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

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Abstract

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

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

26 1. Introduction

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

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

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

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

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

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

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

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

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

118 2. Material elaboration and characterization

119 2.1. Elaboration process using porogen leaching technique

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

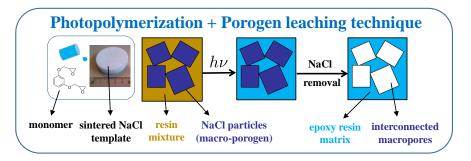


Figure 1: Schema of porogen leaching technique using NaCl particles as macro-porogen.

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

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

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

162 2.2. Experimental pore characterization

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

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

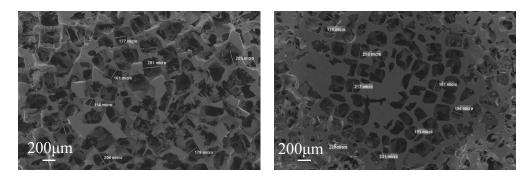


Figure 2: SEM micrographs: (left) on cross-section; (right) on external surface

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

187 3. Homogenization and effective properties

In what follows, the Cartesian reference system is used in which the orthonormal basis 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 time coordinate is denoted by t. The time derivative is denoted by a superimposed dot. Einstein's summation convention about the repeated indices is used. The imaginary pure number denoted by i is defined by $i^2 = -1$. All fields are written in the frequency domain, considering a harmonic regime with functions proportional to $\exp(i\omega t)$ where ω is the angular frequency.

195 3.1. Two-scale asymptotic homogenization procedure

The details of the two-scale asymptotic homogenization procedure, the derivation of cell problems and macroscopic state equations can be found, for instance, in [17] and [18]. The 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 η 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 Γ^{sf} (see Fig. 3):

$$Y = Y^s \cup Y^f \cup \Gamma^{sf}, \qquad Y^s \cap Y^f = \emptyset, \qquad \Gamma^{sf} = \partial Y^s \cap \partial Y^f.$$
(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:

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

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

where ρ^s, u_i^s and σ_{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 ε_{kl}^s are the components of the infinitesimal strain which read:

$$\varepsilon_{kl}^{s} = \frac{1}{2} \left(\frac{\partial u_{k}^{s}}{\partial x_{l}} + \frac{\partial u_{l}^{s}}{\partial x_{k}} \right)$$
(3)

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

$$\rho^f \frac{\partial v_i^f}{\partial x_i} + \mathrm{i}\omega \delta^f = 0 \qquad \text{within } Y^f, \tag{4a}$$

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

where p^f and v_i^f are respectively the pressure and the components of the velocity of the fluid phase; P^f , ρ^f and T^f are respectively the pressure, the density and the temperature of the fluid phase in the rest state; δ^f and τ^f are respectively the perturbations of the density and 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:

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

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

$$\sigma_{ij}^f = 2\eta \dot{\varepsilon}_{ij}^f - \frac{2}{3}\eta \dot{\varepsilon}_{kk}^f \delta_{ij} - p^f \delta_{ij}, \tag{6a}$$

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

²⁰⁵ where δ_{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 κ_{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:

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

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

For an ideal gas, Mayer's relation is valid and is given by the relation $C_P^f - C_V^f = R_g$, where C_P^f is the specific heat under constant pressure. It is also convenient to introduce the 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 Γ^{sf} of the solid and fluid phases for the normal stress, the velocity and the temperature are given as follows:

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

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

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

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

211 3.1.2. Scaling and asymptotic expansions

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

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

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

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

$$u_{i}^{s} = u_{i}^{s(0)}(\boldsymbol{x}, \boldsymbol{y}) + \epsilon u_{i}^{s(1)}(\boldsymbol{x}, \boldsymbol{y}) + \epsilon^{2} u_{i}^{s(2)}(\boldsymbol{x}, \boldsymbol{y}) + \dots,$$

$$v_{i}^{f} = v_{i}^{f(0)}(\boldsymbol{x}, \boldsymbol{y}) + \epsilon v_{i}^{f(1)}(\boldsymbol{x}, \boldsymbol{y}) + \epsilon^{2} v_{i}^{f(2)}(\boldsymbol{x}, \boldsymbol{y}) + \dots,$$

$$p^{f} = p^{f(0)}(\boldsymbol{x}, \boldsymbol{y}) + \epsilon p^{f(1)}(\boldsymbol{x}, \boldsymbol{y}) + \epsilon^{2} p^{f(2)}(\boldsymbol{x}, \boldsymbol{y}) + \dots,$$

$$\tau^{f} = \tau^{f(0)}(\boldsymbol{x}, \boldsymbol{y}) + \epsilon \tau^{f(1)}(\boldsymbol{x}, \boldsymbol{y}) + \epsilon^{2} \tau^{f(2)}(\boldsymbol{x}, \boldsymbol{y}) + \dots,$$

$$\delta^{f} = \delta^{f(0)}(\boldsymbol{x}, \boldsymbol{y}) + \epsilon \delta^{f(1)}(\boldsymbol{x}, \boldsymbol{y}) + \epsilon^{2} \delta^{f(2)}(\boldsymbol{x}, \boldsymbol{y}) + \dots,$$
(11)

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

222 3.1.3. Cell problems

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

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

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

$$\int_{Y^s} \chi_p^{kl}(\boldsymbol{y}) \mathrm{d}V = 0. \tag{12b}$$

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

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

$$\int_{Y^s} \beta_k(\boldsymbol{y}) \mathrm{d}V = 0. \tag{13b}$$

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

$$\int_{Y^{f}} \rho^{f} \mathrm{i}\omega\xi_{i}^{k}(\boldsymbol{y})\delta\xi_{i}^{k}(\boldsymbol{y})\mathrm{d}V + \int_{Y^{f}} \eta \frac{\partial\xi_{i}^{k}(\boldsymbol{y})}{\partial y_{j}} \frac{\partial\delta\xi_{i}^{k}(\boldsymbol{y})}{\partial y_{j}} \mathrm{d}V$$
$$- \int_{Y^{f}} \frac{\partial\delta\xi_{i}^{k}(\boldsymbol{y})}{\partial y_{i}} \pi^{k}(\boldsymbol{y})\mathrm{d}V = \int_{Y^{f}} \delta\xi_{i}^{k}(\boldsymbol{y})\mathrm{d}V, \qquad \forall\delta\xi_{i}^{k}(\boldsymbol{y}) \in \mathcal{C}_{\#}(Y^{f}), \quad (14\mathrm{a})$$

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

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

$$\int_{Y^f} \pi^k(\boldsymbol{y}) \mathrm{d}V = 0.$$
 (14d)

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

$$\int_{Y^{f}} \frac{1}{\mathrm{i}\omega\rho^{f}C_{p}^{f}} \kappa_{ij}^{f} \frac{\partial \zeta(\boldsymbol{y})}{y_{j}} \frac{\partial \delta\zeta(\boldsymbol{y})}{\partial y_{i}} \mathrm{d}V + \int_{Y^{f}} \zeta(\boldsymbol{y})\delta\zeta(\boldsymbol{y})\mathrm{d}V$$
$$= \int_{Y^{f}} \delta\zeta(\boldsymbol{y})\mathrm{d}V, \qquad \forall \delta\zeta(\boldsymbol{y}) \in \mathcal{C}_{\#}(Y^{f}) \quad (15a)$$
$$\zeta(\boldsymbol{y}) = 0 \qquad \text{on } \Gamma^{sf}. \tag{15b}$$

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

230 3.1.4. Effective properties and macroscopic governing equations

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

$$\sigma_{ij} = C^*_{ijkl} \varepsilon^s_{kl}(\boldsymbol{x}) - \alpha_{ij} p^f(\boldsymbol{x}), \qquad (16a)$$

$$p^{f}(\boldsymbol{x}) = -M\left[\frac{1}{\mathrm{i}\omega}\frac{\partial w_{i}}{\partial x_{i}} + \alpha_{ij}\varepsilon_{ij}^{s}(\boldsymbol{x})\right]$$
(16b)

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

$$C_{ijkl}^{*} = \left\langle c_{ijpq}^{s} \left(\frac{\partial \chi_{p}^{kl}(\boldsymbol{y})}{\partial y_{q}} + \delta_{pk} \delta_{ql} \right) \right\rangle, \qquad (17)$$

$$\alpha_{ij} = \phi \delta_{ij} + B_{ij}, \quad B_{ij} = \left\langle c^s_{ijkl} \frac{\partial \beta_k(\boldsymbol{y})}{\partial y_l} \right\rangle = -\left\langle \frac{\partial \chi^{ij}_k(\boldsymbol{y})}{\partial y_k} \right\rangle, \tag{18}$$

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

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

$$\langle * \rangle = \frac{1}{|Y|} \int_{Y} * \,\mathrm{d}V \tag{20}$$

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:

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

The macroscopic governing equations can be written as:

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

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

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:

$$H_{ik} = \left\langle \xi_i^k(\boldsymbol{y}) \right\rangle. \tag{23}$$

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

$$k_{ik} = \eta H_{ik}.\tag{24}$$

At the limit $K^f/K^s \to 0$ 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 ρ_{eq} and the equivalent dynamic bulk modulus K_{eq} [18, 22]. Note that ρ_{eq} is a second-order tensor. These two equivalent parameters can respectively be expressed in terms of the effective dynamic mass density ρ_{eff}^f [23] and the effective dynamic bulk modulus K_{eff}^f [24] of the fluid phase as follows:

$$\boldsymbol{\rho}_{eq} = \frac{1}{\phi} \boldsymbol{\rho}_{eff}^{f}, \qquad K_{eq} = \frac{K_{eff}^{f}}{\phi}.$$
(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 $\xi(\boldsymbol{y})$ as:

$$\rho_{eff,ik}^{f} = \frac{\phi}{\mathrm{i}\omega} \left\langle \xi_{i}^{k}(\boldsymbol{y}) \right\rangle^{-1}.$$
(26)

231 3.2. Numerical implementation

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

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

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

246 4. Construction of idealized representative volume elements

247 4.1. Construction of idealized elementary pore and matrix

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

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

274 4.2. Construction of representative volume elements

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

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

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

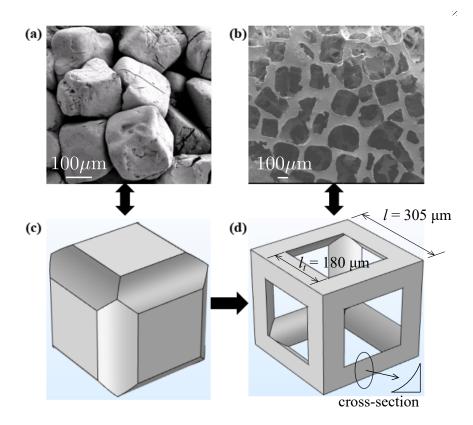


Figure 4: Construction of the elementary pore and matrix by the idealized periodic microstructure. (a) SEM image of sintered NaCl particles; (b) SEM image of the present porous material; (c) Elementary cubic pore; (d) Elementary cubic matrix.

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

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 $\boldsymbol{x} = (x_1, x_2, x_3)$. Because the study focuses on the

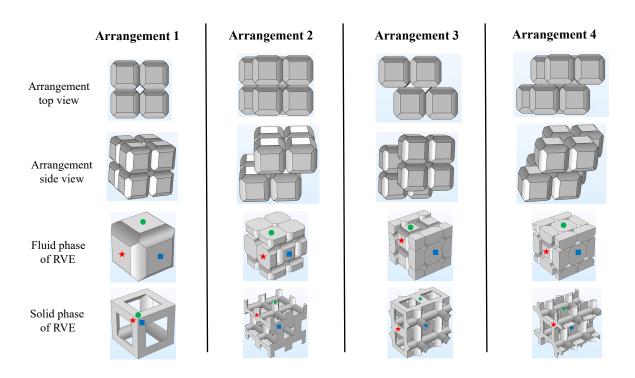


Figure 5: Representative volume elements of the four pore arrangements. The star-, square- and circlemarked surfaces correspond to the surface orthogonal to the incidence direction of the plane pressure described in section 5.

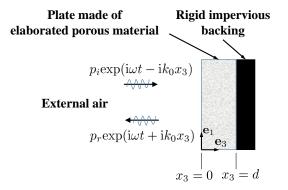


Figure 6: Normal incidence acoustic problem for a plate of thickness d made of the elaborated porous material.

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:

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

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

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

where $Z_0 = \rho_0 c_0$ is the characteristic impedance of the air with ρ_0 the density of the air at rest and c_0 the sound velocity in the air. Subsequently, the absorption coefficient α_a is determined by [30]:

$$\alpha_a(\omega) = 1 - |R|^2. \tag{30}$$

304 6. Numerical results and discussion

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

310 6.1. Effect of pore arrangements on effective properties

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

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

In the case of arrangement 1, the numerical results of effective elasticity tensor C^* in the Voigt notations and Biot's tensor α are as follows:

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

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

Regarding frequency-dependent properties, namely Biot's modulus M, the effective compressibility c and the effective dynamic permeability tensor k, their values at frequency of 10 Hz read:

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

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

$$\boldsymbol{k} = \begin{bmatrix} 1.05 \times 10^{-9} & 2.06 \times 10^{-13} & 8.71 \times 10^{-13} \\ & 1.05 \times 10^{-9} & 9.68 \times 10^{-13} \\ & & 1.05 \times 10^{-9} \end{bmatrix}$$
(33)

$$\begin{bmatrix} \text{sym.} & 1.05 \times 10^{-9} \end{bmatrix} \\ -\text{i} \begin{bmatrix} 1.01 \times 10^{-11} & 2.42 \times 10^{-15} & 1.25 \times 10^{-14} \\ & 1.01 \times 10^{-11} & 1.39 \times 10^{-14} \\ \text{sym.} & 1.01 \times 10^{-11} \end{bmatrix} (\text{m}^2)$$
(34)

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

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

17

321

		Pore arrangements					
		1	2	3	4		
Components of tensor 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	$\operatorname{orthotropic}$	orthotropic		
Components	α_{11}	0.969	0.965	0.959	0.965		
of	of α_{22}		0.966	0.980	0.966		
tensor $\boldsymbol{\alpha}$ α_{33}		0.969	0.977	0.959	0.966		

Table 1: Non-zero components of effective elasticity tensor C^* in the Voigt notations and Biot's tensor α in different cases of pore arrangements.

In Figs. 7 and 8, we present respectively results of the effective dynamic permeability and 347 effective dynamic compressibility. The absorption characteristics of the considered porous 348 materials are quantified using the complex values of these two parameters. The presence 349 of components of the permeability tensor in Fig. 7 indicates the properties of this tensor 350 and reflects the symmetry type corresponding to each arrangement. For instance, in the 35 case of arrangement 1, the permeability tensor is spherical for which $k_{11} = k_{22} = k_{33}$, 352 implying the cubic symmetry, whereas for arrangement 4, the tensor has a diagonal form 353 with 2 independent components, representing the transversely isotropy. Comparing the 354 permeability results for arrangement 1 with those for other arrangements, one can notice 355 that while arrangements 2, 3 and 4 have the permeability component versus frequency 356 curves with similar corresponding patterns, the curves for arrangement 1 follow their own 357 specific patterns and there is a difference of at least 10 times between the permeabilities 358 for arrangement 1 and those for the others. In addition, regarding the two arrangements 359 corresponding to the transverse isotropy, an attention is also paid to the fact that the 360 permeability component related to the axis of rotation symmetry has the lowest absolute 361 value compared with two other components in case of arrangement 3 and inversely has the 362 highest absolute value in case of arrangement 4. Regarding compressibility, Fig. 8 shows that 363 the results for the three arrangements 2, 3 and 4 prove to be similar, while being slightly 364 different from the compressibility for arrangement 1. This reveals that the arrangements 365 have not much influence on the compressibility of the materials. 366

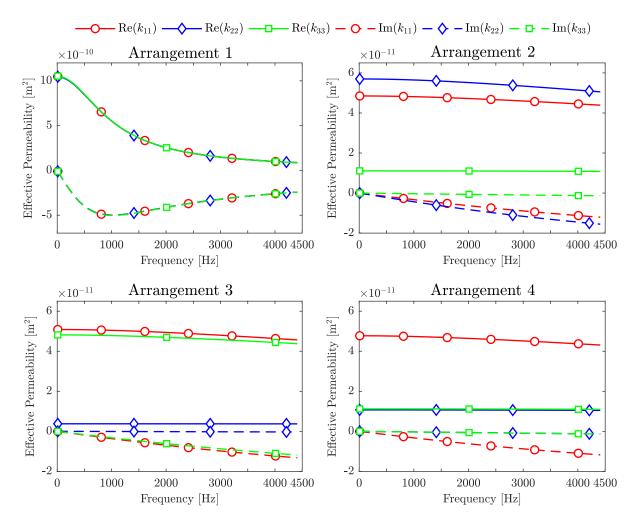


Figure 7: Dependence of non-zero components of the effective dynamic permeability tensor on different pore arrangements.

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

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

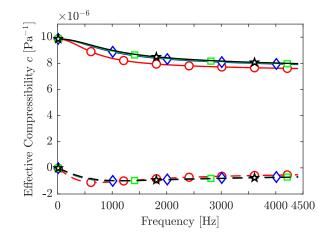


Figure 8: Dependence of the effective dynamic compressibility on different pore arrangements. Solid lines with circular, diamond, square and star markers represent respectively real parts of compressibility in case of arrangements 1, 2, 3 and 4. Dashed lines with circular, diamond, square and star markers 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 380 directions; inversely, the dominance of sound absorption is recorded at high frequencies in 38: directions corresponding to a larger permeability. The fact that the compressibility values 382 are similar for these three cases implies that the permeability, not the compressibility, is 383 the key parameter resulting in the different sound absorption behaviours related to different 384 placements of unit cells. Regarding arrangement 1 and in the frequency range of interest, 385 one may notice the smallness of the sound absorption coefficient in this case, compared with 386 those in other arrangements in spite of the fact that the permeability for this arrangement 387 is much higher than those for other configurations. Normally, the increase in the permeabil-388 ity implies the entrance of more sound waves and hence the possibility of more attenuated 389 sound energy. This result has to be relativized since it is generally valid for relatively large 390 thicknesses. Nonetheless, besides higher permeability, the microstructure of the material 391 resulting from arrangement 1 has also a larger pore entrance dimension compared to those 392 for other configurations. This leads to the decrease of viscous damping and thermal dis-393 sipation, and therefore, results in the less effective sound absorption. This phenomenon is 394 also observed in some parametric studies carried out in two dimensions for fibrous media 395 [31] as well as in three dimensions for foams [14]. Because the pore arrangement duplicates 396 the arrangement of porogen particles, we can link the porogen arrangement and the sound 397 absorption. Although the porogen arrangement is not controlled yet in the present elabora-398 tion processing, the results of this section suggest that the arrangement of porogen particles 399 should fall into types 2, 3 or 4 for the possibly highest sound absorption. 400

401 6.3. Experimental validation

402 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

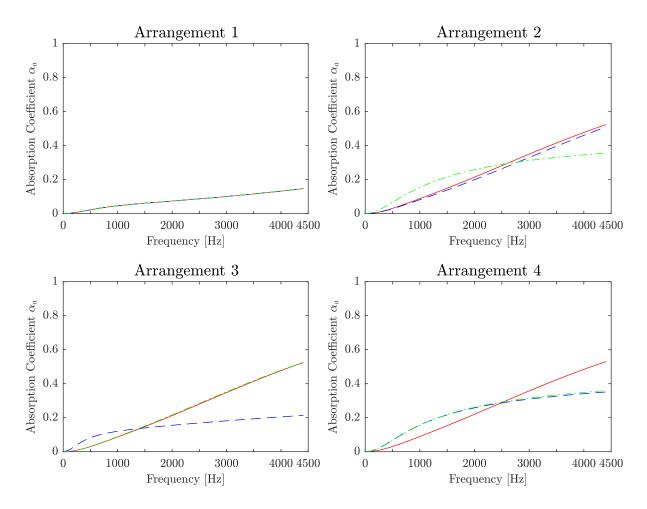


Figure 9: Dependence of sound absorption coefficients on different pore arrangements. The plate thickness is 0.5 cm. Solid, dashed and dashed-dot lines correspond to cases in which star-, square- and circle-marked surfaces of representative volume elements are orthogonal to the normal incidence direction,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

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

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

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

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

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

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

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

In Fig. 12, the maximum value of relative standard deviation of α_a is about 15.9%.

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

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

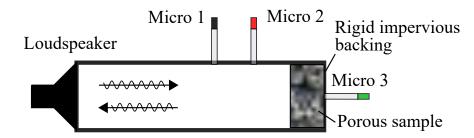


Figure 10: Sketch of experimental setup of three-microphone impedance tube experiment.

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

The comparison is also carried out in terms of the equivalent dynamic density and the 444 equivalent dynamic bulk modulus (see Fig. 13). A good agreement between model and 445 experimental results is figured out for these two parameters in the frequency range higher 446 than 100 Hz. When the frequency is lower than this value, there are fluctuations in the ex-447 perimental values of both parameters and accordingly the model's results deviate from the 448 experimental ones. The divergences observed for these two equivalent parameters, but also 449 for the surface impedance, may be attributed to a lack of accuracy due to edge constraints 450 on small samples. Additionally, the mismatch in the frequency range lower than 100 Hz may 45:

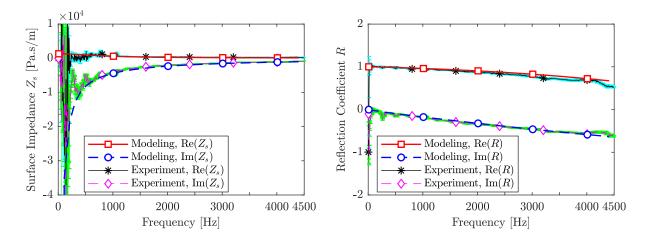


Figure 11: Comparison of model and experimental results of surface impedance Z_s (left) and normal incidence reflection coefficient R (right). The sample thickness is 0.5 cm. The green and cyan colours are used for the standard deviation.

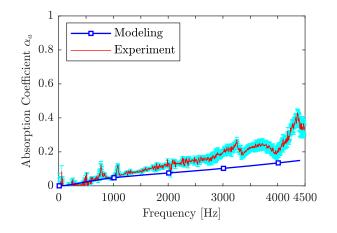


Figure 12: Comparison of model and experimental results of normal incidence absorption coefficient α_a (see eq. (30)). The sample thickness is 0.5 cm. The green colour is used for the standard deviation.

⁴⁵² be explained by the dependence of the three-microphone impedance tube testing on the up⁴⁵³ 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

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

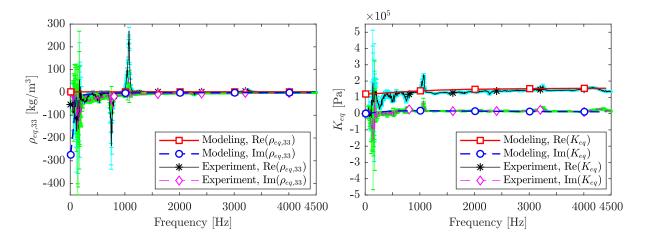


Figure 13: Comparison of numerical and experimental results of equivalent dynamic density component $\rho_{eq,33}$ (left) and equivalent dynamic bulk modulus K_{eq} (right). The sample thickness is 0.5 cm. The green and cyan colours are used for the standard deviation.

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

It should be noted that the viscous characteristic length Λ [33] is a pore-volume-to-surface 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.

	ϕ	$\Lambda \ (\mu m)$	$k \ (\mathrm{m}^2)$
Simulation	0.83	112	10.1×10^{-10}
Experiment	0.83 ± 0.02	36 ± 23	$(10.1 \pm 3.0) \times 10^{-10}$
	α_{∞}	$\Lambda' \ (\mu m)$	$k' (\mathrm{m}^2)$
Simulation	$\frac{\alpha_{\infty}}{1.23}$	$\frac{\Lambda' \ (\mu m)}{203}$	$\frac{k' (m^2)}{22.6 \times 10^{-10}}$

468

⁴⁶⁹ Consequently, the quantity Λ 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 Λ 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 α_{∞} [34] is in agreement with the one actually computed. The thermal charac-⁴⁷⁵ teristic length Λ' [35] is another factor like Λ above, but without weighting and is therefore

a simple length scale that characterizes, in some way, the pore diameter. Comparing the 476 numerical and experimental values of the thermal characteristic length Λ' , it can be seen 477 that the experimental value of the effective pore diameter is lower than the one given by the 478 idealized model. Figures 4a) and 4b) show a surface roughness increasing the surface area of 479 the pore-solid interface, which has the effect of decreasing the overall pore-volume-to-surface 480 ratio. We note that the other measured macroscopic parameters, namely the porosity ϕ and 48 the static viscous permeability k, are in agreement with the computed ones. Also worth 482 being noted, the characterized static thermal permeability k' [36], which should satisfy the 483 inequality $k' \geq k$, is close to the value of the static viscous one. Johnson et al. [34] and 484 Lafarge *et al.* [36] showed that the equivalent dynamic density $\rho_{eq,33}(\omega)$ and the equiva-485 lent dynamic bulk modulus $K_{eq}(\omega)$ can be adequately described by approximate but robust 486 semi-phenomenological models based on these macroscopic parameters $\phi, \Lambda', k, \alpha_{\infty}, \Lambda$ and 487 k'. The better agreement between the numerically characterized and measured behaviour 488 of the sound absorption is indicative of the fact that the periodic unit cell captures most 489 of the physics of the visco-thermal exchanges, but that a more accurate representation of 490 the high frequency behaviour could eventually be obtained by introducing a disorder in the 491 pore space. 492

493 6.4. Effect of porosity

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

500 6.4.1. Effect of porosity on effective properties

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

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

	ϕ	69%	83%	96%
Components of	C_{11}^{*}	783.7	332.9	60.4
tensor C^*	C_{44}^{*}	117.1	21.0	0.6
(10^6 Pa)	C_{12}^{*}	135.8	34.4	2.3
Component of	0	0.92	0.97	1
tensor $oldsymbol{lpha}$	α_{11}	0.92	0.97	1

Table 3: Independent components of effective elasticity tensor C^* in the Voigt notations and Biot's tensor α in different cases of porosity ϕ .

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

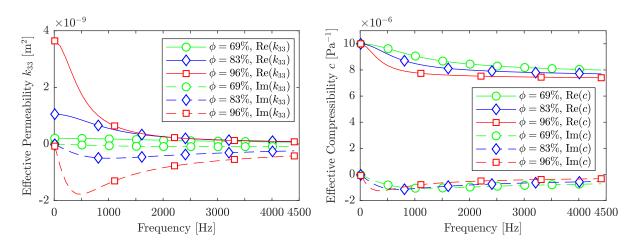


Figure 14: Dependence of the effective dynamic permeability (left) and the effective dynamic compressibility (right) on porosity.

518 6.4.2. Effect of porosity on sound absorption

The effect of porosity on the sound absorption coefficient of the material is highlighted 519 by Fig. 15. It can be remarked that the absorption coefficient increases slightly with respect 520 to the rise of the porosity within the range of frequencies lower than approximately 1000 Hz. 52: whereas the noticeable improvement on the sound absorption is accompanied by the decrease 522 of the porosity for higher frequencies. In the process of making the porous material by using 523 the Spark Plasma Sintering (SPS) technique, the resulting porosity depends on the pressure 524 and temperature. Higher pressure and higher temperature normally induce a sintered poro-525 gen template with more porogen constituents in a volume than in case of a template resulting 526 from a process with lower pressure and lower temperature [39, 15], meaning higher-porosity 527 materials in case of higher pressure and higher temperature. The results of this section 528 suggest that to promote a better sound absorption at frequencies lower than 1000 Hz, we 529

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

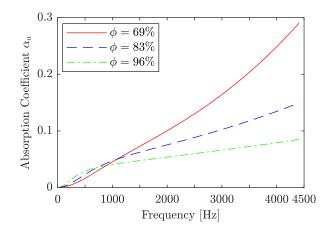


Figure 15: Dependence of sound absorption coefficient on porosity. The plate thickness is 0.5 cm.

538 6.5. Effect of pore size

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

543 6.5.1. Effect of pore size on effective properties

In this study, the pore size is linked to the cell size. Within the framework of homoge-544 nization method, the effective elasticity tensor C^* and Biot's tensor α are modified by the 545 porosity, not by the cell size or pore size. Nevertheless, the change of pore size or cell size 546 influences both the effective dynamic permeability and effective dynamic compressibility as 547 shown in Fig. 16 where the variation of these properties with respect to the frequency corre-548 sponding to three different pore sizes of 45, 350 and 710 μ m are presented. It can be noted 549 that at very low frequencies (inferior to 500 Hz), the larger the pore size, the higher the 550 permeability. This is explained by the fact that in case of a larger pore size, more entrance 55: of the fluid through the material is possible, favouring higher permeability. However, in the 552 range of higher frequencies, the permeabilities become lower and closer to each other except 553 that the permeability, in case of the smallest pore size, *i.e.*, 45 μ m, remains very low and 554 proves to be unchanged in the whole frequency range. Similarly to the permeability, the 555

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

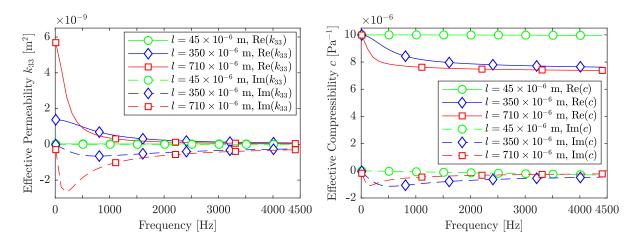


Figure 16: Dependence of the effective dynamic permeability (left) and the effective dynamic compressibility (right) on pore size. The porosity is fixed at $\phi = 83\%$.

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 μ m with a step of 5 μ 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:

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

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

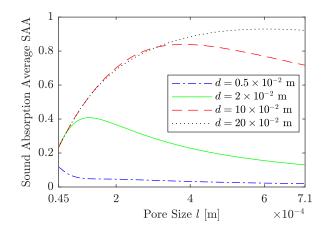


Figure 17: Normal incidence sound absorption average SAA for different cases of the pore size and the porous plate's thickness. The porosity is fixed at $\phi = 83\%$.

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

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

When the plate becomes thicker, *i.e.* 2, 10 and 20 cm thickness, the SAA is noticeably 586 improved. For the case of 2 cm thickness plate, the SAA attains its maximum value of 0.41587 at the pore size of 120 μ m while its minimum value is 0.13 at 710 μ m pore size. For 10 588 and 20 cm thickness plates, the SAA reaches its maximum values of 0.84 and 0.93 at pore 589 sizes of 365 and 600 μ m, respectively. This improvement in sound absorbing performance 590 comes from the fact that the increase in the plate thickness makes the propagation of sound 591 waves from the front to the back of the plate face more obstacles, meaning that more sound 592 energy is attenuated. Furthermore, in these three cases of plate thickness, we can observe the 593

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

616 7. Conclusion

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

In this study, homogenization calculations have been performed on representative volume elements which have been based on the idealized fillet-edge cubic elementary pore and matrix obtained from the experimental pore characterization of the material. Following the determination of effective properties by the homogenization method, a normal incidence sound absorption problem has been considered with the use of a plate made of the elaborated material as a porous absorber. After validated by a good match between numerical acoustic properties and experimental ones resulting from the three-microphone impedance tube testing, the sophisticated framework has been utilized to carry out a parametric study of the effect of the pore arrangements, the porosity and the pore size on the effective properties and acoustic performance of the material.

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

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

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

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

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.

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