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# How reproducible is the acoustical characterization of porous media?

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## Abstract

There is a considerable number of research publications on the characterization of porous media that is carried out in accordance with the ISO 10534-2 [ISO 10534-2, 2001] and/or ISO 9053 standards ISO 9053, 1991]. According to WEB OF SCIENCE™ (last accessed on 22 of September 2016) there were 339 publications in the Journal of the Acoustical Society of America alone which deal with the acoustics of porous media. However, the reproducibility of these characterization procedures is not well understood. This paper deals with the reproducibility of some standard characterization procedures for acoustic porous materials. The paper is an extension of the work published in [K.V. Horoshenkov *et. al.* , J. Acoust. Soc. Am. 122 (1), 2007]. One novelty of this paper is that independent laboratory measurements were performed on the same material specimens so that the naturally occurring inhomogeneity in materials was controlled. Another novelty of this work is that it presents the reproducibility data for the characteristic impedance, complex wavenumber and for some related pore structure properties. This work can be helpful to understand better the tolerances of these material characterization procedures so the improvements can be developed to reduce the experimental errors and improve the reproducibility between laboratories.

## I. INTRODUCTION

The characterization of porous media has become a standard procedure which is carried out in several laboratories worldwide to validate new models for the acoustical properties of porous media, to measure the acoustical performance of new types of porous media used in noise control applications, and/or to deduce the parameters of their porous micro-structure. In addition, a number of industries rely heavily on their ability to model the acoustical properties of porous media *in-situ*. For this purpose they need to have accurate data on the acoustic impedance of porous media and propagation constant. With this is mind, it is important to have a clear understanding of the dispersion of acoustical data caused by the differences in the equipment and natural variation in the material formulation. However, this information is scarce and the standard ISO 10534-2 procedure[1] is rather ambiguous in terms of the quality and uniformity of material samples, environmental and operational conditions, the quality of setup and signal processing method. It is fair to say that the reproducibility of the standard acoustical method (ref. [1]) in application to the porous media characterization has not been properly investigated. As a result, the uncertainties of the characterization procedures are largely unknown. There are three basic questions which remain unanswered: (i) 'How accurate our acoustic material data actually are?' (ii) Would we get the same result as published by our colleagues if we test these materials in our own lab?' (iii) 'If we develop a new model is it actually more accurate than existing models in terms of any potential measurement errors we can incur?' A while ago the authors of this paper attempted to answer some of these questions through a series of experiments designed to evaluate the reproducibility in normal incidence sound absorption coefficient and surface impedance of porous specimens which were cut independently from flat sheets of porous materials sent to the 6 partners by 3 material manufacturers[2]. These experiments were carried out in 2-microphone impedance tubes in compliance with the ISO 10534-2[1] standard. As a general summary of the results, higher variations in the measured spectra for the surface impedance and acoustic absorption coefficient were observed between individual samples and individual laboratories in the case of low permeability, low homogeneity, broad pore size distribution and reconstituted porous rubber. The smallest variations (<20%) in the data were observed in the case of high permeability porous reticulated foam, although the mounting conditions for this material were difficult to reproduce in independent acoustic laboratories which resulted in a shift of the frame resonance frequency affecting the absorption coefficient in a certain frequency range. Finally, medium level variations in the measured acoustical absorption data (> 20%) were observed in the case of fiberglass. These variations were attributed to change in specimen thickness during the mounting within the measurement tube.

At the moment, the authors are not aware of any studies which provide experimental data from independent laboratories for characteristic acoustical properties (i.e. characteristic impedance and complex wavenumber) and for several physical parameters describing their micro/macro structure (airflow resistivity, open porosity, tortuosity and viscous and thermal characteristic lengths) measured for the same material specimens. Among physical parameters, only airflow resistivity can be measured according to a standard (ISO 9053[3]) and considerable work has been carried out by Garai and Pompoli[4] who coordinated the European Inter-Laboratory test as per that standard. The results of this work are limited to melamine foam samples and show that most laboratories have good internal repeatability, particularly for single sample measurements. In comparison with repeatability, the overall reproducibility is not so good mainly due to systematic deviations inherent to current laboratory practice. In this respect, there is a lack of reproducibility data which are obtained for the same material specimen tested in independent acoustic laboratories.

Therefore, the aim of this paper is to determine the dispersion of surface acoustical data (i.e. surface impedance, $z_s$, and absorption coefficient, $\alpha$), characteristic properties (i.e. characteristic impedance, $z_c$, and complex wavenumber, $k_c$), and related pore structure parameters (airflow resistivity, $\sigma$, open porosity, $\phi$, tortuosity, $\alpha_\infty$, and viscous, $\Lambda$, and thermal, $\Lambda'$, characteristic lengths) obtained for the same material sample, but tested in different acoustic laboratories. The meaning of these parameters is detailed in ref. [5].

This paper is organised as follows: section II outlines the methodology; section III presents the results of from individual laboratories and inter-laboratory data. Concluding remarks are made in the last section.

## II. METHODOLOGY

In this work, seven acoustic research centers were involved. These are: the University of Ferrara (Italy), the University of Perugia (Italy), Katholieke Universiteit Leuven (Belgium), Matelys/ENTPE in Lyon (France), Gesellschaft für Akustikforschung Dresden (Germany), the University of Bradford (UK), and Sherbrooke University (Canada). Three different porous materials were investigated: reticulated foam, consolidated flint and reconstituted porous rubber, denoted materials A, B, and C, respectively (Fig.1).

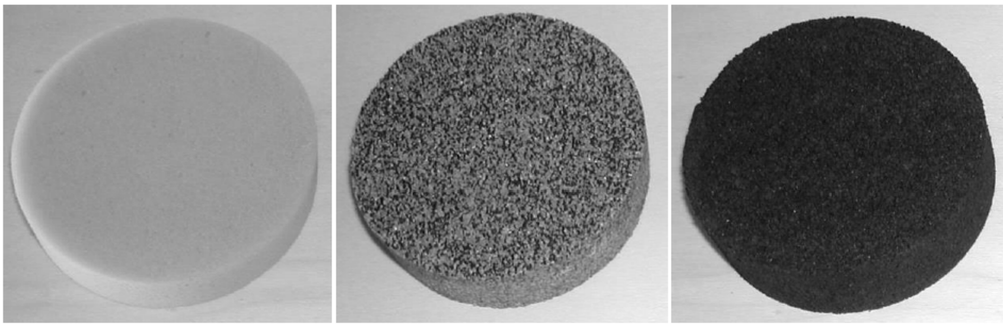

Figure 1 – Tested materials. Tested materials. A: reticulated foam (left), B: consolidated flint (center), C: reconstituted porous rubber (right).

In this research, the same set of specimens for porous materials with different diameters (99 mm, 44mm and 29 mm) was provided and shared amongst laboratories. Materials A and C were identical to those used in ref. [2], that are reticulated foam and reconstituted rubber, respectively. Material B was consolidated flint particles to minimize the effect of mounting thickness variations within the impedance tubes. In this way samples of each material were not exactly identical among all the partner laboratories because they were cut for a range of impedance tube diameters. Table I presents a basic description of the materials which were used in the inter-laboratory experiment. Table II lists the acoustical and pore structure parameters and partner laboratories in which these parameters were measured.

Table I. The porous materials used in the inter-laboratory experiment.

| Material | Description | Thickness [mm] | Density [kg/m$^3$] | Diameters [mm] | Number of samples for each diameter |
|---|---|---|---|---|---|
| A | Reticulated foam | 20±0.1 | 8,8 | 29/44/99 | 4 |
| B | Consolidated flint | 31±0.1 | 1500 | 29/44/99 | 6 |
| C | Reconstituted porous rubber | 28±0.1 | 242 | 29/44/99 | 6 |

Table II. The list of the acoustical and related pore structure parameters and partner laboratories in which these parameters were measured.

| Partner | $z_s$ , $\alpha$ | $z_c$ , $k_c$ | $\sigma$ | $\phi$ | $\alpha_\infty$ | $\Lambda$ | $\Lambda'$ |
|---|---|---|---|---|---|---|---|
| 1 | • | • | • | • | • | • | • |
| 2 | • | | • | • | • | • | • |
| 3 | • | • | • | • | | | |
| 4 | • | • | • | • | • | • | • |
| 5 | • | | • | | | | |
| 6 | • | | • | • | • | • | • |
| 7 | • | • | | | | | |

### A. Measurement of acoustical properties

The acoustical properties measured directly in accordance with the ISO 10534-2[1] were the normalized surface acoustic impedance $z_s$ [-] (for plane waves at normal incidence) and the normal incidence sound absorption coefficient $\alpha$ [-] of the material sample backed by a rigid wall. The size and diameter of the standing wave tube, the manufacturers and the excitation stimulus used by the partners are detailed in Table III. The following methods of sample mounting conditions were adopted (see Table III): (i) the diameter of the cut samples was close to or slightly smaller than the diameter of the tube and the samples were wrapped in tape to prevent any leakage around the edge - tape constraint (TC); (ii) the diameter of the sample was exactly equal to that of the tube - perfect fit (PF).

All the partners applied the amplitude and phase mismatch calibration procedures before tests (with the exception of Partner 4 who used a single microphone) in accordance with ISO 10534-2[1]. All the microphones used in these experiments were standard 1/4 inch microphones (see Table III). Partner 1 – 5 carried out tests in the frequency range consistent with that suggested in ref. [1] for a given tube diameter and microphone spacing. Partner 6 provided data in the frequency range between 200 Hz and 1600 Hz because of a low signal-to-noise ratio. It should be noted that the ISO 10534-2:2001 standard does not define the exact frequency range for a given tube diameter and microphone separation, but recommends the bounds for the lower and upper frequencies in the range (see Section 4.2 in ref. [1]). Therefore, the partners chose the frequency ranges to satisfy the standard requirements for the level of nonlinearities, frequency resolution, measurement instabilities and signal-to-noise ratio recommended in ref. [1].

Table III. The equipment and mounting conditions used to determine the acoustic absorption coefficient and surface impedance (HM: homemade equipment; TC: tape constraint; PF: perfect fit).

| **Partner** | **Tube diameter / tube manufacturer** | **Tube length [m] / microphone spacing [m]** | **Mounting conditions** | **Stimulus** | **Electronic hardware** | **Microphone type** | **Frequency range [Hz]** |
|---|---|---|---|---|---|---|---|
| 1 | 45 mm / HM | 0.5 / 0.03; 0.1 | TC | Sweep | NI USB 4431 | PCB 377C10 | 100-4200 |
| 2 | 29 mm / HM | 0.4225/0.02 | PF | White noise | SR-8 Channel Analyzer (DSP Board) | BK2670 | 400-6900 |
| 3 | 29 mm / B&K 4206 | 0.4225/0.02 | PF | White noise | Bruel and Kjear Pulse type - 2827 | BK2670 | 260-6400 |
| 4 | 29 mm / HM | 0.35/0.02 | PF | Pseudo random noise | NI PXI 4461 | BK4187 | 400-6900 |
| 5 | 29 mm / B&K 4206 | 0.4225/0.02 | TC | White noise | NI USB-9233 | MT Gefell M 365 | 200-6400 |
| 6 | 38 mm / HM | 1/0.02;0.03;0.05 | PF | Sweep | GPIB-USB | GRAS40BP | 200-1600 |
| 7 | 29 mm / B&K 4206 | 0.4225/0.02 | TC | White noise | Brüel & Kjær PULSE Type 3560-B-030 | BK4187 | 400-6400 |

Each impedance tube was driven by a single loudspeaker which was adapted to the size and the frequency range of the impedance tube and it was assumed tube vibration effect could be ignored. Regarding nonlinearity in speaker response the impedance tubes used in these experiments were designed in accordance with the ISO 10534-2:2001 [1], in which Section 4.8 suggests that "*The errors in the estimated transfer function $H_{12}$ due to nonlinearities, resolution, instability and temperature sensitivity of the signal processing equipment shall be* *less than 0,2 dB.*" This is a very small effect and authors believe that it was insignificant in experiments given a relatively high natural inhomogeneity in the material specimens and effects of specimen mounting in the tube. The sampling frequency and the sequence length used in the Fourier analysis were chosen to cover the desired frequency range and to provide adequate frequency resolution in the transfer function spectrum as suggested in ref. [1]. The effects of temperature and variations in atmospheric pressure were compensated for as suggested in ref. [1]. The material thickness was measured to ±0.1 mm using calibrated calipers.

In addition, the normalized characteristic impedance, $z_c$, and the complex wavenumber, $k_c$, were measured using a well-established 4 microphone and transfer matrix technique as described by Song and Bolton[6]. Partner 4 used a 3 microphones technique as described in ref. [7]. The details of the equipment and measurement techniques are summarized in Table IV. The equipment used in 3- or 4-microphone tests was properly calibrated prior to the start of the experiments to compensate for microphone channel mismatch using the procedure similar to that suggested in ref. [1]. All the microphones used in these experiments were standard 1/4 inch measurement microphones (See Table IV). For the frequency range for these experiments was chose to meet the recommendations for the impedance tube setup as suggested in the ISO 10534-2 [1].

Table IV. The equipment, measurement technique and sample mounting conditions used to determine the characteristic impedance and complex wavenumber (TC: tape constraint; PF: perfect fit).

| **Partner** | **Tube diameter / tube manufacturer** | **Measurement technique** | **Mounting conditions** | **Stimulus** | **Electronic hardware** | **Microphone type** | **Frequency range [Hz]** |
|---|---|---|---|---|---|---|---|
| 1 | 45 mm / HM | 4 microphones techniques – refs [6-8] | TC | Sweep | NI USB 4431 | PCB 377C10 | 100-4200 |
| 3 | 44 mm / HM | 4 microphones techniques – refs [6] | PF | Pulse | ND | BK2670 | 188-3500 |
| 4 | 29 mm / HM | 3 microphones technique – ref [7] | PF | Pseudo random noise | NI PXI 4461 | BK4187 | 400-6800 |
| 7 | 29 mm / B&K 4206 | 4 microphones techniques – refs [6] | TC | White noise | Brüel & Kjær PULSE Type 3560-B-030 | BK4187 | 400-6400 |

### B. Measurement of pore structure properties

The airflow resistivity, $\sigma$, was measured by the participants using the procedure described in ISO 9053[3]. This standard indicates that the value of airflow resistivity has to be determined for the airflow velocity of less than 0.5 mm/s. When this is not possible the standard suggests repeating tests at different values of airflow velocity and extrapolating the value of the airflow resistivity at the nominal value of 0.5 mm/s. Table V describes the equipment, the measurement techniques and the procedures used by the partners to measure the flow resistivity.

Table V. The equipment and measurement technique used to determine the airflow resistivity.

| Partner | Tube diameter / tube manufacturer | Measurement technique | Pressure transducer / Pressure range | Extrapolation of d at 0.5 mm/s |
|---|---|---|---|---|
| 1 | 100 mm / HM | ISO 9053-Method B | BK4186 | Linear best-fit between pressure difference and velocity passing through zero |
| 2 | 99 / 44 mm / HM | ISO 9053-Method A | MKS Type 698A (0.1-1000 Torr) | No extrapolation |
| 3 | 100 mm / HM | ISO 9053-Method A | FCO 34 (0-10 Pa) | Linear best-fit between pressure difference and velocity passing through zero |
| 4 | 29 mm / HM | ISO 9053-Method A | MKS 120AD Baratron 1 torr (0-1 Torr) | Direct measurement at 0.5 mm/s |
| 5 | 99 mm / HM | ISO 9053-Method A | SET-D267MR-6 (-100-100 Pa) | Linear best-fit between pressure difference and velocity passing through zero |
| 6 | 38 mm /HM | ISO 9053-Method A | Not declared | No extrapolation |

Five partners measured the open porosity, $\phi$, using the equipment and measurement techniques as described in Table VI. Partners 1-4 used the isothermal compression of volume (Boyle's law) experiment[9] to measure the porosity. Partner 7 used an acoustic method based on the analysis of the wave reflected from the sample at oblique incidence[10].

Table VII gives an overview of the measurement techniques for the measurement of high frequency limit of tortuosity $\alpha_\infty$ and characteristic lengths ($\Lambda$ and $\Lambda'$). A majority of partners obtained the tortuosity and characteristic lengths from the curve fitting of acoustical data and theoretical modelling as described in refs. [13-15]. Partners 1 and 6 performed measurements of tortuosity by means of ultrasonic tests[11-12]. Partners 1 and 2 used samples of different diameters to measure the flow resistivity and acoustical properties. This means that two different sets of material specimens were used by this partner in the reported experiments.

Table VI. The equipment and measurement technique used to determine the open porosity (HM: homemade equipment).

| Partner | Tube diameter / tube manufacturer | Measurement technique |
|---|---|---|
| 1 | 99 mm / HM | Isothermal compression of volume[9] |
| 2 | 99 mm / HM | Isothermal compression of volume[9] |
| 3 | 29 mm / HM | Isothermal compression of volume[9] |
| 4 | 29 mm / HM | Isothermal compression of volume[9] |
| 6 | 38 mm /HM | Ultrasonic reflection method[10] |

Table VII. The equipment, measurement techniques used to determine the tortuosity and characteristic lengths.

| Partner | Device | Measurement technique |
|---|---|---|
| 1 | 99 / 45 mm / HM | Ultrasonic test[11-12] and fitting from acoustical data[13] |
| 2 | 44 mm kundt tube / HM | Fitting from acoustical data[14] |
| 4 | 29 mm / HM | Fitting from acoustical data[15] |
| 6 | 38 mm /HM | Ultrasonic test[11] / fitting from acoustical data |

### C. Error analysis

Each laboratory carried out two different sets of measurements: (i) tests on different samples of each material (with the exception of Partner 6), (ii) tests on the same sample for each material (with the exception of Partner 4 and 6). The relative errors for a quantity (here generically named as *x*) measured from these tests were defined as the ratio between its standard deviation and mean value (and expressed in percentage):

$$\varepsilon_x = \frac{\sigma_x}{\langle x \rangle} \times 100, \quad [\%] \qquad (1)$$

$\langle x \rangle$ and $\sigma_x$ being the mean value and the standard deviation, respectively.

The statistical procedures for the analysis of the sound absorption coefficient, airflow resistivity and open porosity described in the ISO 5725-1 and 5725-2 standards[15, 16] were applied.

According to the ISO 5725-2, the repeatability standard deviation is a measurement of the dispersion of the distribution of independent test results obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time. The reproducibility standard deviation is a measurement of the dispersion of the distribution of test results obtained with the same method on identical test items in different and independent laboratories with different operators using different equipment. According to these standards it is also possible to define for each of the tested materials the repeatability standard deviation in an acoustical parameter for a single sample measured in laboratory *i*:

$$\bar{\sigma}_{1,i} = \frac{\sum_{j=1}^{n_f} \sigma_{1,ij}}{n_f} \tag{2}$$

where $\sigma_{1,ij}$ is the standard deviation for laboratory *i* at frequency *j* for the measured values of the acoustical parameter for the same one sample and $n_f$ is the number of discrete frequencies at which this parameter was measured. Such deviation depends mainly on the random error in the measurement chain, environmental factors, post-processing of data and mounting conditions for the sample in the tube. The repeatability standard deviation for all the different samples in laboratory *i* can be defined as

$$\bar{\sigma}_{A,i} = \frac{\sum_{j=1}^{n_f} \sigma_{A,ij}}{n_f} \tag{3}$$

where $\sigma_{A,ij}$ is the standard deviation for laboratory *i* at frequency *j* for the measured values of the acoustical parameter between the all different samples. Such deviation depends on random errors, sample mounting conditions, homogeneity and sample preparation techniques. The above quantities can be used to calculate the mean material standard deviation as:

$$\langle \sigma_M \rangle = \frac{\sum_{i=1}^{n_L} \bar{\sigma}_{M,i}}{n_L} \tag{4}$$

where:

$$\bar{\sigma}_{M,i} = \sqrt{\bar{\sigma}_{A,i}^2 - \bar{\sigma}_{1,i}^2} \tag{5}$$

is the material standard deviation for laboratory *i* and $n_L$ is the number of independent laboratories. In the above equation we assume that the total error is a combination of the natural variation in the material properties and that which results from the measurement itself. Therefore, the material standard deviation is a measure of the dispersion in the data due to natural variation in the material properties from sample to sample so that the mean material standard deviation is related mainly to homogeneity and sample preparation technique adopted in this work.

The inter-laboratory standard deviation for a single sample is calculated as :

$$\langle \sigma_{I1} \rangle = \frac{1}{n_f} \sum_{j=1}^{n_f} \sqrt{\frac{\sum_{i=1}^{n_L} \left( m_{I1,ij} - \langle m_{I1,j} \rangle \right)^2}{n_L - 1}} \tag{6}$$

where $m_{I1,ij}$ is the mean value of the acoustic parameter measured for the same sample in the laboratory *i* at frequency *j*. Here:

$$\langle m_{I1,j} \rangle = \frac{\sum_{i=1}^{n_L} m_{I1,ij}}{n_L} \tag{7}$$

is the average of the mean values among different laboratories at frequency *j*.

The inter-laboratory standard deviation for tests on all the material samples can be calculated in a similar manner as:

$$\langle \sigma_{IA} \rangle = \frac{1}{n_f} \sum_{j=1}^{n_f} \sqrt{\frac{\sum_{i=1}^{n_L} \left( m_{IA,ij} - \langle m_{IA,j} \rangle \right)^2}{n_L - 1}} \tag{8}$$

where $m_{IA,ij}$ is the mean value for laboratory *i* and frequency *j* obtained for different samples. Here:

$$\langle m_{IA,j} \rangle = \frac{\sum_{i=1}^{n_L} m_{IA,ij}}{n_L} \tag{9}$$

is the average of the mean values among different laboratories measured at frequency *j*.

In this way the reproducibility standard deviations for a single sample and for all the samples can be calculated as:

$$\sigma_{R1} = \sqrt{\langle \sigma_1 \rangle^2 + \langle \sigma_{I1} \rangle^2} \text{ and } \sigma_{RA} = \sqrt{\langle \sigma_A \rangle^2 + \langle \sigma_{IA} \rangle^2}\,, \tag{10}$$

respectively. Here:

$$\langle \sigma_1 \rangle = \frac{\sum_{i=1}^{n_L} \bar{\sigma}_{1,i}}{n_L} \text{ and } \langle \sigma_A \rangle = \frac{\sum_{i=1}^{n_L} \bar{\sigma}_{A,i}}{n_L} \tag{11}$$

are the mean repeatability standard deviation for a single sample and for all the different samples, respectively. A similar statistical analysis was applied to other material parameters which were measured non-acoustically. In this case the value of $n_f$ in the above equations was set to 1.

## III. RESULTS

### A. Surface impedance and sound absorption coefficient

The error analysis was based only on the 400 - 3500 Hz range to make data from all the 6 partners compatible. The following figures show the raw data in the frequency range which was actually utilized by each individual partner. The results of the inter-laboratory tests show that the relative errors (calculated using Eq. 1) in the real ($\varepsilon_{\Re(z_s)}$) and imaginary ($\varepsilon_{\Im(z_s)}$) parts of the surface impedance and that of the absorption coefficient $\varepsilon_\alpha$, calculated in the frequency range between 400 Hz and 3500 Hz, were 13%, 13% and 4%, respectively. For material B these were 24%, 10% and 19%, respectively. For material C these were 29%, 9% and 7%, respectively. In the case when the same samples were measured by each laboratory, deviations were generally found lower: 11% / 9% / 7% for material A, 8% / 7% / 3% for material B and 8% / 21% / 1% for material C. Such results indicate a gain in the accuracy with respect to the previous inter-laboratory tests mainly because the same set of materials was used minimizing the effect of the variability in the pore microstructure between different material slabs.

Figures 2 to 4 show the comparison of the measured data for the real and imaginary parts of the surface impedance and sound absorption coefficient for all the materials tested in laboratories 1 - 5 and 7. Each curve is the average of all the tests on all the different samples of the same material. The results obtained by laboratory 6 have been omitted from these figures since measurements were carried out on a single specimen for each material since accidently destroyed some samples trying to adapt them to fit the tube.

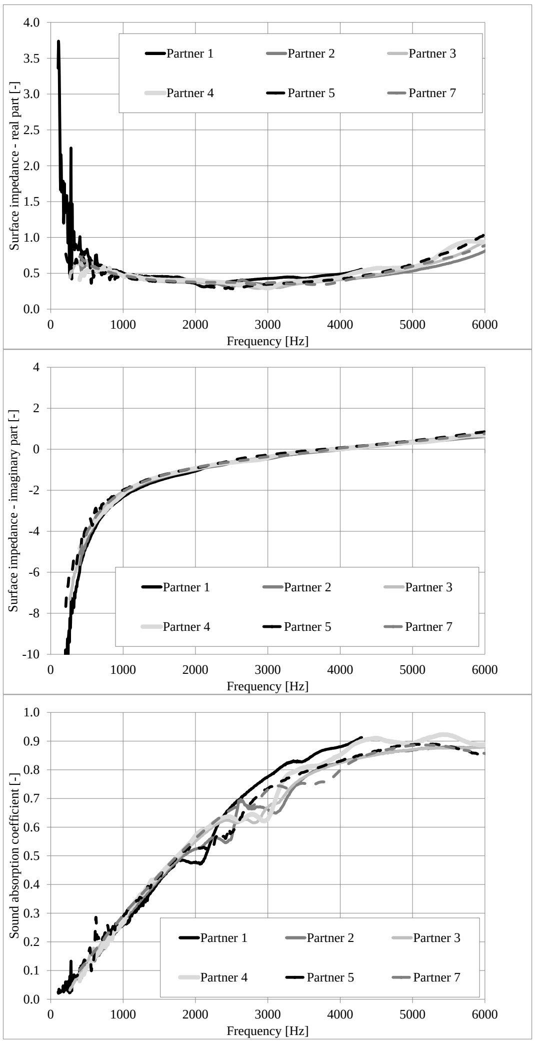


Figure 2 – The average of the real part of surface impedance spectra (top), imaginary part of surface impedance spectra (middle), and the sound absorption coefficient spectra (bottom) measured by the participating partners for material A.

The surface impedance and absorption coefficient spectra for material A are shown in Figs. 2(a)–2(c). There is better than 20% agreement in terms of relative errors between the results for the impedance obtained in the six laboratories. The maximum relative error in the real and imaginary part of the impedance spectrum of ±25% is observed below 3000 Hz (see Figs. 2(a) and 2(b)). A noticeable increase in the dispersion in the absorption coefficient data can be observed around the frequency of the frame resonance above 2000 Hz (see Fig. 2(c)). This resonance is often observed in data for low density, soft porous media[18]. The dispersion in the absorption coefficient due to the frame resonance can amount to values between 20% and 30%.

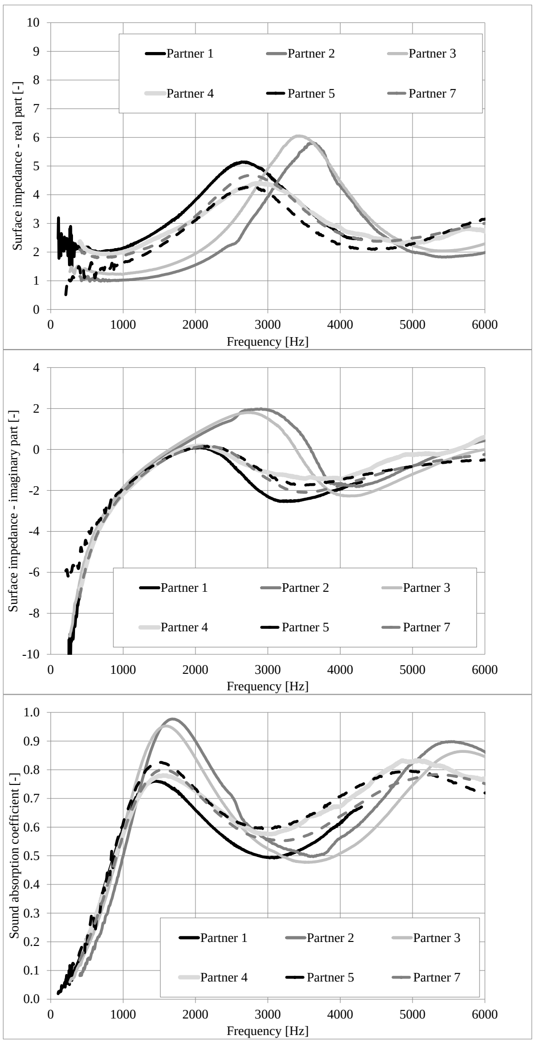


Figure 3 - The average of the real part of surface impedance spectra (top), imaginary part of surface impedance spectra (middle), and the sound absorption coefficient spectra (bottom) measured by each of the participating partners for material B.

In the case of material B the dispersion for all the acoustic quantities is high. The results from partners 2 and 3 are close. These partners used 29 mm diameter impedance tubes, the same type of microphones and similar excitation stimulus. Partners 5 and 7 also used the same diameter tube and similar type of acoustic stimulus. However, their results are noticeably different from those obtained in laboratories 2 and 3. The results from laboratories 1, 4, 5 and 7 follow a similar trend despite some differences in the tube diameter, excitation stimulus and microphone types. The dispersion in the absorption coefficient for frequencies above 1000 Hz is between 20% and 40% (Fig. 3(c)). Given a relatively high rigidity of material B, such differences are likely to be attributed to the differences in the mounting condition. Partners 1, 5 and 7 wrapped the edges of their samples in tape to prevent any leakage around the edge. The other partners reported a very good fit which did not require the sample to be wrapped in tape.

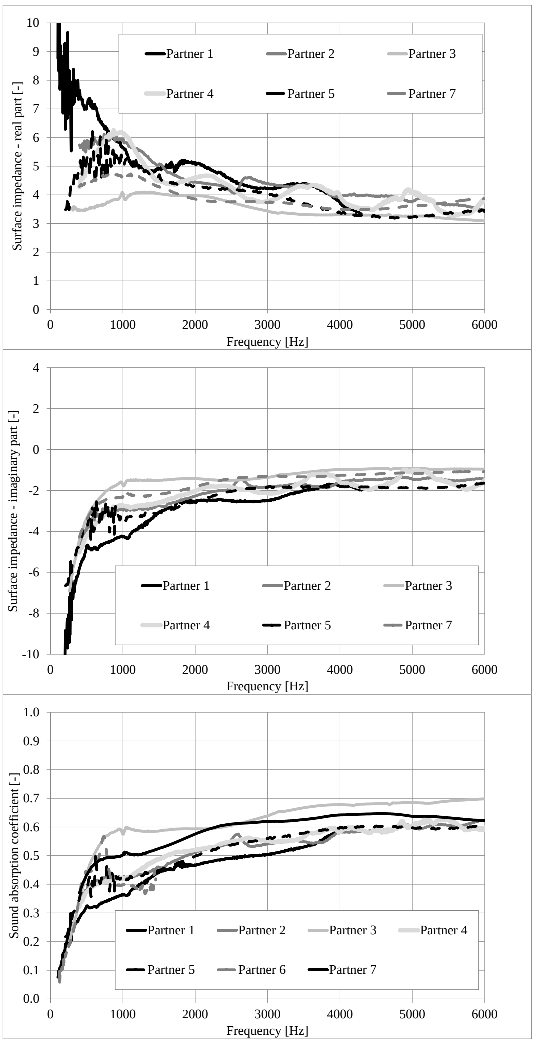


Figure 4 - The average of the real part of surface impedance spectra (top), imaginary part of surface impedance spectra (middle), and the sound absorption coefficient spectra (bottom) measured by each the participating partners for material C.

The results obtained for material C show that there can be a maximum of four to five fold dispersion in the value of the real part of the surface impedance in the low frequency limit below 1000 Hz (Fig. 4(a)). The agreement between the data for the imaginary part is poor across the whole frequency range (Fig. 4(b)). This dispersion is reflected in the erratic behavior of the absorption coefficient which spectra are shown in Fig. 4(c). The obtained data suggest that the absorption coefficient for this material can vary within a 10-20% range. These differences can be attributed to the variability in the mounting conditions. Partner 1 wrapped the edge of their samples in tape and this could have resulted in some degree of pore deformation and increased airflow resistivity which generally leads to an underestimation of the sound absorption coefficient spectrum.

A summary of the statistical error analysis carried out according to ISO 5725-2 can be found in Table VIII which presents the values of standard deviations for the absorption coefficient determined from this inter-laboratory experiment. These results enable us to draw the following conclusions:

- The mean repeatability standard deviation for a single sample $\langle \sigma_1 \rangle$ is relatively low for all the tested materials. This can suggest that random errors and mounting conditions are not dominant (below 0.01).
- The mean repeatability standard deviation for different samples $\langle \sigma_A \rangle$ is significantly (2.8 – 7 times) higher in comparison with that for a single sample test. The lowest value is for material A and it is likely to relate to the structural resonance of the material mounted in the tube. The value of $\langle \sigma_A \rangle$ for material B is the highest, probably due to the inhomogeneity of the material itself. Material C is characterized by an intermediate value of $\langle \sigma_A \rangle$ which may relate mainly to the homogeneity of the material and variation in the mounting conditions. This material has a significantly high airflow resistivity, it is flexible and any lateral compression applied to its edge when inserted in the tube can increase the flow resistivity noticeably.
- The effect of material standard deviation, $\langle \sigma_M \rangle$, is dominant when compared with the effects due to random errors and mounting conditions for a single sample. The material standard deviation is related to the natural inhomogeneity of the material and sample preparation technique. The latter effect is on the sample mounted in the tube, that may cause a change in the sample elastic behavior (e.g. in the case of material A), a leakage between the material edge and tube walls (e.g. in the case of material B) or excessive compression of the sample effectively altering its acoustical properties (e.g. in the case of material C).
- The inter-laboratory standard deviation for a single sample $\langle \sigma_{I1} \rangle$ is approximately 2 times higher than $\langle \sigma_M \rangle$, because it is calculated from the average values of $m_{IA,ij}$ for each laboratory, it is affected by the systematic errors and differences in the equipment used for the impedance tube test.
- The inter-laboratory standard deviations for a single $\sigma_{R1}$ and for different samples $\sigma_{RA}$ are comparable that suggests the dominant influence of different impedance tubes rather than of some systematic errors.
- The reproducibility standard deviation for single $\langle \sigma_{R1} \rangle$ and different $\langle \sigma_{RA} \rangle$ samples is lower than 0.07 for all tested materials.

Table VIII. The standard deviations for the sound absorption coefficient determined in accordance with the ISO 5725-2[17].

| Standard deviation | Sample A | Sample B | Sample C |
|---|---|---|---|
| $\langle \sigma_1 \rangle$ | 0.005 | 0.007 | 0.004 |
| $\langle \sigma_A \rangle$ | 0.014 | 0.039 | 0.028 |
| $\langle \sigma_M \rangle$ | 0.012 | 0.038 | 0.027 |
| $\langle \sigma_{I1} \rangle$ | 0.03 | 0.054 | 0.044 |
| $\langle \sigma_{IA} \rangle$ | 0.025 | 0.056 | 0.056 |
| $\sigma_{R1}$ | 0.031 | 0.055 | 0.044 |
| $\sigma_{RA}$ | 0.029 | 0.068 | 0.062 |

**B. Characteristic impedance and wavenumber**

Partners 1, 3, 4 and 7 also measured the characteristic impedance and complex wavenumber of the same sample (with the exception of Partner 4) and of different samples of each material.

Figs. 5 to 7 show the comparison of the real and imaginary parts of the normalized characteristic impedance and complex wavenumber (normalized by the wavenumber for air $k_0$) for all the 3 tested materials. Each curve is the average of the tests on the different samples. From the data, a consistency in the results between the participating partners is observed although must be an error in the 4-microphone transfer matrix approach[6] used by Partner 3 to invert the characteristic impedance. This approach is not regulated by a standard and it is prone to errors due to the imperfections in the quality of the anechoic termination, edge effect and microphone phase mismatch. The relative errors ( $\varepsilon_{\Re(z_c)}$ , $\varepsilon_{\Im(z_c)}$ , $\varepsilon_{\Re(k_c)}$ , $\varepsilon_{\Im(k_c)}$ calculated using Eq. 1) in the frequency range of 400-3500 Hz was found between 15% and 30% for the characteristic impedance and between 10 and 30% for the complex wavenumber. The deviation in the acoustical property for material A is mainly due to the frame resonance (Figs. 5(a) and 5(b)). The leakage effect between the material edge and tube wall can be the reason for the deviation observed in the case of material B (Figs. 6(a) and 6(b)). Material C is characterized by a higher deviation in the characteristic impedance and complex wavenumber across the whole frequency range which can be attributed to the variability in the mounting conditions in the impedance tube (Figs. 7(a) and 7(b)).

In particular, the tests on a single sample demonstrate that the maximum relative error for all tested materials was found to be lower than 4% for real part of the characteristic impedance, 14% for imaginary part of the characteristic impedance, 2% for real part of the complex wavenumber and 4% for the imaginary part of the complex wavenumber. When different samples of each material were tested, the relative error in data was found to be lower than 30%.

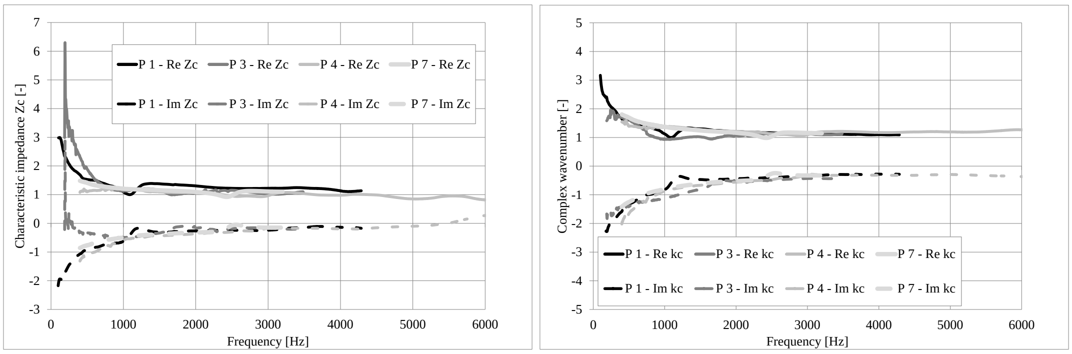


Figure 5 - The average of the real and imaginary part of the normalized characteristic impedance spectra (left), and real and imaginary part of the normalized complex wavenumber spectra (right) measured by each of the participating partners for material A.

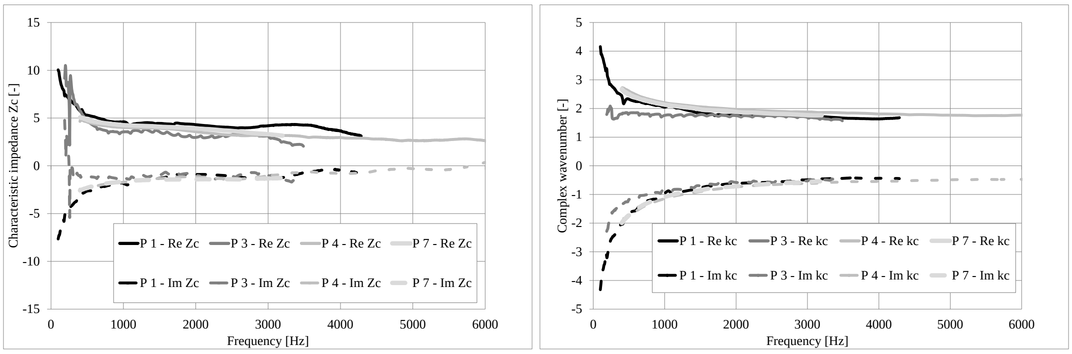


Figure 6 - The average of the real and imaginary part of the normalized characteristic impedance spectra (left), and real and imaginary part of the normalized complex wavenumber spectra (right) measured by each of the participating partners for material B.

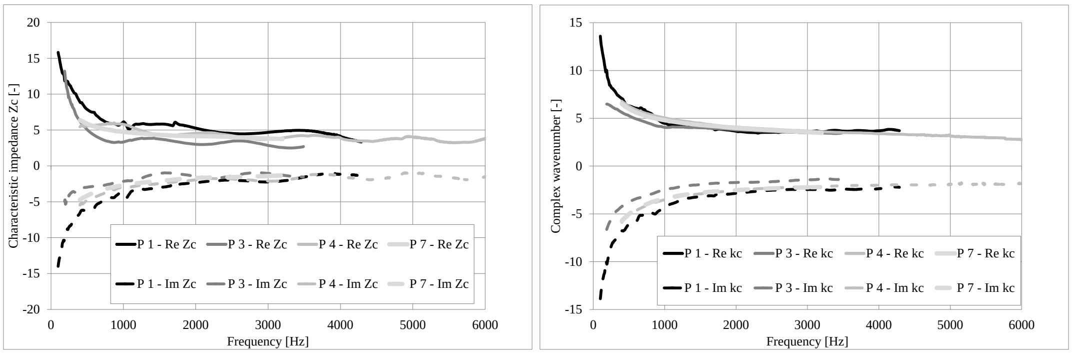


Figure 7 - The average of the real and imaginary part of normalized characteristic impedance spectra (left), and real and imaginary part of the normalized complex wavenumber spectra (right) measured by each of the participating partners for material C.

**C. Pore structure parameters**

In addition, the partners carried out tests on the same sample and on different samples for each material to determine the airflow resistivity, porosity, tortuosity and characteristic lengths. Fig. 8 shows the comparison between the average values of airflow resistivity measured for different samples by each of the participating laboratories. Table IX presents the standard deviations determined in accordance with ISO 5725-2 for airflow resistivity and open porosity. Here, the standard deviations calculated according to the ISO standards have been divided by mean value of the airflow resistivity and open porosity, respectively and data are expressed in percentage. As an example the mean repeatability standard deviation for a single sample for airflow resistivity and open porosity can be written as:

$$\varepsilon_{1,\sigma} = \frac{\langle \sigma_1 \rangle}{\bar{\sigma}} \times 100, \quad [\%] \quad \text{and} \quad \varepsilon_{1,\phi} = \frac{\langle \sigma_1 \rangle}{\bar{\phi}} \times 100, \quad [\%] \tag{12}$$

Similar expressions can be written for other quantities described in Eqs. (2)-(10).

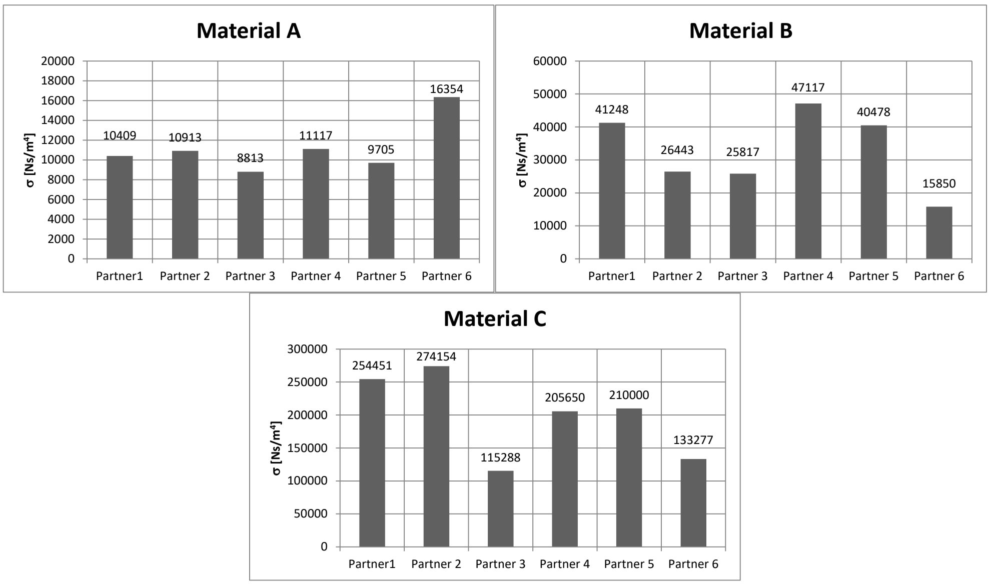


Figure 8 - The average of the airflow resistivity for material A (left), material B (right) and material C (center) measured by each of the participating partners.

The in-laboratory repeatability $\varepsilon_{1,\sigma}$ for the airflow resistivity measured using the same sample is within 1%. In the case of material A the in-laboratory repeatability for different samples $\varepsilon_{A,\sigma}$ of material A are lower than 7% while they can vary between 10% and 25% for materials B and C.

A similar analysis is presented for open porosity tests and Fig. 9 shows the comparison between average values on different samples for each participant. Tests on the same and different samples once again revealed good internal repeatability ($\varepsilon_{1,\sigma}$ lower than 1% for the same sample and $\varepsilon_{A,\sigma}$ below 6% for different samples). Also, comparison between different laboratories is satisfactory for materials A and B (lower than 7%) while measurements on material C from partner 6 (using a method based on ultrasonic surface reflection) seems to significantly underestimate the open porosity value.

From the data shown in the Table IX, it is possible to come to similar conclusions as for the sound absorption coefficient. In fact, for both quantities and for all the tested materials, the mean repeatability standard deviation for a single sample is lower than the mean repeatability standard deviation for several samples; in this case an important role is played by the homogeneity of materials while random errors seem to be negligible. Such results are confirmed by a relatively low value of the material standard deviation. The inter-laboratory standard deviation for a single sample is higher than material standard deviation and this suggests the occurrence of systematic errors for some of the laboratories. Reproducibility standard deviations for single and different samples range from between 10% to 45% for airflow resistivity and 1% to 10% for open porosity.

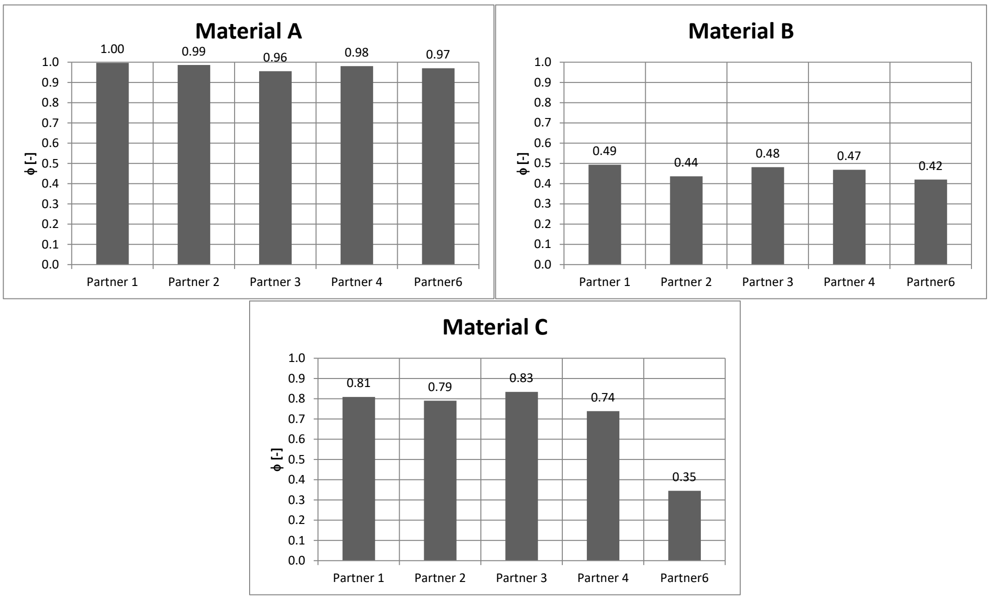


Figure 9 - The average of the open porosity for material A (left), material B (right) and material C (center) measured by each of the participating partners.

Table IX. The repeatability for the airflow resistivity and open porosity determined in accordance with to the ISO 5725-2[17].

| | Airflow resistivity | | | | Open porosity | | |
|---|---|---|---|---|---|---|---|
| % | A | B | C | % | A | B | C |
| $\varepsilon_{1,\sigma}$ | 1 | 1 | 1 | $\varepsilon_{1,\phi}$ | 0,5 | 1,1 | 0,4 |
| $\varepsilon_{A,\sigma}$ | 5 | 14 | 22 | $\varepsilon_{A,\phi}$ | 1 | 6 | 1 |
| $\varepsilon_{M,\sigma}$ | 5 | 14 | 22 | $\varepsilon_{M,\phi}$ | 0,4 | 6 | 1 |
| $\varepsilon_{I1,\sigma}$ | 10 | 31 | 29 | $\varepsilon_{I1,\phi}$ | 2 | 10 | 1 |
| $\varepsilon_{IA,\sigma}$ | 9 | 25 | 30 | $\varepsilon_{IA,\phi}$ | 2 | 6 | 3 |
| $\varepsilon_{R1,\sigma}$ | 15 | 30 | 45 | $\varepsilon_{R1,\phi}$ | 2 | 10 | 1 |
| $\varepsilon_{RA,\sigma}$ | 10 | 29 | 37 | $\varepsilon_{RA,\phi}$ | 2 | 9 | 3 |

Finally, Fig. 10 shows the comparison for average values of tortuosity and characteristic lengths obtained by participants. Here it is worth remembering that the direct tortuosity measurements were executed by partners 1 (on materials A and B) and by partner 6 (only one sample). The remaining data were obtained from the inverse estimation from acoustic data. In any case, the dispersions between different institutions for tortuosity are not negligible for material C (around 85%) while for materials A and B, the dispersion is lower than 15%. The dispersion for characteristic lengths varies between 20% and 80%.

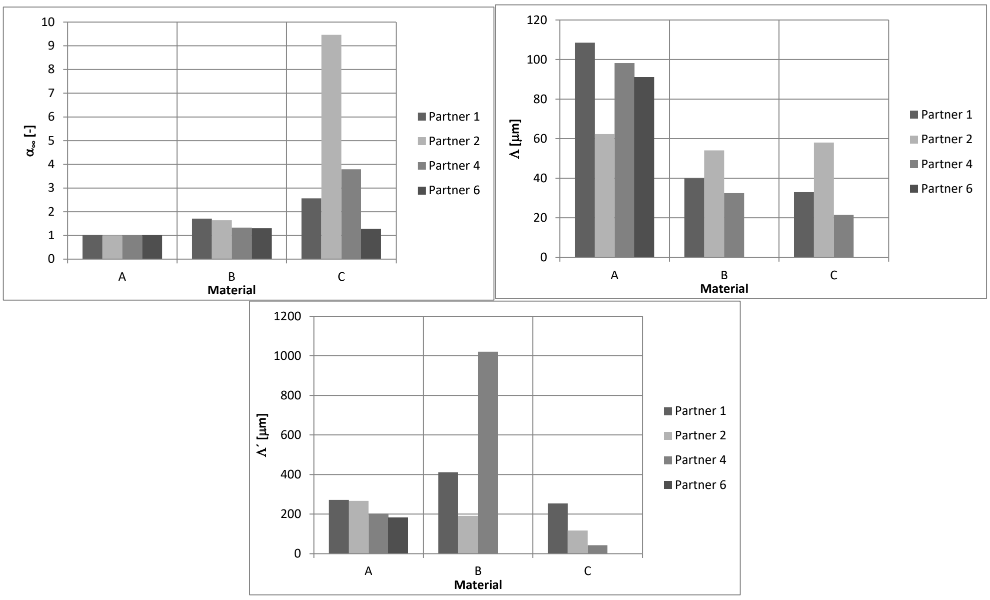


Figure 10 - The average of tortuosity (left), viscous characteristic length (centre) and thermal characteristic length (right) for all the materials measured by each of the participating partners.

## IV. CONCLUSIONS

The inter-laboratory tests on the acoustical and pore structure properties suggest a poor reproducibility between laboratories especially for the acoustical properties of highly resistive materials and granular materials with a rigid frame. The maximum relative errors in the absorption coefficient, real and imaginary parts of the surface impedance were found to be $\varepsilon_{\alpha} = 19\%$, $\varepsilon_{\Re(z_s)}=29\%$ and $\varepsilon_{\Im(z_s)}=13\%$, respectively. A major cause is likely to be the natural inhomogeneity in the material slab from which the samples were cut. Other causes can be the way the sample was actually cut and mounted in the impedance tube. These can lead to systematic errors between laboratories.

There is an obvious need for revision of the current standard[1] where no discussion of potential measurement problems, and no guidance on the installation of the samples is provided, no instrument calibration procedures or procedures for periodic verification of the instruments are detailed, no indications of the number of samples to be measured for the characterization of a material are given and the acceptability of a certain standard deviation on the tests conducted is not discussed.

No ISO standard exists to measure characteristic impedance and complex wavenumber. The inter-laboratory errors reach 30% and the causes are likely to be similar to those discussed earlier in these conclusions. It would be appropriate to extend the standards in refs. [1] to include the methodology detailed in ref. [6] for a more complete characterization of the materials in an impedance tube with 3 or more microphones.

There is a lack of standard to measure those pore structure parameters which are used routinely to predict the characteristic impedance and complex wavenumber of porous media. The only ISO standard in existence is to measure the air flow resistivity[3]. For this parameter, the in-laboratory repeatability is high ($\varepsilon_{1,\sigma}$ =1%). However, the reproducibility is reduced considerably to $\varepsilon_{RA,\sigma}$ =10% for a common poro-elastic material (material A) and to $\varepsilon_{RA,\sigma}$ =37% for a material with high airflow resistivity (e.g. material C).

The values of the inter-laboratory standard deviation determined in our experiments highlight the presence of systematic errors between laboratories, which may be due to the absence of periodic calibration of the static pressure transducers. This procedure is not included in the ISO 9053 standard[3]. This omission suggests that a revision of the ISO 9053 standard is desirable to reduce errors in the airflow resistivity measurements. One recommendation is to introduce a standardized porous sample with known and well predicted flow resistivity. Modern methods of 3D printing enable manufacturing of samples with highly reproducible porous structure and dimensions which enable the sample to fit in the flow resistivity tube perfectly.

The measurement of open porosity of poro-elastic materials is not described by any standard. In this paper, the isothermal compression of volume (Boyle's law) method (ref. [9]) was used by participating partners 1-4 to measure the porosity. The results show an excellent internal repeatability $\varepsilon_{1,\phi}$ <1.1%. The reproducibility error is $\varepsilon_{RA,\phi}$ < 9%. Partner 6 used the ultrasonic reflection method (ref. [10]), which seems to underestimate the porosity systematically by up to 45% in the case of material C (see Figure 9).

Similarly, the measurement of tortuosity and characteristic lengths of porous media is not described by any standard. In this work some of the partners used acoustical inversion methods to determine these parameters[13-15]. The reproducibility was relatively poor because of large dispersion in the tortuosity was observed in the case of material C. A considerable dispersion in the results was observed. As a general conclusion for such parameters, when a direct measurement method was applied errors were lower than 15%. On the contrary, the use of inverse method could lead to errors which could reach up to 80 %. These findings suggest that new standards are needed to define procedures for measurement of the related pore structure parameters of porous media.

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