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Sound absorption study of raw and expanded particulate vermiculites

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Abstract Expanded and raw vermiculite minerals were studied for their ability to absorb sound. Phase and structural characterization of the investigated vermiculites was found similar for both types, while morphology and surface properties vary. Sound waves reflect in wedge-like structure and get minimized, and later are absorbed totally. We found that thanks to porous character of expanded vermiculite the principle of absorption of sound into layered vermiculite morphology is analogous to principle of sound minimization in "anechoic chambers." It was found in this study that the best sound damping properties of the investigated vermiculites were in general obtained at higher powder bed heights and higher excitation frequencies.

1 Introduction

The noise is defined as undesirable sound, annoying and disturbing people in some cases even detrimental [1]. It is a negative phenomenon of any technically developed civilization. Noise elimination can be achieved using various approaches, e.g. by application of sound absorbing materials. Materials with porous, spongy and fibrous structures are considered as suitable materials to absorb sound [2–5]. For example, polyurethane foams and alumina foams with porosity of 88–90% have excellent sound absorbing properties [6]. Sound absorption of loose materials is influenced by many facts such as particle shape and size, thickness, excitation frequency and/or particle density.

Vermiculite is one of the materials used for sound absorption, especially expanded vermiculites [7] due to their low density combined with a relatively high structural strength, high physical–chemical stability and low cost [8]. Raw vermiculites are an industrially significant material occurring in nature. They are available, inexpensive and environment friendly with wide range of applications. They belong to group of 2:1 phyllosilicates, build on tetrahedral and octahedral sheets that form silicate layers characterized by a negative charge, which is compensated by hydrated cations in the interlayer space. Vermiculites are considered to be porous materials, and the variability of porosity originates from their morphology. Interlayer, interparticle and interagregate pores are distinguish in its structure; therefore, vermiculite contains pores of different sizes and shapes [9–11]. Vermiculites are easy to modify, which is broadening their applicability to various areas such as environment protection as sorbents [12, 13].

Expanded vermiculite is prepared by the temperature treatment of raw material at temperature above 700 °C. The structure and composition are altered, and a large proportion of semiclosed pores, in which can account for up to 90% of the particle volume, are formed [8, 9]. This material represents porous structure where acoustics

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performance is assumed by a large network of macropores and micropores created among the individual grains [8].

In our work, we aim to explain the principle of sound wave minimization and absorption for two different types of particulate vermiculites: raw natural mineral and expanded vermiculite unconsolidated material. Precise characterization of particulate materials structure and morphology is needed before the statement concerning acoustic characteristics could be listed.

2 Experimental

2.1 Materials and analytical methods of characterization

Two different vermiculites were used for this study: (1) raw Mg vermiculite denoted as sample "Ver raw" and (2) commercially expanded Mg vermiculite denoted as sample "Ver expand." Both materials were supplied from Brasilia (Grena, a.s., Czech Republic) and used as received without any treatment.

Several methods for characterization of material were used: particle size distribution (PSD) analysis (Laser Scattering Particle Size Distribution Analyzer LA-950, Horiba) and bulk density measurement, specific surface area (SSA) measurement and mesopores and micropores volumes analysis (Sorptomatic 1990), chemical composition using X-ray fluorescence spectroscopy (XRFS) (Spectro X-LabPro), phase and structure characterization using IR spectroscopy (FTIR spectrometer Nicolet 6700) and scanning electron microscopy (SEM) analyses (scanning electron microscope PHILIPS XL-30).

2.2 Methodology of sound absorption measurement

The material ability to absorb sound is most frequently expressed by the sound absorption coefficient α, which is given by the ratio of dissipated power to incident power in an investigated material [14].

Frequency dependencies of the normal incidence sound absorption coefficient of the investigated vermiculites were obtained by the transfer function method ISO 10534-2 [15–17] and were measured using two-microphone impedance tube BK 4206 in combination with three-channel signal pulse multianalyzer BK 3560-B-030 and power amplifier BK 2706 (all from Brüel and Kjær, Denmark) in the frequency range of 150–6400 Hz.

The complex acoustic transfer function H_{12} is expressed by the equation:

$$H_{12} = \frac{p_2}{p_1} = \frac{e^{k_0 \cdot x_2 i} + r \cdot e^{-k_0 \cdot x_2 i}}{e^{k_0 \cdot x_1 i} + r \cdot e^{-k_0 \cdot x_1 i}}, \quad (1)$$

where p_1 and p_2 are the complex acoustic pressures at two microphone positions, k_0 is the complex wave number, x_1 and x_2 are the distances between the microphones and the tested material sample, r is the normal incidence reflection factor [10], which is defined as follows:

$$r = r_r + i r_i = \frac{H_{12} - H_I}{H_R - H_{12}} \cdot e^{2k_0 \cdot x_1 i}, \quad (2)$$

where r_r and r_i are the real and imaginary components of the normal incidence reflection factor, H_I is the transfer function for the incident wave, and H_R is the transfer function for the reflection wave. The transfer functions H_I and H_R are expressed by the following formulas:

$$H_I = e^{-k_0 \cdot (x_1 - x_2) i}, \quad (3)$$

$$H_R = e^{k_0 \cdot (x_1 - x_2) i}. \quad (4)$$

Then the sound absorption coefficient α is defined by the equation:

$$\alpha = 1 - |r|^2 = 1 - r_r^2 - r_i^2. \quad (5)$$

Frequency dependencies of the sound absorption coefficient α of loose powder materials can be used in order to determine the noise reduction coefficient, the sound speed and the longitudinal elastic coefficient of powder beds.

The noise reduction coefficient (NRC) takes into account an influence of excitation frequency on the sound absorption coefficient. It is defined as the arithmetical average of the sound absorption coefficients of a given material at the excitation frequencies 250, 500, 1000 and 2000 Hz [18, 19]:

$$\mathrm{NRC} = \frac{\alpha_{250} + \alpha_{500} + \alpha_{1000} + \alpha_{2000}}{4}. \quad (6)$$

It was found [20] that the sound speed c of elastic wave through powder beds corresponds to the primary absorption peak frequency f_{p1} by the equation:

$$c = 4 \times t \times f_{p1}, \quad (7)$$

where t is the thickness of a given powder bed. The longitudinal elastic coefficient K of a powder bed is similar to Young's modulus of the material. It is proportional to the sound speed of longitudinal elastic wave and is defined as follows [17]:

$$K = c^2 \times r_b = (4 \times t \times f_{p1})^2 \times r_b \quad (8)$$

where ρ_b is the bulk density of the powder bed.

3 Results and discussion

3.1 Material characterization

Characterizing both materials, it was found that they have similar chemical composition and size of the particles. Results of chemical analysis, particle size measurement and other structural parameters are presented in Table 1. Both vermiculites are determined as Mg vermiculites with similar chemical composition. Particle size distribution analysis is represented with Gauss curve comparable for both studied materials (Fig. 1) and relatively similar average particle size; however, distribution curve for expanded vermiculite is broader showing the presence of much smaller particles size of few 100s µm (up to 2% of analyzed material) and on the other side of curve large particles bigger than 3000 µm (about 2% of analyzed material). This nonuniform particle size distribution differentiate studied material one from another in sense of granulometry.

The characteristics evolved due to thermal treatment of expanded vermiculite cause differences in density and specific surface area of studied materials.

Density is approximately 6 times higher for raw vermiculite while the specific surface area is more than 15 times smaller than expanded vermiculite. Remarkable differences are for porosity values, where expanded sample contains 8 × more mesopores than original raw mineral, but amount of micropores is comparable for both samples.

3.1.1 IR spectroscopy analysis

FTIR analysis confirmed that both samples are phyllosilicate materials [21] with a different occurrence of hydroxyl groups in their structures (absorption bands of 3300–3600 cm^{-1}), which corresponds to content of water molecules in structures of expanded and non-expanded vermiculites; see Fig. 2. Visibly disappearing band 3596 cm^{-1} of Ver raw sample for Ver expand is confirmation of reduction in crystalline water in the structure.

3.1.2 Micro-morphology characteristics

SEM analysis of both samples showed differences in the common morphology of non-expanded (raw) vermiculite and expanded vermiculite (Fig. 3). While raw vermiculite is formed by compact layers difficult to differentiate individuals, the layered character of expanded vermiculite is clearly visible. Expanded vermiculite open layer morphology corresponds with porosity characteristics, where greater number of macropores will give different physical properties and different values of acoustic testing. The size of layer openings can be estimated to values below 20 µm.

3.2 Acoustic and mechanical properties

Examples of the measured frequency dependencies of the sound absorption coefficient of the studied vermiculite samples for different material thicknesses in the frequency range of 150–6400 Hz are shown in Fig. 4. Results of

Table 1 Basic characteristics of raw and expanded vermiculites

Parameters	Ver raw	Ver expand
Oxides composition (% mass concentrations)		
SiO_2	41.940 ± 0.030	41.930 ± 0.030
MgO	22.400 ± 0.050	21.040 ± 0.050
Al_2O_3	9.760 ± 0.022	11.130 ± 0.020
Fe_2O_3	6.219 ± 0.008	7.450 ± 0.009
CaO	3.200 ± 0.007	3.272 ± 0.008
K_2O	1.117 ± 0.005	2.486 ± 0.008
Particle size distribution (µm)		
Median size	805.366	858.851
Mean size	818.090	846.985
Geo. mean size	705.934	516.147
Bulk density ρ_b (kg m^{-3})		
	973	164
Specific surface area (m^2 g^{-1})		
	0.82	15.74
Cumulative pore volume (cm^3 g^{-1})		
Mesopores	0.031598	0.22597
Micropores	0.00075824	0.0011263

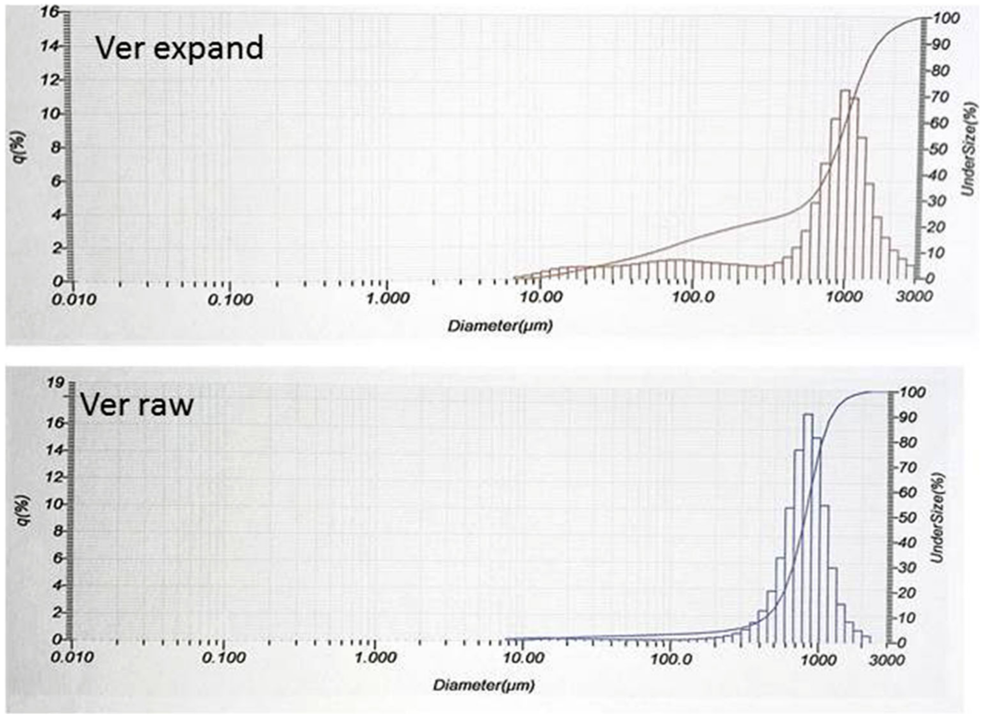

Fig. 1 Particle size distribution for both vermiculites

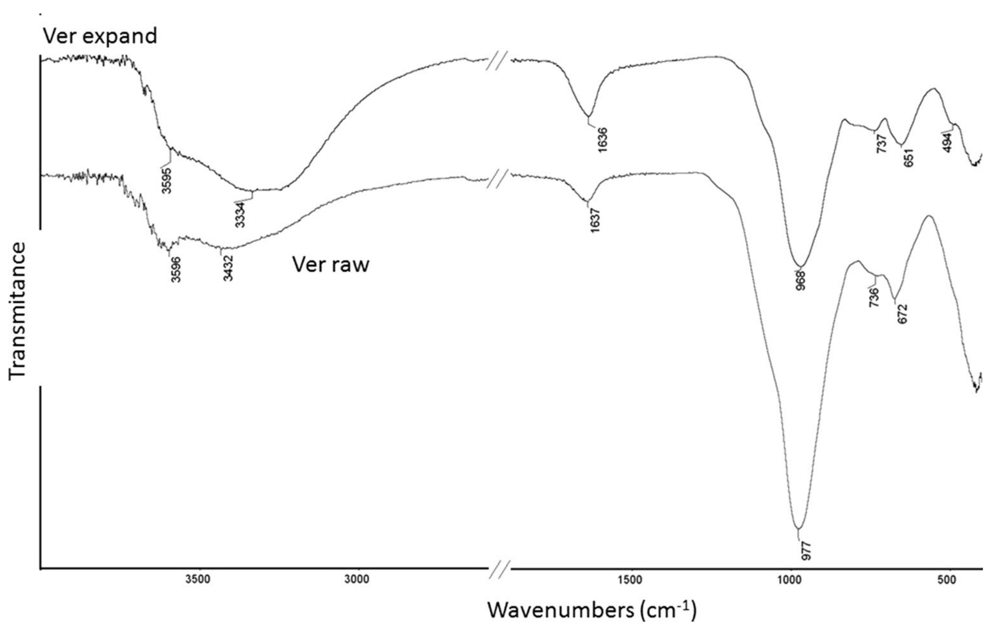

Fig. 2 FTIR spectra of both vermiculites—expanded (*upper*), raw (*lower*)

calculated and measured acoustical and mechanical properties are summarized in Table 2. It is evident (see Fig. 4) that the material thickness has a positive influence on sound absorption. It is visible mainly at low excitation frequencies as noted before in work [16]. The effect of the material thickness on sound damping corresponds with the observed the highest magnitude of the noise reduction coefficient NRC (see Eq. (6)) for all tested powder bed heights (see Table 2); it means that generally in thicker bed sound will be reduced greater. Better sound absorption properties of the investigated vermiculite samples are in general obtained at higher excitation frequencies. The maximum values of the sound absorption coefficient (i.e. $\alpha_{max} = 1$) are observed for the raw vermiculite samples with the thicknesses $t = 10$ mm and $t = 15$ mm. In the case of the expanded vermiculite, the maximum sound absorption coefficient magnitude of 0.96 was obtained for the powder bed of 7.5 mm.

The sound absorption curves of raw vermiculite are at lower range of absorption coefficient than expanded

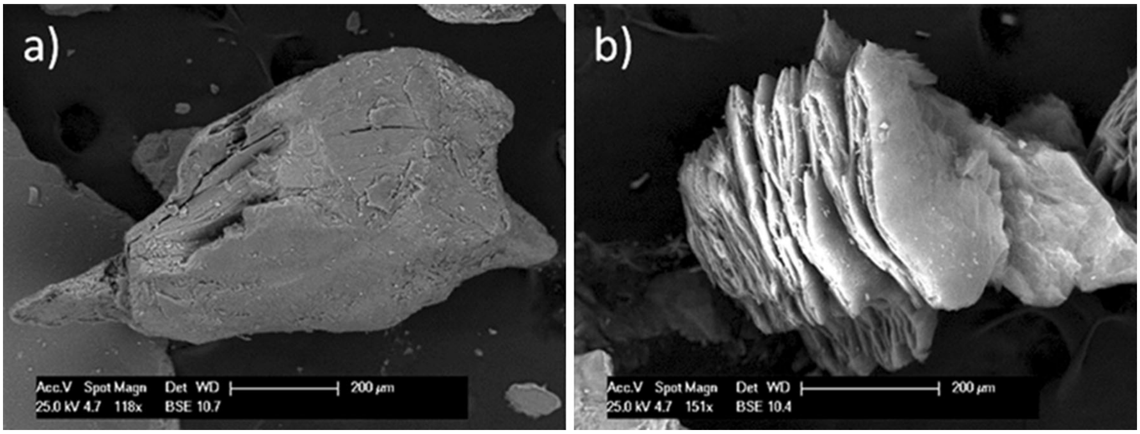

Fig. 3 SEM images of raw vermiculite (**a**) and expanded vermiculite (**b**) captured using back scattered electrons detector

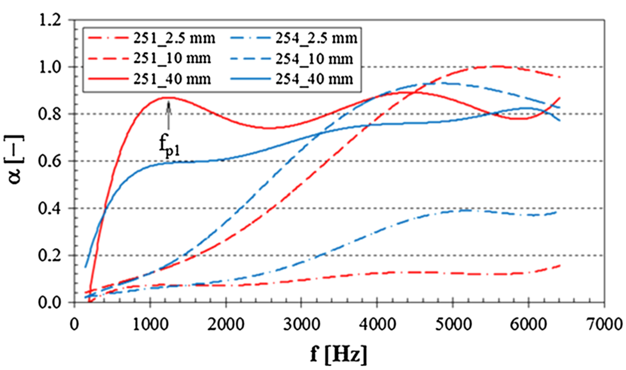

Fig. 4 Comparison of frequency dependencies of the sound absorption coefficient of the investigated vermiculites (raw vermiculite—251, expanded vermiculite—254)

vermiculite sample. There is one exception, raw vermiculite at 100-mm-thick layer shown higher α ~ 0.8 for full examined frequency range. This phenomenon is caused by relatively great thickness of the layer and compacted character of material. We can observe opposite situation of very low α for 2.5-mm-thick layer of Ver raw caused by inability of compacted thin layer to eliminate sound. In the transition area (i.e. for the thickness range t = <10; 30> mm), the sound absorption properties of both vermiculites are similar. The expanded vermiculite shows higher sound absorption at higher excitation frequencies [23] as well as at greater material thicknesses compared to the raw vermiculite.

In some cases, the measured frequency dependencies of the normal incidence sound absorption coefficient are characterized by the primary absorption peak at a characteristic frequency f_{p1} (see Fig. 4). It is evident from Table 2 that the primary absorption peak frequency f_{p1} was shifted toward decreasing excitation frequency with increasing power bed height.

The primary absorption peak frequencies were subsequently applied to determination of the sound speed c of an elastic wave through vermiculite beds and the longitudinal elastic coefficient K on the basis of Eqs. (7) and (8). It is

Table 2 Results of calculated and measured acoustical and mechanical quantities of studied vermiculites

Verm. type	Quantity	Sample thickness t (mm)										
		2.5	5.0	7.5	10	15	20	30	40	50	75	100
Raw	α_{max} (–)	0.16	0.44	0.85	1	1	0.96	0.89	0.92	0.87	0.87	0.86
	$f_{\alpha max}$ (Hz)	6400	6400	6400	5520	3704	2352	1456	1120	812	5912	6152
	NRC (–)	0.06	0.06	0.09	0.13	0.23	0.39	0.46	0.56	0.61	0.73	0.77
	f_{p1} (Hz)	–	–	–	5520	3704	2352	1456	1120	812	548	430
	c (m s^{-1})	–	–	–	220	222	188	175	179	162	164	172
	K (MPa)	–	–	–	7.99	8.09	5.80	5.00	5.26	4.32	4.43	4.85
Expand	α_{max} (–)	0.41	0.87	0.96	0.93	0.82	0.85	0.82	0.81	0.81	0.79	0.80
	$f_{\alpha max}$ (Hz)	4944	6400	6400	4904	2680	6072	6400	6312	6312	6400	6400
	NRC (–)	0.06	0.06	0.10	0.15	0.30	0.38	0.42	0.47	0.48	0.47	0.48
	f_{p1} (Hz)	–	–	–	4904	2680	2016	1288	952	–	–	–
	c (m s^{-1})	–	–	–	196	160	161	155	152	–	–	–
	K (MPa)	–	–	–	3.74	2.52	2.53	2.32	2.26	–	–	–

evident that the sound speed through vermiculite beds and the longitudinal elastic coefficient are in general decreasing with increasing material thickness. Higher magnitudes of these quantities were observed for raw vermiculite samples. It is caused by a higher density of the raw vermiculites compared to the expanded vermiculites. For this reason, the raw vermiculite is stiffer.

4 Discussion

Comparison of both types of minerals and their properties evolved during testing is giving interesting results for application of loose unconsolidated powder. We observed that the principles of sound absorption into layered vermiculite could be affected by both the phase composition of material, and morphology of particles. Although the mineral particles are in range from 100 to 3000 µm (in case of Ver expand), there might be analogy to principle of sound minimization in macro-world of "anechoic chamber" [24, 25]. The mechanism by which anechoic chambers minimize the reflection of sound waves impinging onto their walls is as follows: In the included Fig. 5, an incident sound wave I is about to impinge onto a wall of an anechoic chamber. This wall is composed of series of wedges W with height H. After the impingement, the incident wave I is reflected as a series of waves R which in turn "bounce up-and-down" in the gap of air A (bounded by dotted lines Fig. 5) between the wedges W. Such bouncing may produce (at least temporarily) a standing wave pattern in A. During this process, the acoustic energy of the waves R gets dissipated via the air's molecular viscosity, in particular near the corner C [22].

Both vermiculites are layered materials with porous structure. However, expanded vermiculite has greater porosity at macropores level (larger than 75 µm). Inspecting the character of morphology of expanded vermiculite, we can observe that it could be paralleled to geometrically perfect anechoic chambers. The expanded layers of vermiculite are forming wedge-like structure of several tens of micrometers wedge "entrance." Even though the incident sound wave is larger ($f = 200$–7000 Hz, $\lambda = 1750$–49 mm) than gaps in expanded layers of vermiculite, we presume that traveling wave could be partially absorbed just as in case macroscale wedges (Fig. 5). This absorption–reflection effect can take place on the particle as well as surrounding of particle, since mineral is forming non-compact sample. It is confirmed by calculated sound speed values that they are lower for expanded vermiculite, because sound wave is slowed down in wedge-like character of bulk material. Therefore, we can compare the sound absorption in particulate expanded vermiculite bed as set of randomly oriented micro-anechoic chambers (Fig. 6).

In case that we would be able to prepare oriented sample of vermiculite powder, the absorption of sound could be much greater reaching absolute values.

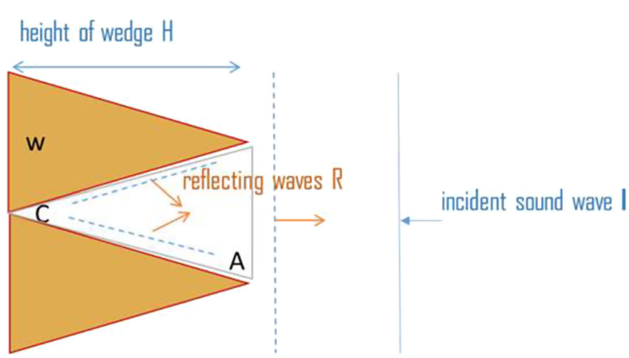

Fig. 5 Schema of sound wave minimization at wedges (w) construction of anechoic chamber's walls (C—corner of wedges, A—air atmosphere)

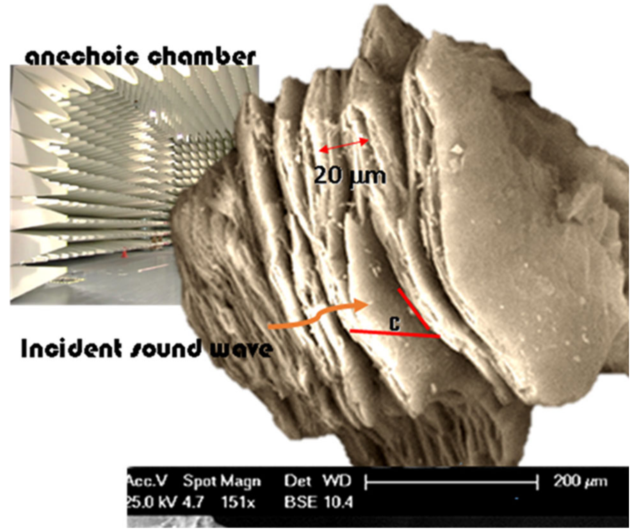

Fig. 6 Comparison of expanded vermiculite morphology with construction in anechoic chamber (C—corner of wedges as at schema Fig. 5)

5 Conclusion

The aim of this research was to investigate the effect of two vermiculites with similar chemical composition but different morphology on their acoustical and mechanical properties. One of the tested materials was a raw material; the second was thermally expanded vermiculite.

It was found that the vermiculite morphology characteristic has a significant influence on the investigated properties. The raw material is characterized by a higher bulk density, smaller particle sizes and lower values of cumulative pore volumes as was verified by physical methods. There is lower friction during acoustic wave propagation through raw material pores and smaller transformation of acoustic energy into heat compared to the expanded vermiculite.

It was verified that better sound damping was in general obtained at higher excitation frequencies and material thicknesses of both vermiculites.

On the basis of the measured frequency dependencies of the sound absorption coefficient, it was possible to determine the sound speed of the investigated loose vermiculites. The way, how sound wave gets absorbed in expanded vermiculite bed, could be compared to micro-anechoic chamber. Expanded vermiculite structure is buildup of wedge-like layers, where gap of entrance is size of several tens micrometers. In case that we would be able to prepare oriented sample of vermiculite powder, the absorption of sound could be much greater reaching absolute values.

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