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Characterization of porous absorbent materials

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Over the past twenty years, theoretical and experimental research on the acoustical properties of air-saturated porous materials for engineering applications has considerably progressed thanks to the contributions of many researchers in different institutes in the world. Accompanying the research on the acoustical properties of absorbent materials, new methods and experimental techniques were developed for the measurement of important physical parameters of these materials, necessary for the acoustic models. The attention is focused on the particularly important contributions of two laboratories: the Laboratoire d'Acoustique de l'Université du Maine (LAUM) in France and of Laboratorium voor Akoestiek en Thermische Fysica (ATF) in Belgium. A review of the main methods developed over the year in these two laboratories is proposed. This research has been very important for the validation of the models. The methods developed concern the measurement of porosity, tortuosity, flow resistivity, viscous and thermal characteristic lengths, thermal permeability and also the mechanical properties of porous materials (Young's modulus and the Poisson ratio).

1 Introduction

The study of sound propagation in air-saturated porous materials is of great importance for sound insulation and vibration damping applications. Over the past 20 years, a number of models to calculate the acoustical behaviour of porous materials have been developed. Although these models are based on physical sound theories, they require a number of material parameters and the output of a calculation will depend on the accuracy of the input parameters. Depending on the complexity of the porous material and the configuration to be modeled, up to seven physical parameters and two or more elastic parameters may be needed. A major reference in this area is the book by J. F. Allard [1] (first and second editions) that provides the theoretical framework to solve a great number of engineering acoustics problems.

2 Models and parameters

2.1 Different models

The interest for the sound propagation in porous materials goes back to the end of the XIXth century with the work of Lord Rayleigh. In 1949, Zwikker and Kosten [2] contributed substantially to the field by offering a model of sound propagation in materials containing cylindrical pores taking into account the viscous and thermal interaction between air and the solid. In 1956, M. A. Biot [3,4] published a refined model of the acoustic wave propagation in fluid saturated porous media accounting for elastic, inertial and viscous couplings between the two phases. In this model, the medium is poroelastic and viscous frictions between the solid and the fluid are included. Biot studied the low frequency [3] and the high frequency [4] behaviours. For the latter, the fluid flow in the pore deviates from Poiseuille's flow. In the high frequency regime, Biot introduced a sinuosity factor and a structural factor related to the pore geometry. The Biot model is self-consistent and self-sufficient at high frequencies as well as at low frequencies. However, more recent developments, worthy of interest have been proposed. Yet, the Biot theory remained relatively unrecognized and unappreciated until the years 1970-1980. From these years, this theory has been rediscovered and its success never diminished since then. The scientific community realized the great potential of Biot's theory in numerous fields such as geophysics, petroleum prospection, the automotive industry or medicine. The Johnson-Champoux-Allard in acoustic

engineering as well as many other models developed recently are based on this theory.

Over the years, other types of models than Biot's theory were also proposed. Some of them are simple and empirical. For example, in 1970, Delany and Bazley [5] proposed an empirical model describing the sound wave propagation in fibrous materials. This model is simple and has been very popular in engineering acoustics. More recently, Miki [6] has extended the work of Delany and Bazley and has proposed several empirical expressions for the wave number and the characteristic impedance for several materials and for several frequency ranges. Others models are more refined and are based on other principles such as the Wilson model [7], which is based on relaxation processes.

This article is concerned with the most refined models derived from the Biot theory and capable of accounting for poroelasticity, if needed. The attention is also focused on the parameters (microstructural, physical and mechanical) associated with these models. Attenborough [8] has shown the importance of tortuosity and of the parameters related to the complexity of the pore geometry at high frequencies. In his model, he introduced a dynamic pore shape factor. Johnson et al. [9] have studied the high frequency asymptotic behaviour of a Newtonian fluid subjected to a pressure gradient in the pores of a porous medium. They have introduced the concepts of dynamic tortuosity and of dynamic permeability. They studied the low and high frequency behaviours of these functions and proposed a function linking these two behaviours. In this process, they defined a physical macroscopical parameter Λ , named "viscous characteristic length". This parameter is related to the pore microgeometry. Following the work by Johnson et al., Champoux and Allard [10] studied the thermal exchanges between the different fluid layers in the boundary layers in the vicinity of the pore walls for air saturated materials. They introduced the "thermal characteristic length" Λ' . The model incorporating both the viscous and thermal characteristic lengths is one of the most popular in engineering acoustics nowadays. It is referred to the Johnson-Champoux-Allard (JCA) model and involves 5 physical parameters. The parameters of the models are reviewed further down in this article. Lafarge et al. [11] brought a further refinement to the JCA model by defining a new parameter, namely the thermal permeability k'_0 , which is used to model the thermal exchanges between the solid and the fluid at low frequencies. Therefore, their model involves 6 parameters. More recently, Pride et al. [12] studied the low frequency inertial behaviour of the equivalent fluid and proposed a correction of the low

frequency limit of the real part of the dynamic tortuosity of Johnson et al.

The different models derived from Biot's theory (the models by Attenborough, Johnson et al., Johnson-Champoux-Allard, Lafarge et al., Pride et al.) are capable of incorporating the poroelastic behaviour of the solid frame, if necessary (in the case where the solid is much more rigid and heavier than the fluid, the porous medium can be considered as an "equivalent fluid" in the "rigid frame approximation". In this case, only one of the two Biot coupled equations is necessary and the acoustic model is simpler). These different models can be incorporated into numerical methods such as the Finite Element Method for applications involving the vibrations and the acoustics of poroelastic materials. The numerical solution of certain problems involving the two Biot coupled equations of poroelasticity involving the solid and the fluid displacements ($\mathbf{u}, \mathbf{U}$) can require a high computation time due to the great number of degrees of freedom. For this reason, Attala et al. [13] proposed a new formulation of Biot's coupled equation in which, the conjugate variables ($\mathbf{u}, \mathbf{U}$) are replaced by ($\mathbf{u}, p$), where p represents the fluid pressure. Since p is a scalar, This formulation allows, without any concession on the generality, to reduce the number of degrees of freedom and therefore, the computation time. More recently, a new formulation has been proposed by Dazel et al. [14] in which the fluid displacement potential ϕ is used.

2.2 The parameters of the models

The porosity ϕ was the first parameter defined in early models. From Darcy's law of fluid flow through porous media, the notion of permeability k_0 was then introduced in the acoustics of porous media to account for viscous frictions. In his original work of 1956, Biot [4] introduced the concepts of tortuosity (sinuosity factor ξ) and also the structural factor δ (deviation from cylinders with constant circular cross section) characterizing the complexity of the pore geometry at high frequencies. Attenborough [8] offered an alternative definition for the tortuosity (q) and proposed a description in terms of static and dynamic shape factors. Johnson et al. [9] introduced the concepts of dynamic tortuosity (with its high frequency limit α_∞) and permeability and of viscous characteristic length Λ . The dynamic compressibility and thermal characteristic lengths Λ' were then introduced by Champoux and Allard [10]. Lafarge et al. [11] introduced the thermal permeability k'_0 . Pride et al. [12] refined the low frequency asymptotic behavior of the model by Johnson et al. and studied the low frequency limit of the tortuosity.

3 Characterization of porous acoustic materials

3.1 Physical parameters

Two kinds of methods can be considered for the measurement of porosity and other parameters : the non-acoustic methods and the acoustic methods. The non-acoustic methods include different methods, such as a method based on Boyle's law for isothermal processes (Beranek [15]), the principle of which has become standard (ASTM D2856-949 [16]). An improved and modernized version was proposed by Champoux et al. [17]. Leclaire et

al. [18] used an improved version of Beranek's experimental setup to compare air volumes. Other well-known classical methods are based on density measurements. Inspired from this principle, Panneton and Gross [19] proposed a method based on pressure/mass measurements. Salissou et al. [20] presented a method based on a missing mass.

For the flow resistivity or the permeability (the flow resistivity being inversely proportional to the permeability), classical methods are based on the pressure gradient and flow velocity measurements [21,22].

Acoustic methods are based on the inversion of physical parameters from acoustic measurement data. Depending on the known parameters in the problem considered, the appropriate parameters can be deduced. Different versions of acoustic methods were used in the past to determine the porosity, the tortuosity, the permeability or the flow resistivity, or even the viscous and thermal characteristic lengths. In addition, low frequency methods (impedance tube measurements) and high frequency methods (ultrasonic measurement) can be distinguished. In the low frequency methods (for example [23-25]), the impedance and transmission tube with inverse or indirect methods can be used. The high frequency methods (for example [26-30]) involve the use of ultrasonic waves in air saturated materials.

We now focus our attention on methods based on the propagation of airborne ultrasonic waves. Based on the pioneering experiments by Nagy [31,32], original characterization methods were developed over the years, in particular in the Laboratorium voor Akoestiek en Thermische Fysica (ATF) of the K.atholieke Universiteit Leuven in Belgium and in the Laboratoire d'Acoustique de l'Université du Maine (LAUM) in France. Allard et al. [26] were the first to measure the tortuosity of highly porous polyurethane foams saturated by air. Later on, Leclaire et al. [27,28] improved this method to determine the tortuosity but also the viscous and thermal characteristic lengths. These methods make use of the relatively simple expressions for the wavenumber and related functions at high frequencies. Several extensions have been proposed since these first experiments [29,30,33]. Fellah et al. [29] proposed, in the time domain, a method for measuring the porosity and tortuosity based on the solution of inverse problem using the reflected waves by the first interface of porous material. This method required measurements with two different incidence angles. Umnova et al. [34] proposed a method to determine the tortuosity and the porosity from transmission and reflection on thick samples of porous material in a tube at normal incidence. Based on Panneton et al. approach [23] for low frequencies, Groby et al. [30] proposed an analytical method in frequency domain to estimate the JCA macroscopic parameters (including the porosity) with transmitted and reflected coefficients.

3.2 Mechanical parameters

Approximating the material as a rigid porous material gives good results in many applications for many sound-absorbing materials. However, this approximation will not always hold, and in a number of cases, the acoustic material will have to be described with the full theory for sound propagation in poroelastic media. For instance, if the porous layer is fixed to a vibrating plate, the (complex) elastic coefficients of the material will be of crucial importance [35]. If the flow resistivity and the tortuosity of

the material are high (as is often the case for good sound-absorbing materials), the movement of the frame cannot be neglected and the elasticity will have to be included in the model. The elastic moduli of poroelastic materials saturated by fluids were defined by Biot and Willis [36] in 1957 to accompany Biot's theory. Evaluating these coefficients is less trivial than it appears. First of all, the elastic coefficients of the "dry" (unsaturated) frame are needed in the models. This leads to two options: either designing an experiment in vacuum where the numerical inversion to extract the elastic coefficients will be easy or designing an experiment in air that is easier to perform but where the numerical inversion may require the full model. Pioneering work on this subject has been done by Pritz [37]. Moreover, many sound absorbing or sound-damping materials are viscoelastic and their elastic coefficients may depend on frequency and temperature. However, many interesting studies have been proposed over the past 20 years by ATF and LAUM laboratories.

Low frequency methods

The simplest experiments to evaluate the elastic coefficients are based on the measurement of a vibrational resonance frequency in a sample (rod-shaped, bar-shaped or a plate) of small dimensions compared to the wavelength involved. If the material is isotropic, two complex elastic coefficients are needed. In case the material is anisotropic, more parameters are needed [38,39]. These methods are based on the vibration of a sample of finite size resulting in a transfer function with resonance peaks. The effect of resonances in the transfer function is that the moduli are determined with a precision that varies and depends on the frequency area examined. The precision is best around the resonance frequencies and poorer elsewhere. In addition, in the experiment for measuring Young's modulus, the wavelengths involved are greater than or in the order of the length of the rod, they must be greater than the lateral size and method is limited to low frequencies (typically 400 Hz). Since most the porous materials are also designed to attenuate structure-borne sound, their structural damping is high, and as a consequence, the elastic coefficients are frequency dependent. Evaluating the elastic coefficients at low frequencies can have a poor predictive value for higher frequencies.

Higher frequency methods

Higher frequency methods for measuring the shear modulus based on quarter wavelength acoustic resonances of porous layers [40] or on structure-borne Rayleigh wave propagation at the interface between a porous layer and the surrounding fluid [41] were proposed. The Rayleigh wave method allowed measurement up to 3 kHz. Other methods based on the propagation on guided and interface waves were also proposed more recently by Boeckx et al. [42,43]. Together with a complete description of a soft porous layer lying on a rigid substrate, they proposed a novel experimental technique for measuring the phase velocities of all the possible modes. The method is based on the generation and detection of standing waves in the layer and on the spatial Fourier transform of the standing-wave pattern. The principle of this method was inspired from the well-known impedance tube measurement method. Research in this area and on the influence of temperature in a wide frequency range is promising and ongoing.

4 Recent developments in material characterization

4.1 New parameters and new characterization methods

Recent research on the acoustical properties of porous materials [44-46] has revealed the possible existence of pores that are partially opened (pores opened at one end only or dead-end pores). It is thought that complex materials such as porous metallic foams, some recycled materials, asphalt, porous rocks or even bones can exhibit these features. For these materials, the Biot assumption on the flow of a fluid inside a pore may not be fulfilled. Although dead-end porosity is not new and has already been reported by geophysicists no model was able until now to describe precisely its influence on the acoustical properties of porous materials. In the model proposed in Ref. [46], two new physical parameters were introduced: namely the average length of the dead ends l_{DE} and the dead-end porosity ϕ_{DE} .

The introduction of the new parameters opens up numerous interesting new research scopes in the theoretical and numerical description of these materials and in material characterization. Research on the measurement of these two parameters is currently ongoing and in the continuity of the research developed at ATF and LAUM, which has brought numerous new methods over the years, new research is being developed. First results were obtained recently, some of which were presented at a conference [47].

As an example, recent results on new methods for measuring the kinematic porosity (or Biot porosity) are now presented. The kinematic porosity is related to the displacement of water moving in a permeable medium. It is equivalent to the ratio of the volume of the interstices actually traversed by moving water and the total volume of the medium. The kinematic porosity can be approximated by drainage porosity, which is defined as the ratio of the volume of water drained by gravity from a saturated representative sample to the total volume of the sample. Figure 1a) illustrate the distinction between the Biot porosity and the dead-end porosity on a simplified artificial sample with well controlled parameters and fabricated in order to validate the theoretical predictions in refs [44-46]. The Biot porosity is a fraction of the total open porosity. The relationship between total porosity ϕ_T , the Biot (kinematic) porosity ϕ_B and the dead-end porosity ϕ_{DE} is:

$$\phi_T = \phi_B + \phi_{DE}. \quad (1)$$

For the measurement of ϕ_B , two methods can be used depending on the data at our disposal, a low and a high frequency methods. The results presented here are taken from references [46], [47] and [48]. Only the principles of the methods are presented.

High frequency methods

In this method, high frequency measurements obtained from ultrasonic experiments are used in conjunction with the theoretical value of the intercept with the vertical axis at zero frequency of the modulus of the logarithm of the transmission coefficient. The experimental principle consists in using ultrasonic wave transmission experiments (see refs [26-28]). It can be shown that methods based on ultrasonic reflection [29,30] cannot be applied here.

The porosity actually “seen” by the ultrasonic wave corresponds to the kinematic porosity and therefore provides a measure of ϕ_B . Since, the total open porosity can be measured by classical method (or even calculated in the example of Figure 1), the dead-end porosity can be deduced from these measurements.

Figure 1b) illustrate the principle of the method involving the high frequency ultrasonic experimental data, the linear behavior and the intercept with the vertical axis). The high frequency behaviour was studied by Moussatov et al. [33]. The intercept with the vertical axis is obtained by extending the straight line that fits the experimental data. Its value being a function of porosity (the Biot porosity here), the determination of the intercept yields the sought porosity.

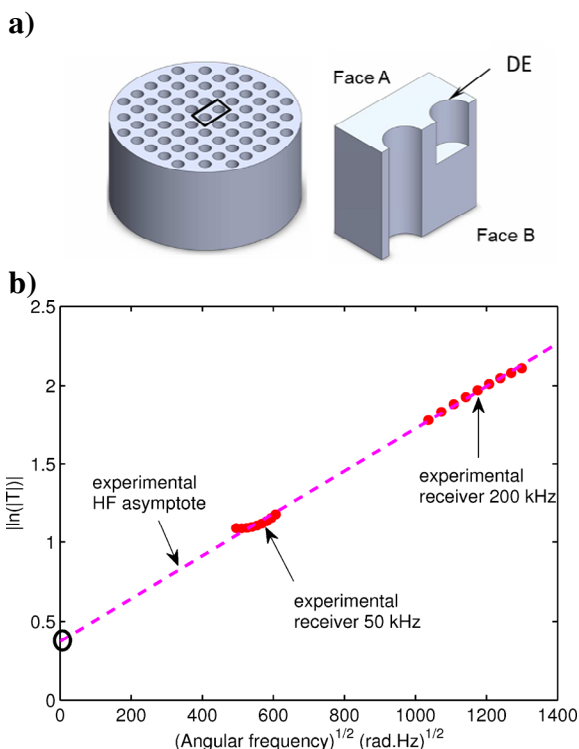


Figure 1: a) Simplified sample studied [48]. b) Modulus of the transmission loss (theoretical) and ultrasonic data (experimental) on this sample [47].

Low frequency method

In this method, low frequency measurements obtained from transmission experiments are used in conjunction with the theoretical high frequency asymptotic behaviour of the phase velocity of the ultrasonic waves. As seen in Figure 2, The low frequency behaviour of the experimental data can be fitted by a straight line. The intercept with the theoretical asymptote corresponds to the Biot characteristic cut-off frequency separating the low and high frequency behaviours. This frequency is

$$f_c = \sigma \frac{\phi_B}{2\pi \alpha_\infty \rho_f} \quad (2)$$

where σ is the flow resistivity, α_∞ the tortuosity and ρ_f the density of the fluid. These parameters are of course supposed to be known. The Biot frequency depends on the sought porosity (the Biot porosity here).

Both the low and high frequency methods proposed here are original inasmuch as they involve experimental data obtained either from ultrasonic experiments or from

experimental data obtained at audible frequencies (transmission tube for example), in combination with low or high frequency asymptotic behaviour that are fairly simple

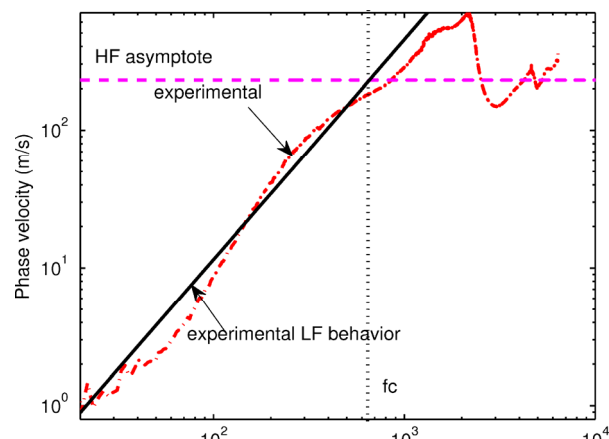


Figure 2: Log-Log plot of the phase velocity of transmitted sound waves in a porous layer as a function of frequency. The horizontal asymptote represents the high frequency behaviour [47].

4.2 Effect of dead-end porosity on the acoustic transmission loss in a porous metallic foam

Figure 3 show the influence of dead-end porosity on experimental data on the transmission loss in an aluminum foam of total open porosity $64.5 \pm 3 \%$. The dead-end porosity measured by the low frequency method was found to be $8.6 \pm 3.2 \%$. The results show a better match of the JCA model with the experimental results when the dead-end porosity is accounted for in the model.

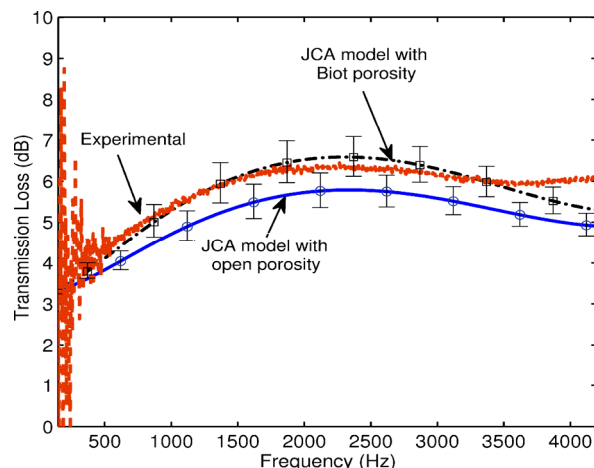


Figure 3: Comparison between theoretical and experimental results for the transmission loss in a porous aluminum [47].

5 Conclusion

In this article, a review of the theoretical description of porous absorbent materials saturated by air was presented together with their associated physical parameters and characterization methods developed over the years in ATF (Belgium) and LAUM (France). In the continuity of this research, recent developments on materials containing dead-end pores and new characterization methods for these materials were also proposed.

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