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The Acoustic Performance of Expanded Perlite Composites Reinforced with Rapeseed Waste and Natural Polymers

Silviu Nastac [1,*], Petronela Nechita [2,*], Carmen Debeleac [1], Cristian Simionescu [2] and Mihai Seciureanu [1]

1 Research Center for Mechanics of Machines and Technological Equipments, Engineering and Agronomy Faculty, "Dunarea de Jos" University of Galati, 810017 Braila, Romania; Carmen.Debeleac@ugal.ro (C.D.); mihai.seciureanu@gmail.com (M.S.)

2 Research and Consultancy Center for Agronomy and Environment, Engineering and Agronomy Faculty, "Dunarea de Jos" University of Galati, 810017 Braila, Romania; csimionescu@ugal.ro

* Correspondence: snastac@ugal.ro (S.N.); petronela.nechita@ugal.ro (P.N.); Tel.: +403-7465-2572 (S.N.)

Abstract: Lignocelluloses residues from the post-harvest crop are receiving great scientific attention nowadays. Generally, the composite materials based on lignocelluloses waste present low density and weight, and better insulation properties compared with those petroleum-based. This study presents the results of experimental investigations regarding soundproofing capabilities for a composite material based on expanded perlite (EP) and natural polymers matrix (starch) reinforced with rapeseed stalks waste. The preparation of light-weight samples of composites was performed at room temperature through a mechanical mixing process of EP with starch polymers and rapeseed residues until optimum moisture content composition was obtained. Rapeseed stalks long fibers were avoided through the preliminary dry grinding procedure, and the composite was air-dried at room temperature for 48 h. Four samples of composites with different ratio of EP and rapeseed waste were considered. The evaluation of sample sound insulation characteristics was performed using the transfer-matrix method based on a four-microphone acoustic impedance tube. The paper concludes that the proposed composite provides comparative sound insulation capabilities to actual materials, with few particular aspects presented within the paper. Thus, these new materials are promising as a viable alternative to the actual large-scale utilization solutions in soundproofing applications.

Keywords: lignocelluloses waste; rapeseed stalks; expanded perlite; starch; composites; sound absorption; sound transmission loss; noise reduction coefficient

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1. Introduction

Currently, existing sound insulation materials are composed of petrochemical-based polystyrene or natural products of mineral origin, such as fiberglass and rock wool. Keeping in mind the sustainability concerns, natural or recycled materials would be able to reduce the consumption of non-renewable resources and simplify the processes of reuse or recycling. There are indications that the materials based on renewable resources could compete with conventional materials, and the local availability of these materials could also lead to a reduction of the economic and environmental impacts, mainly in the case of residues and by-products from agricultural and forestry industry [1]. Noise control is an important parameter to assure the environments with acoustical comfort. The utilization of materials with composite structures represents one of the most important factors in noise reduction. Generally, porous materials with low weight and density are very useful for products with appropriate soundproofing properties such as sound barriers, walls, or road surfaces. In order to meet the deficit between production and consumption, and to protect the environment without compromising the performance and quality of the product, recycling and waste recovery are considered viable solutions for the future [2]. Furthermore, the use of biodegradable waste and materials obtained from natural and renewable sources would be one of these [3–7].

Due to the environmental concern, in the last years considerable efforts have been placed towards the utilization of renewable resources to obtain alternative composite materials that are lightweight, biodegradable and biocompatible under well-defined environmental conditions. In this context, the valorization of biodegradable waste (recycled paper, agricultural residues, sawdust, etc.) in the structure of conventional building materials is a sustainable alternative to obtain lighter products with higher thermal and acoustic insulating performances compared with petroleum-based ones [8–11]. In particular, the introduction of natural fibers in sound insulation, gains an economy in terms of resources and energy, in the allocated time for material production, and fosters the recovery, reuse and recycling of the products prior to the final disposal. In addition, comparatively with natural fiber-based composites, synthetic insulation materials have a consistently higher global warming potential (GWP) [1].

The building sector is the most indicated technological activity to valorize the solid wastes, due to the large quantity of raw materials and final products used. Currently, the converting of industrial or biomass wastes into useful products, with maintaining the quality requirements at low cost and environmental impact, became a challenge for the production of building materials. Thus, the embedding of different types of wastes in the structure of building materials with sound and thermal insulating properties has been reported in many research studies [12–17]. A large field of natural materials is known as quickly renewable raw materials added as appropriate components to obtain the lightweight composite materials with application in building, such as sound or thermal insulators. Generally, most of synthetic polymers based on polystyrene, poly (vinyl chloride), polyurethane and poly (acrylic ester)s have implementation at an industrial scale, in order to minimize the noise, through properly acoustical isolation of these materials, like fiber-reinforced polymeric composites. However, we must also highlight the weaknesses of these materials, such as acoustic material cost (in terms of expensiveness) or potential human health hazards during normal utilization [18]. According to the standard ASTM C423—17, the sound absorption performance is divided into six categories see Table 1 having as criterion the sound absorption coefficients (SAC) [3,19].

Table 1. The categories of sound absorption coefficient (SAC) [3].

Sound Absorption Category	SAC Domain
A	0.90–1.00
B	0.80–0.85
C	0.60–0.75
D	0.30–0.55
E	0.15–0.25
F	0.00–0.10

In this context, the lignocelluloses residues from the post-harvest crop are receiving great scientific attention due to their wide variety of uses. These wastes have already been used to obtain different grades of composite materials (i.e., rice straw, rice husk, hemp husk, wheat straw, wheat husk, rapeseed straw, etc.) [20–22]. Moreover, due to their very good performance in sound absorption (Table 2), in different studies, it is reported that these natural fibers are widely used as reinforcement in industries for soundproofing composite panel manufacturing [23].

Table 2. The absorption performance of lignocelluloses fiber reinforced composite materials.

Type of Fiber Reinforced Composite Material	SAC	Frequency, Hz	Reference
Ramie fiber-reinforced composite with polylactic acid PLA	0.089–0.353	250–1600	Chen et al. [24]
Ramie fiber-reinforced composite with polylactic acid PLA	0.121	500–600	Chen et al. [24]
Banana fiber-reinforced composite with epoxy resin	0.11	500–600	Khusairy et al. [25]
Fiber-reinforced composite based on wood/ oil industry waste and formaldehyde resin	0.8–0.9	400–3200	Bratu et al. [26]
Ramie, flax and jute fiber-reinforced composite with epoxy resin	0.6–0.65	2000	Yang et al. [27]
Sugarcane bagasse fiber-reinforced composite with polyester resin	0.63	4000	Abdulah et al. [28]
Banana fiber-reinforced composite with polyester resin	0.68	4000	Abdulah et al. [28]
Rice straw fiber-reinforced composite with polypropylene	0.008	2000	Jayamani et al. [29]
Kenaf fiber-reinforced composite with urea	0.065	2000	Jayamani et al. [29]
Sisal fiber-reinforced composite with PLA	0.085	2000	Jayamani et al. [30]
Coconut/coir fiber-reinforced composite with epoxy resin	0.078	6000	Jayamani et al. [30]
Flax fiber-reinforced composite	0.96	250–10,000	Zang et al. [31]
Basala wood fiber-reinforced composite	0.58	250–10,000	Zang et al. [31]
Hemp husk waste-reinforced composite with hydrated lime	0.15–0.18	4000–6000	Fernea et al. [32]
Hemp husk waste-reinforced composite with hydrated lime	0.22–0.25	1000	Fernea et al. [32]

Generally, composite materials based on lignocellulosic waste have low density and higher insulation properties in respect to those that contained petroleum [33]. These residues are nonabrasive, present high filling levels that possibly result in high stiffness properties, have high specific properties, are easily recyclable, biodegradable, have low energy consumption and a low cost. Compared to conventional fibers, it should be noted that lignocellulose fibers do not cause skin irritation, cancer or other health problems [34].

Rapeseed, scientifically known as *Brassica napus* L., is a very important and widely cultivated arable crop throughout the world for the production of animal feed, vegetable oil for human consumption and the production of biodiesel for acting engines of self-propelled equipment. Rapeseed, also known as canola, has achieved a worldwide commodity status, being extensively cultivated in Europe, Asia and North America, due to the policies of encouraging the production and use of biofuels [35].

Rapeseed stalks are residues that remain on agricultural lands after seed cropping. These residues represent about 2/3 of rapeseed production and are considered an important biomass resource, insufficiently exploited in Romania. For example, in 2014, rapeseed production was 738,900 tons and the number of rapeseed stems was about 492,600 tons [36]. The farmers are only interested in the rapeseed seed and consider stems as a zero–value byproduct. In the majority of cases, these agro-residues are used as livestock bedding or incinerated with the consequent CO_2 emissions. Therefore, if this waste is used as reinforcement in composite materials, the added value of rapeseed would be extended.

The advantages, from a technical and economic point of view, regarding using perlite aggregates as a building material, are highlighted in many kinds of research, this being widely used as loose-fill insulation in masonry construction, due to its outstanding insulating characteristics and light weight [37]. Furthermore, the significant characteristics, such as a high porosity degree, large surface area, high resistance to fire, low degree of sound transmissibility, low rate moisture retention, low environmental impact and cost effectiveness lead to the fact that perlite provides more valuable and inexpensive matrix solution for preparing composite materials [37]. Perlite aggregate is a mineral rock with a chemical composition based on the two main components: silicon dioxide (70–80%) and alumina (12–16%) [38,39]. The perlite is a volcanic rock similar to glass, which, by fast heating at 870–1100 °C temperatures, expands in a manner similar to popcorn, increasing its volume 4 to 20 times compared to the original value. As result, lightweight spheroids are obtained, with a volumetric density of about 120–250 kg/m^3, compared with 2000–2500 kg/m^3 for natural perlite. Expanded perlite is inert and water-insoluble. It is presented as a white powder with fine particles, and sound insulating properties about 18 dB at 125 Hz Figure 1 [2,8].

Figure 1. Several structural aspects of perlite: (**a**) rock; (**b**) ground; (**c**,**d**) expanded form.

Given the versatility of expanded perlite, it is successfully used worldwide in the manufacture of sound blocking and sound absorbing products. The cellular aspect of perlite structure does not allow for sound propagation, thus being recommended for filling the gaps in structure, used for sound blocking and sound-absorbing solutions. For an alternative combination of layers and structures, through the interconnected pore of acoustic panels and cavity sound absorption, the sound waves are progressively absorbed in many levels, enabling the superior performance of this kind of sound absorption/blocking complex materials [39]. In consequence, the expanded perlite has usually applied as a primary constituent, especially in lightweight sound insulating panels manufacturing. Therefore, the intrinsic porous nature of lightweight expanded perlite absorbs sound waves and minimizes the reverberation time [39]. In addition, perlite can also be used as filler in spray-on absorbers products because in the mixing process with a binding agent and water, occurs a soft lightweight material with a coarse surface texture and high sound absorption characteristics [40]. In some cases, a spray-on absorber based on perlite acquires a noise reduction coefficient value of 0.70 [41].

Most literature reviews report the utilization of expanded perlite aggregates as filler, to improve the filtering performances of depth filter composites, used for retaining contaminants from alimentary liquids, and only a few of them are related to the use of perlite as a matrix for lignocellulose waste within reinforced composites for insulating applications [42–44].

In their research, Maslowski and coworkers (2019) had obtained hybrid composites based on lignocellulose straw and perlite, for reinforcing of natural rubber. By the mechanical modification of straw waste and their mixing with smooth particles of perlite aggregates, hybrid structures are obtained with improved mechanical strength and hardness. In addition, these composites exhibit and improve tearing durability and aging resistance, as well as the damping performances, when are used in insulating applications. The utilization of straw waste and perlite mixture as filler for natural rubber has, as a result, the improving of the barrier properties by vulcanization [45].

In other studies, Palomar et al. assess the effect of perlite and cellulose fibers on the structural and mechanical properties of lime-cement mortar composites for acoustic insulation application. The obtained results emphasized that the perlite content refines the mesopores network and increases the volume of macropores due to the filler effect of the crushed particles. The amount of cellulose fibers increases the thickness of the lime cement layer and reduces the volume of voids, with an effect on modifying both the thermal and acoustic properties [46].

Zhang et al. (2020) and Rozlowski et al. (2008) report the improvement of flammability properties of the plant fiber-reinforced composites, by using inorganic fillers, such as clay or perlite [47,48].

Nowadays, because a lot of bio-based polymers are commercialized at a large scale, it is expected a scientific interest to encourage new investigations on their future application as matrices for bio-composites (fatty acids, ricin-oleic acid, starch, isoprene, etc.). In this respect, starch is a widely available and inexpensive agricultural raw material, and a promising biopolymer used in the production of bio-composite materials, due to its availability and high biodegradability. In many studies, researchers found a high degree of compatibility between natural fibers and starch, which provides the greatest stiffness [49–51]. In addition, its thermoplastic derivate could be a proper solution to substitute synthetic polymers (usually supplied from thermoplastic or thermosetting resins) as matrix within composite materials structure [52].

In order to be used as biopolymer into the structure of the composite material, starch is solubilized in cold water and cooked at 90 °C for about 20 min, when the gelling phenomenon occurs. As a result of temperature increases, the viscosity of the water-starch mixture suddenly increases and this becomes transparent (Figure 2). Furthermore, at high temperatures (90–180 °C) and at high shear rates, the starch melts and flows, allowing its use in extrusion, injection or molding processes, similar to synthetic thermoplastic polymers.

(**a**) (**b**)

Figure 2. Composite structures based on starch: (**a**) rapeseed residues with starch; (**b**) gelled starch.

The chemical structure of starch is based on two main components: amylose and amylopectine, which differ in terms of proportion, depending on biomass sources. Amyloses are linear components with few side chains, formed by 500 to 2000 glucose units, whilst amylopectines are branched structures containing from 10,000 to 100,000 glucose units. The adhesiveness of a starch polymer is given by the high content of amylopectine, and this predicts its performance for improving the strength when it is used in composite materials structure.

Generally, the biopolymers based on starch are the most frequently present bio-additives for cellulose fibers in the paper industry, being widely used for both wet-end, and surface or coating applications. In their native form, starch can be used as a sizing agent (in size press of paper machine) while, after chemical, physical, and/or enzymatic modifications, it can be used as a dry strength additive for improving the paper mechanical properties or as coating additive due to its excellent film-forming ability. Based on their chemical structure, when is used in combination with cellulose fibers starch, it improves the fibrous network resistance due to the formation of a high number of hydrogen bonds. Additionally, by supplying mineral pigments (hyper platy nano clays, montmorillonite, perlite, etc.) into the starch suspension, its hydrophobicity is improved and, as result, the water barrier properties of the composite formula is increased [53,54].

Therefore, there is an urgent need to identify and develop sound absorption materials, reasonably priced, eco-friendly and biodegradable. Thus, an economy of non-renewable resources and non-biodegradable materials for acoustic applications could be obviously possible. In this way, the impact and direct implications are to ensure the protection of the environment from sources of significant pollution and noise [3].

In this paper, the flat-pressed composite materials, based on EP and starch, reinforced with rapeseed stalks waste, were obtained at laboratory scale. The purpose of this study was concerning the evaluation of different characteristics of these composite materials, such as sound absorption and sound transmission loss, water absorption and their structural properties, versus other commercial insulation materials based on petroleum (e.g., polyurethane-based materials).

2. Materials and Methods

2.1. Materials

Lignocellulose wastes based on rapeseed stalks, free of leaves and roots, were collected after the seed harvesting process, placed in Braila County, Romania. According to literature reported results, the main components of rapeseed stalks are 39.9% cellulose, 23.4% hemicelluloses, 21.5% lignin and 6.8% extractives, respectively [35].

Expanded perlite–Harbolite 350–was purchased from the World Mineral Company, in particulate form (with 25 µm particle size and white color), with bulk density 256 g/L and thermal conductivity 0.04–0.047 W/mK.

Corn starch was purchased from Agrana Company, Romania, having 24% amylose and 76% amylopectin content, and used as water dispersion with 25% performed to cook of starch at 90 °C for 20 min.

In order to facilitate the comparative analyses of the results, a high-density polyurethane sample with 96 kg/m^3 density and 15 mm thickness, was considered as a reference sample.

2.2. Methods

2.2.1. Preparing the Lignocellulose Residues

The agro-residues based on rapeseed stalks were air-dried and stored in a dry location. In order to homogenize the particles size and to avoid the presence of long fibers, the rapeseed stalks were dry-ground, using a laboratory apparatus presented in Figure 3. Thus, small amounts of particulate material, with 1–3 mm particles size, have been obtained.

Figure 3. Dry grinding of rapeseed stalks waste.

2.2.2. Preparing the Rapeseed Waste Reinforced Composites

With the above-prepared materials, the lightweight samples of composites were manufactured at room temperature by mixing expanded perlite with rapeseed residues and starch polymers, in order to obtain a pasty composition with optimum moisture content. It should be mentioned that this pasty was molded into a metallic form (with dimensions 10 × 10 × 1.5 cm). After this phase, the molded material was subjected to a technological pressing process (for 5 min, until 100 bars) using a laboratory uniaxial press (Figure 4), in order to avoid the achievement of an uneven structure material.

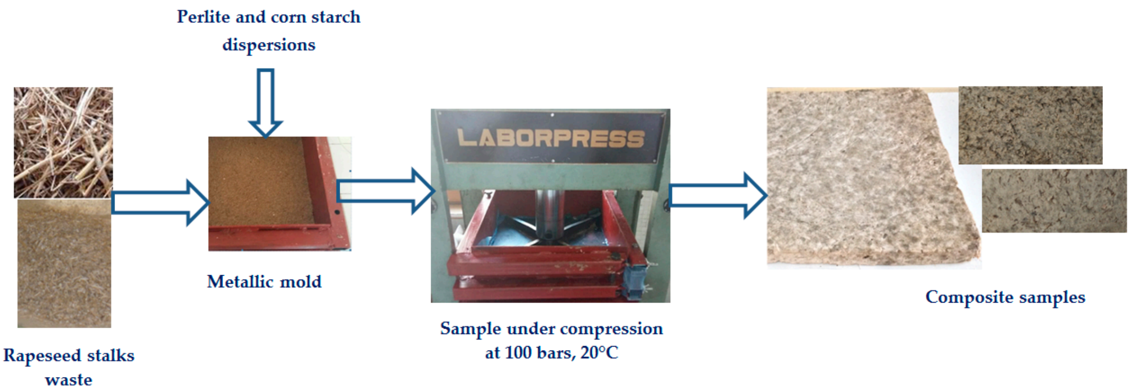


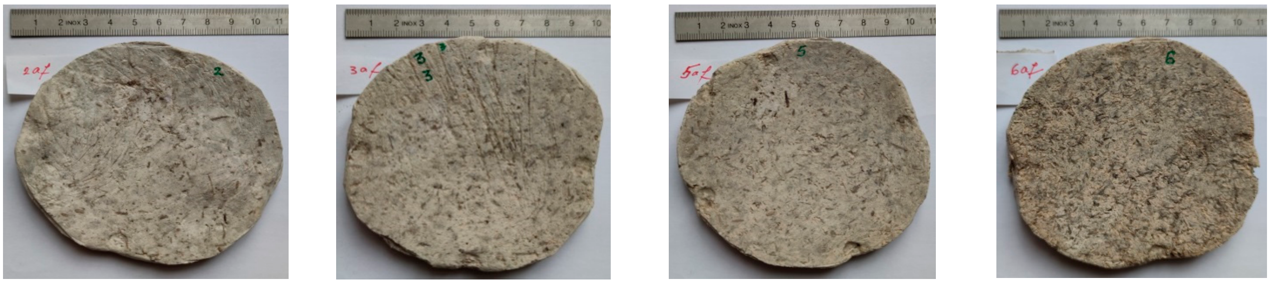
Figure 4. The schematic diagram for obtaining composite samples.

The composition of samples with different mixing ratios of EP, rapeseed waste and corn starch are presented in Table 3.

Table 3. The composition of experimental samples.

Material	P2af	P3af	P5af	P6af
Expanded perlite, %	76	72	56	40
Lignocellulose residues from rapeseed stems, %	4	8	24	40
Corn starch, %	20	20	20	20
Dimensions, cm	10 × 10 × 1.5	10 × 10 × 1.5	10 × 10 × 1.5	10 × 10 × 1.5

Laboratory samples of composite materials were subjected to an air-drying process for 48 h at room temperature (Figure 5).



(**a**) (**b**) (**c**) (**d**)

Figure 5. Dry form of rapeseed waste reinforced composites prepared for sound absorption tests within acoustic impedance tube: (**a**) P2af; (**b**) P3af; (**c**) P5af; (**d**) P6af.

2.2.3. Acoustic Methods

An experimental laboratory setup with a four-microphone acoustic impedance tube configuration was considered in order to assess the sound insulation characteristics of both composite samples and reference. Two different constructive variants of acoustic tubes were considered, having 100 mm and 28.5 mm diameter, respectively (Figure 6). The frequency characteristics of these devices are 10–2400 Hz for the large tube, and 200–7000 Hz for the small tube. Both devices have a complete four-microphone setup configuration and a floating anechoic termination. The evaluation of sample sound insulation properties was performed using the transfer-matrix method, according to SR EN ISO 10534-2:2005 and specifications within international standards ASTM E2611 2006, ISO 10534-2:1998, ASTM E90 and ASTM C423 [19,55–58].

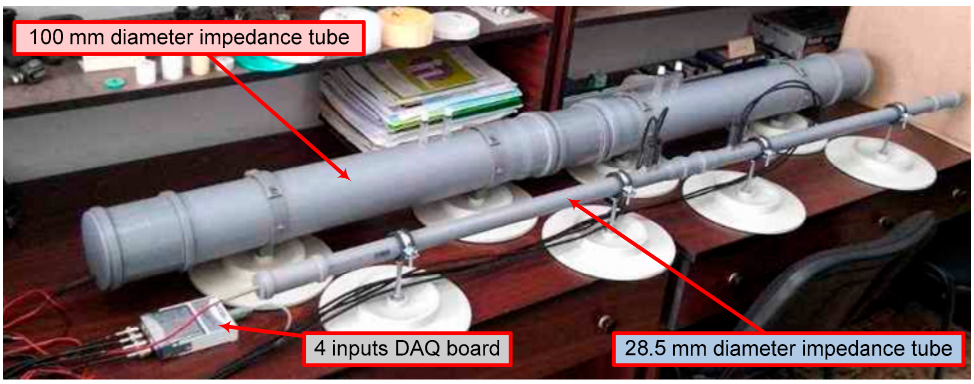


Figure 6. Acoustic impedance tubes used into experimental investigations.

The two-load method, using two different tube loads, such as "nearly anechoic" and, respectively, free termination, were used, in order to evaluate the transfer-matrix terms. It has to be noted that the experimental acoustic tubes have only four acquisition channels, thus that the first microphone, which is nearest positioned by noise source into the upstream tube, was imposed as the reference input accordingly to the method Figure 7 [4,11].

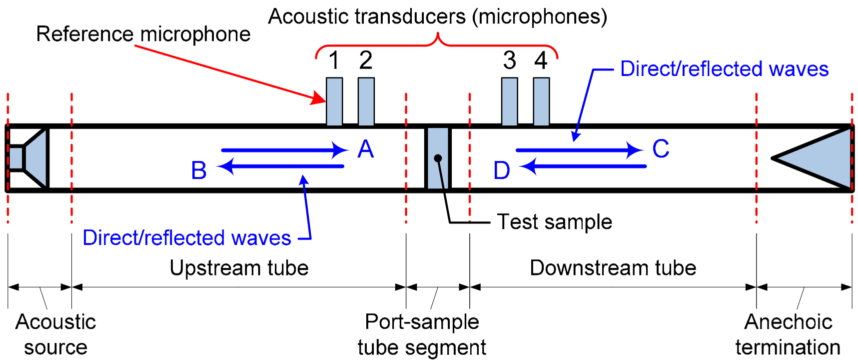


Figure 7. Schematic diagram of four microphones acoustic tube principle.

The acquisition of raw sound signals was performed with the help of National Instruments NI-9233/9162® pair hardware devices and four PCB–130E20 ICP® Electret Array Microphones Figure 6. In order to provide the highest accuracy of acquired signals, a 25 kHz sampling rate was used. Advanced signals processing, according to the standardized transfer-matrix method, was performed based on an application developed into Matlab® software [4,11].

After the acquisition of the four sound pressure signals, in sample upstream and downstream tubes, respectively, the sound transmission loss, sample impedance and sound absorption and reflection coefficients at normal incidence were evaluated. Sound transmission loss and sound absorption characteristics were adopted to be presented in this paper. These characteristics will allow a global and specific identification of proposed composites and their availability for embedding into the practical soundproofing applications [11,17,59–62].

2.2.4. Structural and Water Absorption Properties

Structural and water absorption properties were assessed by density measurements and optical micrographs analysis. An optical microscope was used to capture images aiming to evaluate the internal network structure of composite samples. The micrographs (further on presented in Section 3.2) were obtained using the BRESSER Erudit DLX optical microscope, with achromatic DIN-objectives, coarse and fine focusing drive, and coaxial mechanical desk with nonius. Images were captured with a full HD digital camera (1920 $\times$ 1080 Pixels, 5.86 $\times$ 3.28 mm sensor size).

The water absorption capacity was evaluated by measurement, meaning the weight difference between the dry weight of composite material (denoted M_1) and the weight of samples saturated with water for 24 h and maintained at 27 °C (denoted M_2) [63]. It was calculated with the help of Equation (1)

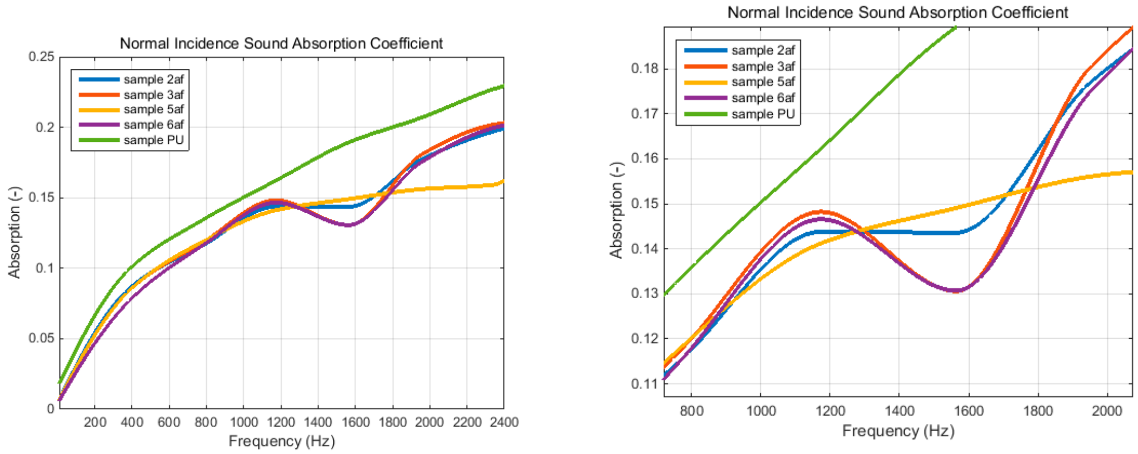
$$W = \frac{M_2 - M_1}{M_1} \times 100. \quad (1)$$

3. Results and Discussions

3.1. Acoustic Properties

The effect of rapeseed waste reinforcement quantity in the composite structure on the sound absorption coefficient and sound transmission loss was analyzed. The main objective was to develop green and lightweight composite materials from natural materials to be used in lightweight panels for sound insulation.

Diagrams within Figure 8 present the sound absorption coefficient at normal incidence according to results of experimental investigations based on a 100 mm diameter impedance tube. The basic diagram (Figure 8a) respects the specific frequency interval of the acoustic tube (10–2400 Hz). Taking into account the general trends of all graphs, discussion has to be conducted separately on the two frequency intervals 10–1200 Hz and, respectively, 1200–2400 Hz. For the first range, tested composites present about the same evolution as the reference sample. The differences, in terms of sound absorption, were negligible, starting from 0.010–0.012 at 10 Hz and growing up to 0.015–0.020 around 1200 Hz.



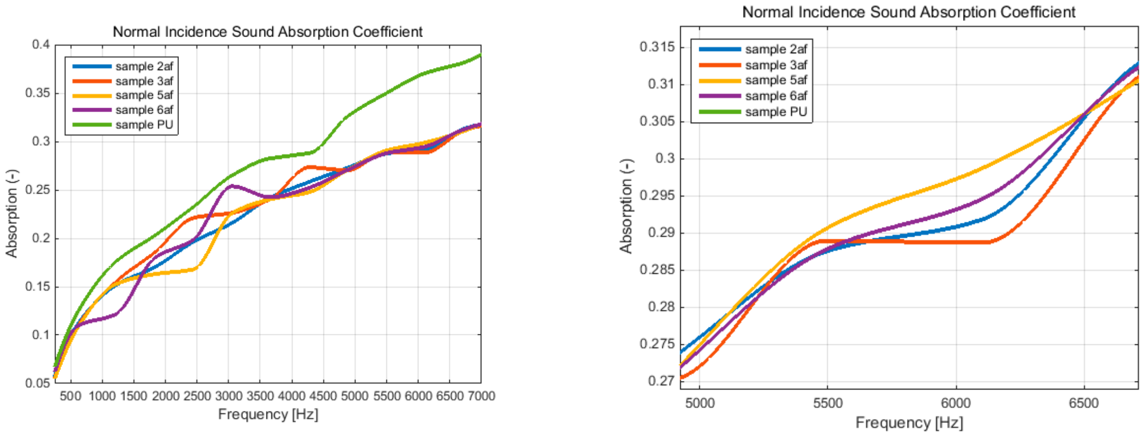
(**a**) (**b**)

Figure 8. The sound absorption coefficient at normal incidence gained by 110 mm diameter acoustic impedance tube: (**a**) overall and (**b**) detailed views, respectively.

All proposed composites respect the general increasing trend on this frequency range, with relative maximum differences between them around 400 Hz (in terms of 0.01 value). Passing on the next frequency interval, the analysis had been done both on the basic diagram —see Figure 8a— and the detailed view—see Figure 8b. This detail shows the graphs evolutions beyond 1200 Hz, where each material presents some different evolutions. Thus, the samples 3af and 6af, which have a relative maximum value of SAC at 1190 Hz, present a large decrease to 1585 Hz, after that resuming the general increasing trend. At the end of the analyzed frequency range, these two materials present greater values of SAC (around 0.2 at 2400 Hz). At the same time, the sample 5af presents an evident decreasing trend beyond 1200 Hz, with 0.03 SAC under the rest of the composites. Sample 2af trends to a constant evolution for 1100–1600 Hz range, with SAC = 0.145, and respects the general increasing trend outside of this interval.

Concluding previous aspects results that all samples of rapeseed waste reinforced composites provide good absorption for low frequency and adequate absorption for medium frequency range till 2400 Hz and could be compared with a reference sample (high-density polyurethane sample). In recent studies, Tuffail et al. (2021) report the sound absorption coefficient of about 0.16 for sugarcane reinforced composite (with 20% fiber content) at 1400 Hz frequency, and about 0.13 for cotton fibers reinforced composite at the same fiber content and frequency [3].

In addition, the evolutions of sound absorption coefficient in respect to frequency, based on experiments within the 28.5 mm diameter acoustic tube, were presented in Figure 9. The diagrams obviously respect the specific frequency range of this tube (200–7000 Hz).



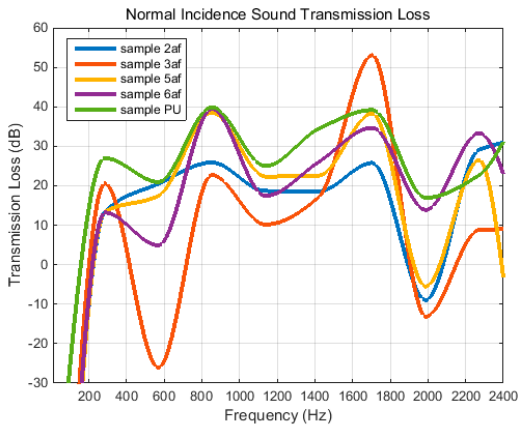
(**a**) (**b**)

Figure 9. Sound absorption coefficient at normal incidence gained by 28.5 mm diameter acoustic impedance tube: (**a**) overall and (**b**) detailed views, respectively.

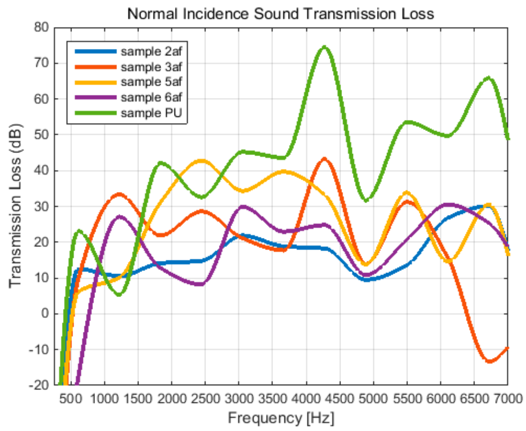
Unlike the large tube situation, the results within a small diameter tube have about the same general growing up tendency—Figure 9a. Certain small differences have been provided by each composite sample in the frequency range 1000–5000 Hz, but with small values, in terms of ±0.05 SAC deviation from the mean. The trend of sample 2af clearly presents minimum deviations from the mean trend and provides an absorption value of between 0.01 and 0.08 smaller than the reference onto entirely analyzed frequency range. Comparatively, each of the other composites has few peaks as follows: sample 3af around 2400 and 4200 Hz, sample 5af presents a single peak at 3000 Hz, but a significant constant evolution on 1500–2500 Hz, and sample 6af presents a relevant peak at 3000 Hz, but also a valley on 500–1200 Hz. Regarding the high-frequency interval, around 6000 Hz, it can be observed on graphs within Figure 9a that all composites present the same small decreasing tendency. This aspect has minor but clear differences between each material (see detail view on Figure 9b). The maximum deviation has been provided by sample 3af and the minimum by sample 5af. Overall, taking into account the investigations with small diameter acoustic tube it can be concluded that rapeseed waste reinforced composites provide adequate absorption for the entire frequency range, with a good absorption coefficient for frequencies greater than 3000 Hz.

Combining the two partial concluding remarks related to both acoustic tubes, result that the materials analyzed within this study provide good absorption characteristics for low frequencies till 800 Hz and for high frequencies beyond 3 kHz, with minor, up or down deviations for medium frequency range.

The second evaluated characteristic was the sound transmission loss (STL) at normal incidence. This parameter takes into account both the absorption and the other specific behavior of the tested sample (reflection at the sample surface, transmission, impedance, etc.). Thus, the evolution of STL can reveals additional specific aspects related to sound insulation applications. Diagrams within Figure 10 represent the STL acquired based on each acoustic tube. First diagram—Figure 10a—denotes that the reference sample and all composites have about similar evolution for 200–1800 Hz frequency interval, with few deviations as follows: strong attenuation of sample 3af and slightly attenuation of sample 6af around 600 Hz, and the maximum peak of STL value provided by sample 3af at 1700 Hz. All tested samples provide significant attenuation around 2000 Hz followed by slightly characteristics till the end of the interval. Nevertheless, the sample 6af presents a relative high evolution (in a range of 5–40 dB) for the entire frequency domain, at the same time that samples 2af and 5af provide good transmission loss till 1800 Hz (within this interval, the sample 2af provides the most smooth evolution).



(**a**)



(**b**)

Figure 10. Sound transmission loss at normal incidence gained with (**a**) 110 mm and, respectively, (**b**) 28.5 mm diameter acoustic impedance tubes.

Passing to the diagrams within Figure 10b, related to the 500–7000 Hz frequency range, the characterization of each sample becomes more evident. Thus, in the order of sample number, the evolutions denote that 2af composite provides a smooth characteristic on the entire range, but with lower STL values; 3af composite provides good characteristic for the medium-high frequency range between 1000 and 6000 Hz; 5af composite provides the best STL values for the medium frequency range (2000–4500 Hz); 6af composite provides roughly similar characteristic with 3af sample, but it covers high frequencies up to 7000 Hz and presents a slight attenuation around 2500 Hz.

The apparent different behavior of the 5af sample in terms of absorption, which is evident especially in Figure 8, beyond 1200 Hz up to 3000 Hz (see Figure 9 for the upper limit), is difficult to explain supposing only absorption diagrams. However, considering that the STL diagram in Figure 10, where sample 5af presents a good behavior especially for medium frequencies, results that this sample enables high reflection characteristic within this frequency range. Additionally, an explanation can be constituted by the differences of normal incidence surface quality of the samples, which was not quite identical for all samples (it results from the sample preparation procedure).

It has to be mentioned that the sound-absorbing capabilities of materials are usually indicated, graphically or tabular, by SAC values for different frequency ranges. This is quite a complex way of sound insulation characterization. Concurrently, a single number NRC (Noise Reduction Coefficient) rating is frequently used within product literature and specifications, in order to indicate the general ability of certain materials to absorb sound. In fact, the NRC is the average of the SAC coefficients evaluated at 250, 500, 1000, and 2000 Hz, according to Equation (2), and rounded off to the nearest 0.05 [64].

$$\mathrm{NRC} = \frac{\mathrm{SAC}_{250} + \mathrm{SAC}_{500} + \mathrm{SAC}_{1000} + \mathrm{SAC}_{2000}}{4} \quad (2)$$

Raw values evaluated with Equation (2) were comparatively presented in Figure 11, respectively for large and small acoustic tubes. Applying round on procedure, to the nearest 0.05, results that all composites have NRC = 0.10, excepting the sample 3af (for small tube tests) that is 0.15 (equal with the NRC of reference sample). Nevertheless, analyzing the raw results presented in Figure 11, it can be clearly observed that the composite samples with higher content of expanded perlite (2af and 3af) exhibit the better noise reduction compared to those with lower content (5af and 6af). These values are related to available information, taking into account that a conformal analysis has to consider the effective thickness of the sample [64–68].

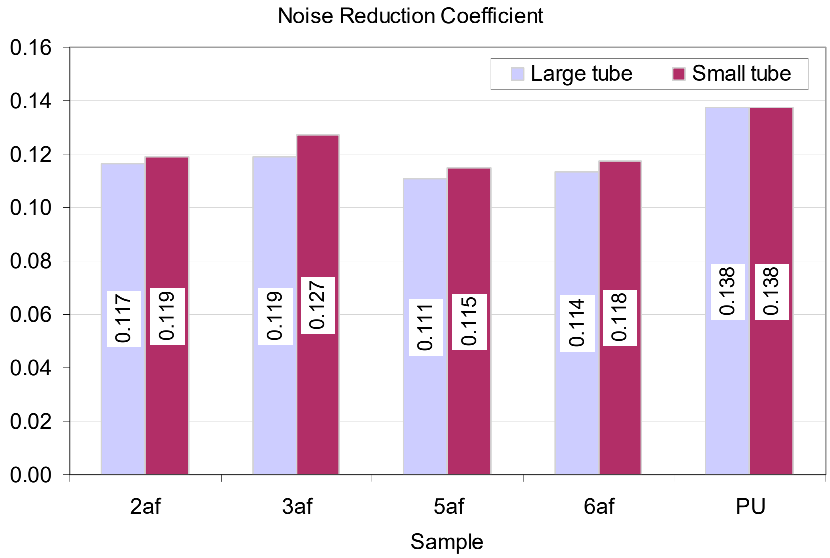
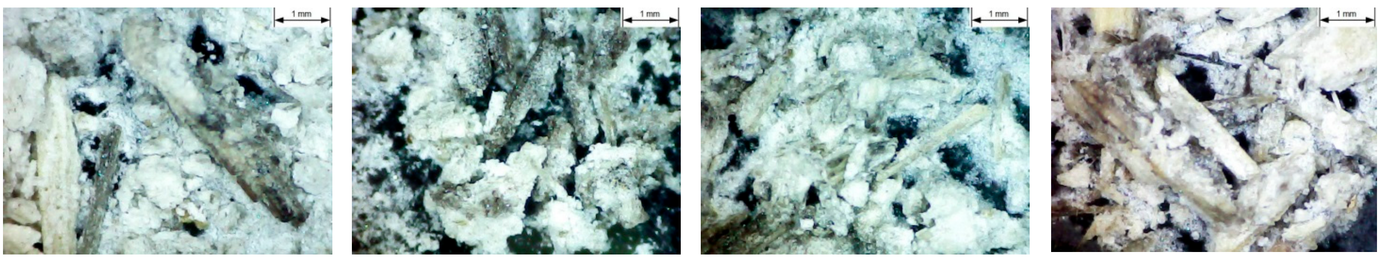


Figure 11. Comparative diagram of noise reduction coefficients for proposed composites.

Supposing the whole results, it can be concluded that the proposed composites present comparative sound insulation characteristics to the reference sample. Each of these bio-composites has a few particular aspects within their sound insulation characteristics, thus that the practical implementation has to consider the frequency spectrum of the source. However, these new materials are promising as a viable alternative to the actual large-scale utilization materials in soundproofing applications.

3.2. Structural and Water Absorption Properties

The structure of composite networking is presented in the four micrographs depicted in Figure 12. It can see that the increase of rapeseed waste content has resulted in the reduction of voids volume into the reinforced composite network. Taking into account the reinforcing properties of natural fibers developed into the entire structure of composite material, the density of reinforced composite increases with respect to the rapeseed waste content. On the other hand, it is observed and has to be underlined that a porous structure with a lower density of composite samples is obtained at high content of expanded perlite. These effects are clearly confirmed by the experimental results presented in Figure 13.



(**a**) (**b**) (**c**) (**d**)

Figure 12. Optical micrographs of rapeseed waste reinforced composites: (**a**) P2af; (**b**) P3af; (**c**) P5af; (**d**) P6af.

In addition, even if there is a higher content of lignocellulose waste in composite network structure, the starch polymer has the ability to fill gaps within the internal structure of the composite material, enabling the effect of growing up their density.

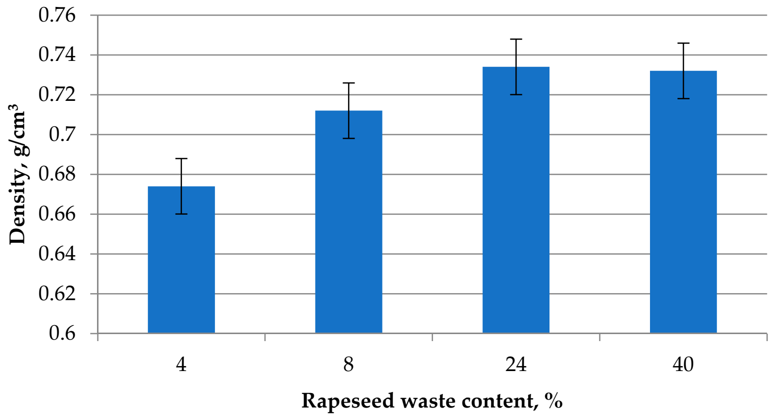


Figure 13. The influence of rapeseed waste content on the density of composite samples.

Generally, the water absorption of composite materials for insulating applications represents an important property that affects the durability of construction. Simultaneously with the decrease of the amount of water that infiltrates into the material, the strength of the composite material to the environmental external actions also increases.

Hereby, the percentage of water absorbed by the rapeseed reinforced composite samples is shown in Figure 14. It can be observed that the increase of rapeseed waste content in the sample composition leads to the high absorption level of water. The water absorption is not improved by starch addition into the composite mass. The high affinity for water of tested composite materials is justified by the fact that both rapeseed waste and starch biopolymer have high hydrophilic character, determined by the presence of a large number of free hydroxyl groups within their chemical structure.

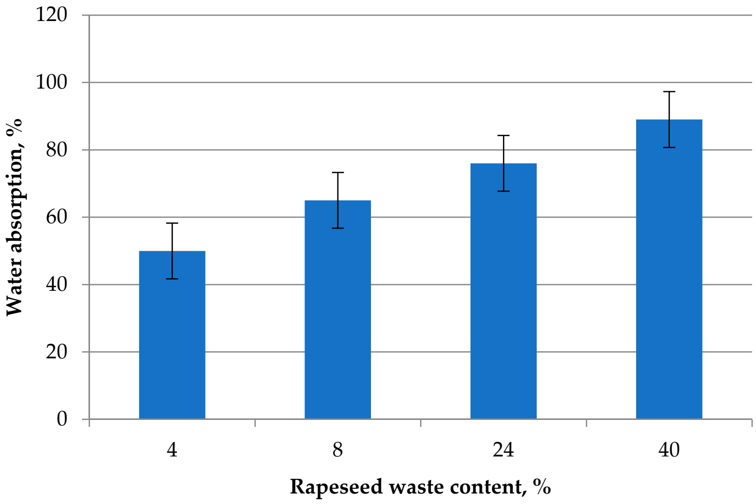


Figure 14. The influence of rapeseed waste content on the water absorption of composite samples.

This leads to the arguments that rapeseed fibers interact with water not only on the surface, but also in the bulk. Therefore, the water is absorbed in lumen, cell wall and starch biopolymer, also. Even if the starch fills the voids between rapeseed waste and perlite network, due to its hygroscopic nature and lower crystallinity, increases the water absorption. Generally, to reduce the water absorption, and improve the hydrophobic properties, the lignocellulose fibers are surface modified by alkali treatments. As result, the chemical composition of the fibers and degree of polymerization are modified by removing the lignin and hemicelluloses and molecular orientation of the cellulose crystallites. This can potentially lead to better water resistance characteristics when used as reinforcement for composite structures [69,70].

Based on these results and assumptions it can be stated that, at this moment, the performances of the rapeseed reinforced composite materials and expanded perlite are not quite similar to those of reference sample material (polyurethane composites) and would be more appropriate to use as filler for sandwich insulation structures [63].

4. Conclusions

In this study, the composite materials with expanded perlite matrix and corn starch polymer reinforced with rapeseed waste have been obtained at a laboratory scale. The effect of the content of rapeseed fibrous waste on the acoustic performance, structural and water absorption properties of these composite materials has been investigated. The acoustic performance was compared with those of commercial high-density polyurethane sound absorbers as reference.

The obtained results reveal that the sound absorption performances for all samples of rapeseed waste reinforced composites could be compared with the reference sample and provide a good absorption for low frequency and adequate level for medium frequency range till 2400 Hz.

Depending on the rapeseed content in composite samples structure, these materials exhibit few particular aspects within their sound insulation characteristics, thus that the practical implementation has to consider the frequency spectrum of the source.

The increasing of rapeseed content and including the starch in the composite structure have, as result, the increasing of both density, and water quantity absorbed by the samples. The starch polymer fills the voids within the internal structure of composite material, in order to gain stronger and more compact composition, with higher density. However, both the rapeseed waste and starch biopolymer have hydrophilic character and, as a result, a higher quantity of water is absorbed within the internal structure of composite materials. On the other hand, the higher perlite content has, as a result, a lower density of composite samples, with better sound absorption performances, as well as the water absorption properties.

Having the starting point and also the goal of this research in noise mitigation performance analysis of the proposed composites, it can conclude that these materials are promising as a viable alternative to the existing insulation materials and with possible applications in soundproofing.

To improve the water absorption, the lignocellulose fibers will be surface modified by alkali treatments for increasing their hydrophobic properties. Even if hygroscopic in its nature, the starch biopolymer must be used in the composite structure to improve their density and as a good carrier for perlite particles or other functional additives (i.e., antimicrobial, antifungal, etc.).

Another way to improve the water absorption is that additional water-resistant layers can be added to this type of composite material when a sandwich insulating structure is obtained.

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