cell. The porosity is varied in a manner so that the pore size remains  
 498 unchanged. Note that in this paper, the pore size means side length of the elementary cube,  
 499 hence referring also to the size of the unit cell in case of arrangement 1, that is  $l = 305 \mu\text{m}$ .

##### 500 6.4.1. Effect of porosity on effective properties

501 The porosity-dependence of independent components of effective elasticity tensor  $\mathbf{C}^*$  and  
 502 Biot's tensor  $\boldsymbol{\alpha}$  is shown in Tab. 3. When the porosity increases, the *moduli* of the elasticity  
 503 tensor  $\mathbf{C}^*$  decrease, particularly the shear components. At the porosity of 96%, the porous  
 504 material nearly loses its shear resistance. This is attributed to the fact that when the  
 505 porosity increases, the thickness of the matrix ligaments accordingly decreases, leading to  
 506 the drop of the *moduli* of the porous material. On the contrary, the increase in the porosity  
 507 is followed by the increase in components of Biot's tensor  $\boldsymbol{\alpha}$ . This phenomenon is explained  
 508 by the fact that higher porosity facilitates an easier fluid flow, favouring the increase in  
 509 Biot's effective stress coefficients [37].

510 The dependence of the effective dynamic permeability and the effective dynamic com-  
 511 pressibility on the porosity is presented in Fig. 14. It can be seen that the increase in the  
 512 porosity leads to the increase in the effective permeability, especially at low frequencies.  
 513 This variation of the permeability with respect to the porosity is explained by the fact that  
 514 increasing porosity encourages more fluid travelling through pore space, implying the in-  
 515 crease in the permeability. Regarding the effective compressibility, this parameter tends to

Table 3: Independent components of effective elasticity tensor  $\mathbf{C}^*$  in the Voigt notations and Biot's tensor  $\boldsymbol{\alpha}$  in different cases of porosity  $\phi$ .

	$\phi$	69%	83%	96%
Components of tensor $\mathbf{C}^*$ ( $10^6$ Pa)	$C_{11}^*$	783.7	332.9	60.4
	$C_{44}^*$	117.1	21.0	0.6
	$C_{12}^*$	135.8	34.4	2.3
Component of tensor $\boldsymbol{\alpha}$	$\alpha_{11}$	0.92	0.97	1

516 decrease when the porosity increases in almost the whole considered frequency range. This  
517 trend can also be found in the experimental results for various rock types as shown in [38].

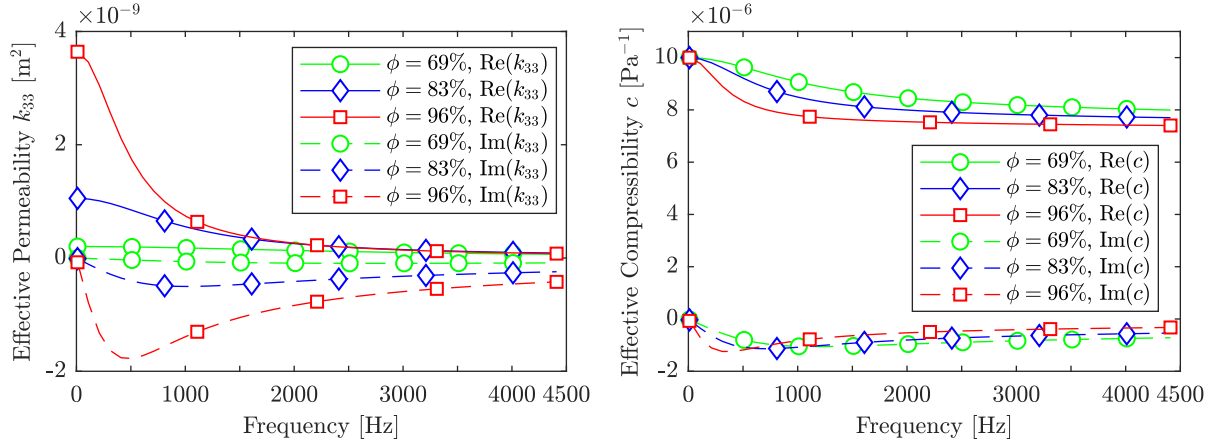


Figure 14: Dependence of the effective dynamic permeability (left) and the effective dynamic compressibility (right) on porosity.

#### 518 6.4.2. Effect of porosity on sound absorption

519 The effect of porosity on the sound absorption coefficient of the material is highlighted  
520 by Fig. 15. It can be remarked that the absorption coefficient increases slightly with respect  
521 to the rise of the porosity within the range of frequencies lower than approximately 1000 Hz,  
522 whereas the noticeable improvement on the sound absorption is accompanied by the decrease  
523 of the porosity for higher frequencies. In the process of making the porous material by using  
524 the Spark Plasma Sintering (SPS) technique, the resulting porosity depends on the pressure  
525 and temperature. Higher pressure and higher temperature normally induce a sintered poro-  
526 gen template with more porogen constituents in a volume than in case of a template resulting  
527 from a process with lower pressure and lower temperature [39, 15], meaning higher-porosity  
528 materials in case of higher pressure and higher temperature. The results of this section  
529 suggest that to promote a better sound absorption at frequencies lower than 1000 Hz, we

530 need to augment the pressure and the temperature during the SPS processing for a material  
 531 with more porosity. Inversely, for the range of higher frequencies, a decrease of pressure  
 532 and temperature should be applied during the SPS processing to reduce the porosity and  
 533 accordingly favour the sound absorption performance of the resulting porous material. Nev-  
 534 ertheless, the pressure and the temperature should not be too low in order to allow porogen  
 535 fusion and ensure the pore interconnection. This implies the existence of optimal pressure  
 536 and temperature during the SPS processing for attainment of interconnected-pore materials  
 537 with the best sound absorption performance at higher frequencies.

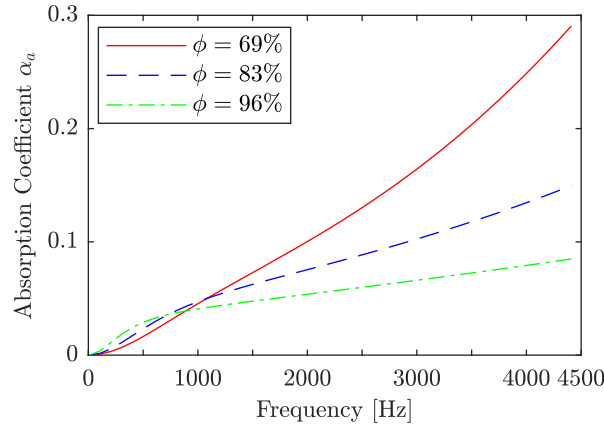


Figure 15: Dependence of sound absorption coefficient on porosity. The plate thickness is 0.5 cm.

### 538 6.5. Effect of pore size

539 In order to understand the relation between the pore size and effective properties as  
 540 well as the acoustic behaviour of the resulting material, simulations are conducted, in which  
 541 arrangement 1 is considered and the pore size is varied while maintaining the porosity at  
 542 83%.

#### 543 6.5.1. Effect of pore size on effective properties

544 In this study, the pore size is linked to the cell size. Within the framework of homoge-  
 545 nization method, the effective elasticity tensor  $\mathbf{C}^*$  and Biot's tensor  $\boldsymbol{\alpha}$  are modified by the  
 546 porosity, not by the cell size or pore size. Nevertheless, the change of pore size or cell size  
 547 influences both the effective dynamic permeability and effective dynamic compressibility as  
 548 shown in Fig. 16 where the variation of these properties with respect to the frequency corre-  
 549 sponding to three different pore sizes of 45, 350 and 710  $\mu\text{m}$  are presented. It can be noted  
 550 that at very low frequencies (inferior to 500 Hz), the larger the pore size, the higher the  
 551 permeability. This is explained by the fact that in case of a larger pore size, more entrance  
 552 of the fluid through the material is possible, favouring higher permeability. However, in the  
 553 range of higher frequencies, the permeabilities become lower and closer to each other except  
 554 that the permeability, in case of the smallest pore size, *i.e.*, 45  $\mu\text{m}$ , remains very low and  
 555 proves to be unchanged in the whole frequency range. Similarly to the permeability, the

556 compressibility remains nearly unchanged according to frequency in case of 45  $\mu\text{m}$  pore size.  
 557 On the contrary, the compressibility in cases of 350 and 710  $\mu\text{m}$  pore size becomes lower  
 558 and closer to each other when the frequency moves to the higher range. The very low values  
 559 of the imaginary parts of both permeability and compressibility at high frequencies imply  
 560 low viscous and thermal dissipation, hence low sound absorption of the material within this  
 561 frequency range.

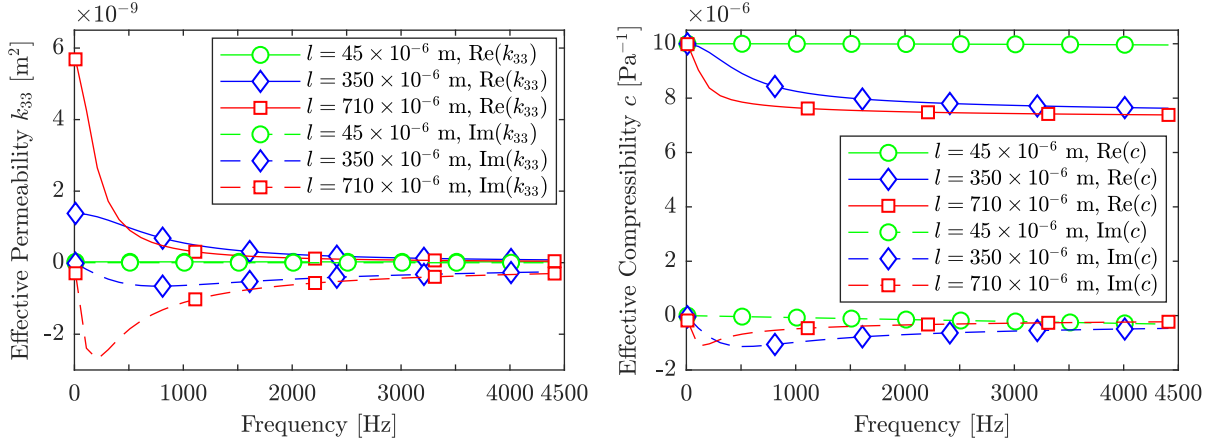


Figure 16: Dependence of the effective dynamic permeability (left) and the effective dynamic compressibility (right) on pore size. The porosity is fixed at  $\phi = 83\%$ .

### 562 6.5.2. Effect of pore size on sound absorption

According to the elaboration process, the pores are obtained by the porogen removal, so the pore size can be considered ideally equal to the porogen size, that is, NaCl particle size. Based on the sieve sizes available on the market for sieving NaCl particles, a variation of the pore size from 45 to 710  $\mu\text{m}$  with a step of 5  $\mu\text{m}$  is taken into account and the normal incidence sound absorption average SAA suggested in the standard ASTM C423 [40] is calculated for each case of the pore size to evaluate the sound absorption capacity of the material at low frequencies. This single number rating is defined as the average, rounded off to the nearest 0.01, of the sound absorption coefficients of a material for the twelve one-third octave bands from 200 through 2500 Hz inclusive, namely 200, 250, 315, 400, 500, 630, 800, 1000, 1250, 1600, 2000 and 2500 Hz according to the relation given by:

$$SAA = \frac{1}{12} \sum_{f_i=200}^{2500} \alpha_a(f_i). \quad (35)$$

563 Note that in this case, the associated wavelength varies in the range given by [0.338, 7.981] m.  
 564 Because the sound absorption of a porous material is generally greater at higher frequencies,  
 565 limiting the frequencies up to 2500 Hz is generally enough to investigate and evaluate the  
 566 sound absorption performance of the material.

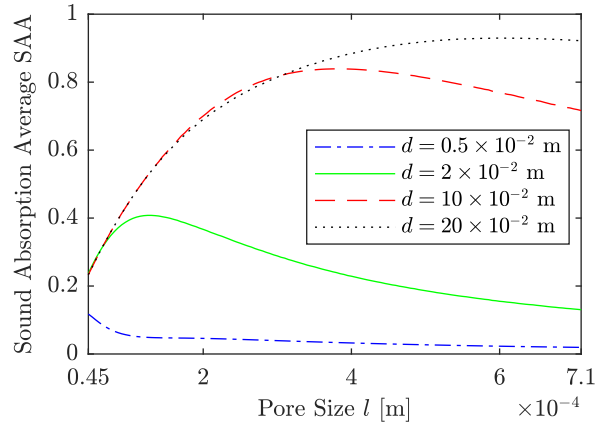


Figure 17: Normal incidence sound absorption average SAA for different cases of the pore size and the porous plate's thickness. The porosity is fixed at  $\phi = 83\%$ .

567 The SAA varies in the range of 0 to 1: SAA = 0 indicates a perfect sound reflection and  
568 SAA = 1 indicates a perfect sound absorption [41]. The use of the single number rating SAA  
569 is a convenient means to provide the insights into the acoustic behaviours of the material  
570 within a wide range of pore size and allows to compare different sound absorption perfor-  
571 mances corresponding to the pore size range of interest. The results of these simulations are  
572 presented in Fig. 17 where the variation of SAA for different cases of the pore size and the  
573 porous plate's thickness is illustrated.

574 It is noted that when the plate thickness is 0.5 cm, the SAA is relatively low with the  
575 maximum value of 0.12 at the pore size of 45  $\mu\text{m}$ . With this plate, when the pore size  
576 increases, the SAA generally follows a decreasing trend. This phenomenon is explained  
577 by the fact that the increase in the pore size encourages the enlargement of the cross-  
578 sectional area of the fluid phase and hence decreases the viscosity effect as well as the  
579 thermal dissipation, leading to the reduction in the sound absorption capability [18]. Note  
580 that for the configuration composed of the porous plate and the rigid backing, one of the key  
581 parameters is the resistance which is the product of the plate thickness by the static flow  
582 resistivity representing the difficulty of the propagation of sound in porous materials. The  
583 thinner the plate, the higher the resistivity, and inversely. In the present case, because the  
584 plate thickness is not much, the resistivity is generally high, explaining the weak absorption  
585 of the material.

586 When the plate becomes thicker, *i.e.* 2, 10 and 20 cm thickness, the SAA is noticeably  
587 improved. For the case of 2 cm thickness plate, the SAA attains its maximum value of 0.41  
588 at the pore size of 120  $\mu\text{m}$  while its minimum value is 0.13 at 710  $\mu\text{m}$  pore size. For 10  
589 and 20 cm thickness plates, the SAA reaches its maximum values of 0.84 and 0.93 at pore  
590 sizes of 365 and 600  $\mu\text{m}$ , respectively. This improvement in sound absorbing performance  
591 comes from the fact that the increase in the plate thickness makes the propagation of sound  
592 waves from the front to the back of the plate face more obstacles, meaning that more sound  
593 energy is attenuated. Furthermore, in these three cases of plate thickness, we can observe the

594 peaks of the pore size *versus* SAA curves. For the range of the pore size on the right-hand  
595 side of the peaks, the increase in the pore size is accompanied by the decrease of the SAA,  
596 whereas an inverse trend is observed on another side. The explanation of this phenomenon  
597 can be deduced by noting again that the principle of sound absorption of porous materials  
598 is that the material permits the propagation of sound waves and then the sound energy  
599 is attenuated inside the porous materials mainly by the viscous damping and the thermal  
600 dissipation. For the range of the pore size on the left-hand side of the peaks, the increase of  
601 the pore size leads to the entrance of more sound waves, and up to the values of the pore  
602 size corresponding to the peaks, the thickness of the plate as well as the viscous and thermal  
603 effects are still important to afford these entering sound waves, meaning that the more  
604 entering sound waves, the more attenuated sound energy. On the contrary, on the right-  
605 hand side of the peaks, when the pore size increases, the cross-section of the fluid phase  
606 proves to be too large, leading to the decrease of viscous damping and thermal dissipation  
607 and therefore resulting in less effective sound absorption for a given thickness. The existence  
608 of the above-mentioned peaks with two opposite trends is also well documented in [14] with  
609 Kelvin's cell (tetrakaidecahedron) for high porosity foams and in [42] for pores of cylindrical  
610 cross-section shape. It is also observed in Fig. 17 that the peak of the curve tends to occur  
611 at the higher pore size when the plate thickness increases. This trend of peak movement  
612 representing the correlation of the pore size and the plate thickness gives an idea of the  
613 material design. Because the pore size refers to the porogen size, based on Fig. 17, for a  
614 given porogen size (or a given plate thickness), we can choose an appropriate plate thickness  
615 (or an appropriate porogen size) to obtain the best sound absorbing performance.

## 616 7. Conclusion

617 In this work, we have undertaken essential numerical characterizations of effective me-  
618 chanical, hydraulic and acoustic properties of a new kind of bio-sourced porous epoxy resin.  
619 This novel material has been the product of an environmentally friendly process combin-  
620 ing delicately the cationic photopolymerization and the porogen leaching technique. The  
621 effective properties of the material have been obtained by conducting an asymptotic homog-  
622 enization procedure which has lead to macroscopic constitutive and governing equations of  
623 an anisotropic poroelastic medium which were first given by Biot. Different from Biot's  
624 theory, which deals with a phenomenological approach of the macroscopic behaviour of the  
625 poroelastic material, and as a consequence, there is no connection with the microstructure,  
626 the applied homogenization method has started from equations at the microscopic scale and  
627 has allowed a parametric investigation of the influences of material microstructures on the  
628 macroscopic properties.

629 In this study, homogenization calculations have been performed on representative vol-  
630 ume elements which have been based on the idealized fillet-edge cubic elementary pore and  
631 matrix obtained from the experimental pore characterization of the material. Following  
632 the determination of effective properties by the homogenization method, a normal incidence  
633 sound absorption problem has been considered with the use of a plate made of the elaborated  
634 material as a porous absorber. After validated by a good match between numerical acoustic

635 properties and experimental ones resulting from the three-microphone impedance tube test-  
636 ing, the sophisticated framework has been utilized to carry out a parametric study of the  
637 effect of the pore arrangements, the porosity and the pore size on the effective properties  
638 and acoustic performance of the material.

639 Based on the results of the parametric investigations, suggestions have been made for  
640 the attainment of porous epoxy resins with good sound absorption. Regarding pore ar-  
641 rangements, the results have indicated that if porogen particles could be ordered to make  
642 the template, the arrangements 2, 3 and 4 should be chosen rather than the arrangement 1  
643 to obtain a material with better sound absorption. Related to influences of the porosity,  
644 the paper has suggested that for better sound absorption at very low frequencies, the ma-  
645 terial would need to have more porosity which could be attained by raising the pressure  
646 and the temperature during the SPS process of the porogen template, whereas at higher  
647 frequencies, the less-porosity material obtained from the decrease in the pressure and the  
648 temperature during the SPS process without losing the pore interconnection should be  
649 used. It is worth reminding that improving the sound absorption at lower frequencies by  
650 increasing the porosity leads to the material with lower stiffness. Therefore, for a specific  
651 application of the material such as in civil engineering, there needs a compromise between  
652 the mechanical and acoustic properties of the material. Conversely, when a reduction in  
653 pressure and temperature is applied to obtain a less-porous and accordingly better sound  
654 absorbing material at higher frequencies, both of these two processing parameters should  
655 not be decreased too much to avoid the loss of contacts among porogen particles, assuring  
656 the availability of sintered porogen templates. Concerning pore size's effects, the obtained  
657 pore size *versus* SAA relationships corresponding to different plate thicknesses can play the  
658 role of a practical guide for material elaboration processing because the pore size also refers  
659 to the porogen size. From these curves, for a given porogen size (or a given plate thickness),  
660 we can choose an appropriate plate thickness (or an appropriate porogen size) to obtain the  
661 material with the best sound absorbing performance.

662 Regarding the experimental aspect, additional systematic experiments are needed to  
663 identify the lowest porosity and the corresponding critical values of pressure and temperature  
664 for ensuring the attainment of sintered templates and hence porous materials. This effort  
665 will help to frame the possible range of porosity of porous materials that result from the  
666 application of the salt leaching technique. Furthermore, in order to favour the wideband  
667 sound absorption of the materials for more potential applications, another attention is paid  
668 to the development of multiple-porosity materials such as doubly porous ones [43]. Within  
669 the context of the porogen leaching technique, besides macropores obtained by using salt  
670 particles as macro-porogens, micropores can be generated by the introduction of porogenic  
671 solvents to the formulation [2]. However, because the porogen leaching technique is employed  
672 in conjunction with the cationic photopolymerization which is solvent-sensitive, the choice  
673 of solvents that are a little harmful and compatible with the cationic photopolymerization  
674 would be an interesting challenge.

675 Concerning the numerical part, the multiscale approach employed in this work concern-  
676 ing the use of ordered microstructures has proved to be useful in the material development  
677 stage for obtaining a first estimate of the main physical properties of the real samples as



678 well as for systematic investigations into the relations between microstructures and effective  
679 properties of the studied material, providing valuable suggestions for the material improve-  
680 ment. Nonetheless, it is likely that deeper insights into the effective material's properties  
681 could be obtained by including a certain degree of randomness in the orientation and dis-  
682 tribution of the pores [44], which will be the topic of a further study. Additionally, for  
683 the case of doubly porous materials as above-mentioned, an adapted three-scale asymptotic  
684 homogenization procedure would be of interest to deal with the simultaneous presence of  
685 macropores and micropores resulting from the use of solid macro-porogens and porogenic  
686 solvents, respectively, which exhibit two distinct populations in terms of pore size, pore  
687 shape, pore arrangement and porosity.

## Data availability

The processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

## Acknowledgements

This work has benefited from a French government grant managed by ANR within the frame of the national program of Investments for the Future ANR-11-LABX-0022-01 (LabEx MMCD project).

## References

- [1] Q.-B. Nguyen, N.-H. Nguyen, A. Rios de Anda, V.-H. Nguyen, D.-L. Versace, V. Langlois, S. Naili, E. Renard, Photocurable bulk epoxy resins based on resorcinol derivative through cationic polymerization, *Journal of Applied Polymer Science* (2020) e49051.
- [2] H.-B. Ly, B. Le Droumaguet, V. Monchiet, D. Grande, Designing and modeling doubly porous polymeric materials, *The European Physical Journal Special Topics* 224 (9) (2015) 1689–1706.
- [3] Y. Nam, T. Park, Biodegradable polymeric microcellular foams by modified thermally induced phase separation method, *Biomaterials* 20 (1999) 1783–1790.
- [4] M. Silverstein, N. Cameron, PolyHIPEs - Porous polymers from high internal phase emulsions, in: John Wiley & Sons, Inc. (Ed.), *Encyclopedia of Polymer Science and Technology*, John Wiley & Sons, Inc., Hoboken, NJ, USA, 2010.
- [5] Q. Qi, P. Zheng, Y. Lei, X. Liu, Design of bi-modal pore structure polyarylene ether nitrile/sio2 foams with ultralow-k dielectric and wave transparent properties by supercritical carbon dioxide, *Composites Part B: Engineering* 173 (2019) 106915.
- [6] G. Chen, T. Ushida, T. Tateishi, Preparation of poly(l-lactic acid) and poly(dl-lactic-co-glycolic acid) foams by use of ice microparticulates, *Biomaterials* 22 (18) (2001) 2563–2567.
- [7] K. Rezwan, Q. Chen, J. Blaker, A. Boccaccini, Biodegradable and bioactive porous polymer/inorganic composite scaffolds for bone tissue engineering, *Biomaterials* 27 (18) (2006) 3413–3431.
- [8] Q. Tann, S. Li, J. Ren, C. Chen, Fabrication of porous scaffolds with a controllable microstructure and mechanical properties by porogen fusion technique, *International Journal of Molecular Sciences* 12 (2) (2011) 890–904.
- [9] J. L. Auriault, C. Boutin, C. Geindreau, *Homogenization of Coupled Phenomena in Heterogenous Media*, Wiley-ISTE, 2009.
- [10] A. Mielke, E. Rohan, Homogenization of elastic waves in fluid-saturated porous media using the biot model, *Mathematical Models and Methods in Applied Sciences* 23 (5) (2013) 873–916.

- [11] R. Del Toro, A. Bacigalupo, M. Paggi, Characterization of wave propagation in periodic viscoelastic materials *via* asymptotic-variational homogenization, *International Journal of Solids and Structures* 172-173 (2019) 110–146.
- [12] M. De Bellis, A. Bacigalupo, G. Zavarise, Characterization of hybrid piezoelectric nanogenerators through asymptotic homogenization, *Comput. Methods Appl. Mech. Engrg.* 355 (2019) 1148–1186.
- [13] L. Maheo, P. Viot, D. Bernard, A. Chirazi, G. Ceglia, V. Schmitt, O. Mondain-Monval, Elastic behavior of multi-scale, open-cell foams, *Composites Part B: Engineering* 44 (1) (2013) 172–183.
- [14] F. Chevillotte, C. Perrot, Effect of the three-dimensional microstructure on the sound absorption of foams: A parametric study, *The Journal of the Acoustical Society of America* 142 (2) (2017) 1130–1140.
- [15] A. Azarniya, A. Azarniya, M. Safavi, M. Ahmadipour, M. Seraji, S. Sovizi, M. Saqaei, R. Yamanoglu, M. Soltaninejad, H. Hosseini, S. Ramakrishna, A. Kawasaki, S. Adams, M. Reddy, Physicomechanical properties of porous materials by Spark Plasma Sintering, *Critical Reviews in Solid State and Materials Sciences* (2019) 1–44.
- [16] H. Giesche, Mercury porosimetry: A general (practical) overview, *Particle & Particle Systems Characterization* 23 (1) (2006) 9–19.
- [17] E. Rohan, S. Naili, R. Cimirman, T. Lemaire, Multiscale modeling of a fluid saturated medium with double porosity: Relevance to the compact bone, *Journal of the Mechanics and Physics of Solids* 60 (5) (2012) 857 – 881.
- [18] T. Yamamoto, S. Maruyama, K. Terada, K. Izui, S. Nishiwaki, A generalized macroscopic model for sound-absorbing poroelastic media using the homogenization method, *Computer Methods in Applied Mechanics and Engineering* 200 (1-4) (2011) 251–264.
- [19] E. Sanchez-Palencia, *Non-Homogeneous Media and Vibration Theory*, no. 127 in *Lecture Notes in Physics*, Springer, Berlin, 1980.
- [20] J.-L. Auriault, L. Borne, R. Chambon, Dynamics of porous saturated media, checking of the generalized law of Darcy, *The Journal of the Acoustical Society of America* 77 (5) (1985) 1641–1650.
- [21] V.-H. Nguyen, E. Rohan, S. Naili, Multiscale simulation of acoustic waves in homogenized heterogeneous porous media with low and high permeability contrasts, *International Journal of Engineering Science* 101 (2016) 92–109.
- [22] J.-F. Allard, N. Atalla, *Propagation of Sound in Porous Media: Modelling Sound Absorbing Materials*, 2nd Edition, Wiley, 2009.
- [23] R. Panneton, X. Olny, Acoustical determination of the parameters governing viscous dissipation in porous media, *The Journal of the Acoustical Society of America* 119 (4) (2006) 2027–2040.
- [24] X. Olny, R. Panneton, Acoustical determination of the parameters governing thermal dissipation in porous media, *The Journal of the Acoustical Society of America* 123 (2) (2008) 814–824.
- [25] COMSOL Multiphysics, COMSOL AB, Stockholm, Sweden (2019).
- [26] K. Gross, L. Rodríguez-Lorenzo, Biodegradable composite scaffolds with an interconnected spherical network for bone tissue engineering, *Biomaterials* 25 (20) (2004) 4955–4962.
- [27] V.-H. Nguyen, S. Naili, Semi-analytical solution of transient plane waves transmitted through a transversely isotropic poroelastic plate immersed in fluid, *Journal of Engineering Mathematics* 86 (1) (2014) 125–138. doi:10.1007/s10665-013-9654-5.
- [28] N. Gorbushin, S. Naili, V.-H. Nguyen, Optimizing microstructure of a poroelastic layer with cylindrical pores for absorption properties, *Mechanics Research Communications* 102 (2019) 103422.
- [29] N. Gorbushin, V.-H. Nguyen, S. Naili, Design optimisation of acoustic absorbers with cross-like pores *via* a homogenisation method, *Acta Acustica united with Acustica* 105 (2019) 638–649.
- [30] H. Kuttruff, *Acoustics: An Introduction*, Taylor & Francis, 2007.
- [31] C. Perrot, F. Chevillotte, R. Panneton, Bottom-up approach for microstructure optimization of sound absorbing materials, *The Journal of the Acoustical Society of America* 124 (2) (2008) 940–948.
- [32] Y. Salissou, R. Panneton, Wideband characterization of the complex wave number and characteristic impedance of sound absorbers, *The Journal of the Acoustical Society of America* 128 (5) (2010) 2868–2876.
- [33] D. Johnson, J. Koplik, L. Schwartz, New pore-size parameter characterizing transport in porous media,

- Phys. Rev. Lett. 576 (1986) 2564–2567.
- [34] D. L. Johnson, J. Koplik, R. Dashen, Theory of dynamic permeability and tortuosity in fluid-saturated porous media, *Journal of Fluid Mechanics* 176 (1987) 379.
  - [35] Y. Champoux, J.-F. Allard, Dynamic tortuosity and bulk modulus in air-saturated porous media, *Journal of Applied Physics* 70 (4) (1991) 1975–1979.
  - [36] D. Lafarge, P. Lemarinier, J. F. Allard, V. Tarnow, Dynamic compressibility of air in porous structures at audible frequencies, *The Journal of the Acoustical Society of America* 102 (4) (1997) 1995–2006.
  - [37] X. Luo, P. Were, J. Liu, Z. Hou, Estimation of Biot’s effective stress coefficient from well logs, *Environmental Earth Sciences* 73 (11) (2015) 7019–7028.
  - [38] R. Baker, H. Yarranton, J. Jensen, *Practical Reservoir Engineering and Characterization*, Elsevier, 2015.
  - [39] L. Zhang, Y. Zhang, Y. Jiang, R. Zhou, Mechanical behaviors of porous Ti with high porosity and large pore size prepared by one-step spark plasma sintering technique, *Vacuum* 122 (2015) 187–194.
  - [40] ASTM International, Standard test method for sound absorption and sound absorption coefficients by the reverberation room method, *American Society for Testing and Materials* (2002).
  - [41] M. Zainulabidin, M. Rani, N. Nezere, A. Tobi, Optimum sound absorption by materials fraction combination, *International Journal of Mechanical & Mechatronics Engineering* 14 (02) (2014) 4.
  - [42] G. Kirchhoff, On the influence of heat conduction in a gas on sound propagation, R.B. Lindsay, Ed., *Physical Acoustics* (1974) 7–19.
  - [43] E. Rohan, V.-H. Nguyen, S. Naili, Numerical modelling of waves in double-porosity Biot medium, *Computers and Structures* 232 (2020) 105849.
  - [44] C. Nguyen, J. Guilleminot, F. Detrez, V. Langlois, M. Bornert, A. Duval, C. Perrot, Micro-macro acoustic modeling of heterogeneous foams with nucleation perturbation, in: 11th International Styrian Noise, Vibration & Harshness Congress: The European Automotive Noise Conference, SAE International, 2020.