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Experimental approximation of the sound absorption coefficient ($\propto$) for 3D printed reentrant auxetic structures of poly lactic acid reinforced with chicken keratin materials

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ABSTRACT

In this research, auxetic composites of polylactic acid (PLA) reinforced by keratin materials (barbs and rachis), were developed through 3D printing manufacture to induce diverse acoustic properties. To evaluate the coefficient of sound absorption, an electro-acoustic experiment was implemented. The capacity of sound absorption for the composites was superior to conventional (honeycomb) and auxetic PLA geometries without reinforcement. Reduction of the free space in cavities (achieved by keratin materials) triggered an increment in the resistance in air flow, improving the sound absorption. Also, increasing the interconnectivity of the cavities provides sound waves with irregular transmission routes, which reduce the wave's energy. Thus, in this work, acoustic properties are associated with 3D geometry of auxetic composites, shape, and concentration of reinforcements; considering the synergistic effect due to this combination, a range of metamaterials with acoustic capabilities were generated.

1. Introduction

Auxetic materials are relatively new, and in the materials science and engineering field are extraordinary because they have a Poisson ratio with negative values. Nowadays, peculiar auxetic materials are developed with very varied and complex structures. Typically, auxetic materials have low density [1]. Actually, auxetic structures can be manufactured by 3D printing techniques, this is a great advantage due to the design versatility and therefore material savings, considering, designs can be modified and corrected.

Although, many polymers can be used in 3D printing, one of the most used is polylactic acid (PLA) [2], because it is a very versatile polymer obtained from renewable products. It has been used in many applications including medical supplies. However, this polymer lacks good tenacity and therefore is slightly brittle. The use of reinforcements such as natural fibers diversifies PLA properties such as: higher Young's modulus, compressive strength, and flexural modulus, but with lower tensile strength, elongation at break, and impact strength than plain PLA [3,4].

Keratin has a structural arrangement that distinguishes it from other natural fibers; depending on its source, diverse mechanical properties and low density are obtained. Also, keratin has improved the mechanical properties of some polymers and even sound absorption [5–7]. The structure of natural keratin materials makes possible to diversify the research options, due to rachis has a honeycomb alike structure, and the barbs and barbules are solid structures. Other natural fibers composites or air plenty microstructures have been independently investigated in relation to their acoustic properties [8,9]. The polymer composites developed in this research show the synergistic effect of auxetic geometry manufactured by 3D printing together with the concentration and shapes of keratin reinforcements and reflected in the acoustic properties. Thus, by the first time, metamaterials including keratin, whose acoustic capabilities could be explained and controlled according to the aforementioned parameters are presented.

2. Experimental

2.1. Materials and processing of composite filaments

Keratin fibers were obtained from chicken feathers. These were supplied by poultry producers from Cadereyta, Queretaro, Mexico.

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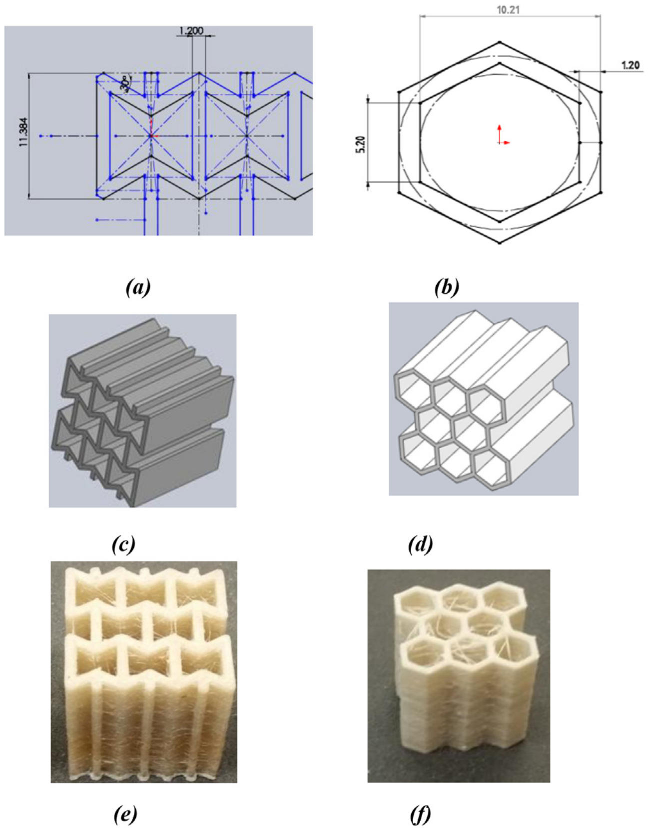

Fig. 1. Cell design for structures: a) Re-entrant auxetic, b) hexagonal; 3D models: c) auxetic, d) hexagonal; and 3D printed specimens: e) auxetic and f) hexagonal.

Flexible polylactic acid was provided by Plaxyz Lab (Mexico) (see PLA properties **Table S1**).

Two different types of keratin materials were used as reinforcements: barbs and rachis. Keratin materials were modified with sodium hydroxide (NaOH) at pH of 13. This treatment was achieved to improve reinforcement dispersion in the polymeric matrix, as it was reported previously [7]. Both reinforcements were crushed with a knife mill, implemented with a 0.5 mm screen, which corresponds to the size of the fibers. Keratin reinforcements were dispersed in the matrix in different concentrations: 0.5, 0.75 and 1 wt%, using a Fillabot® screw extruder. Pelletized PLA matrix was extruded with the keratin reinforcements obtaining filaments of 1.75 mm in diameter (see nomenclature **Table S2**).

2.2. Design and printing of the structures

The development of re-entrant hexagonal and conventional honeycomb structures was proposed, such as those shown in Fig. 1, with a cell wall thickness of 1.2 mm, and a shape relationship with the following parameters: $l = 5.2$ mm, an angle $\theta = -30^\circ$ and 30° respectively, the vertical space $h = 11.3$ mm was maintained constant along the entire direction.

These parameters were elected because the mechanical performance, experimentally obtained by our group and by simulation using finite element (MEF) [10], fit acceptably by both methods. Additive manufacturing of 3D printed structures started with re-entrant and hexagonal type models created by Solids Works® software. After, the designed structures were developed on a DREMEL® printer (using filaments previously extruded).

2.3. Sound absorption coefficient measurement

The wave absorption is the phenomenon in which waves intensity decreases for dissipative effects produced by propagation medium, causing the reduction of the transported energy [11].

The intensity decrease (**Figure S1**) arises in a differential of the material *dx* in the polylactic acid, and can be expressed as:

$$\boldsymbol{dI = -\alpha I dx} \tag{1}$$

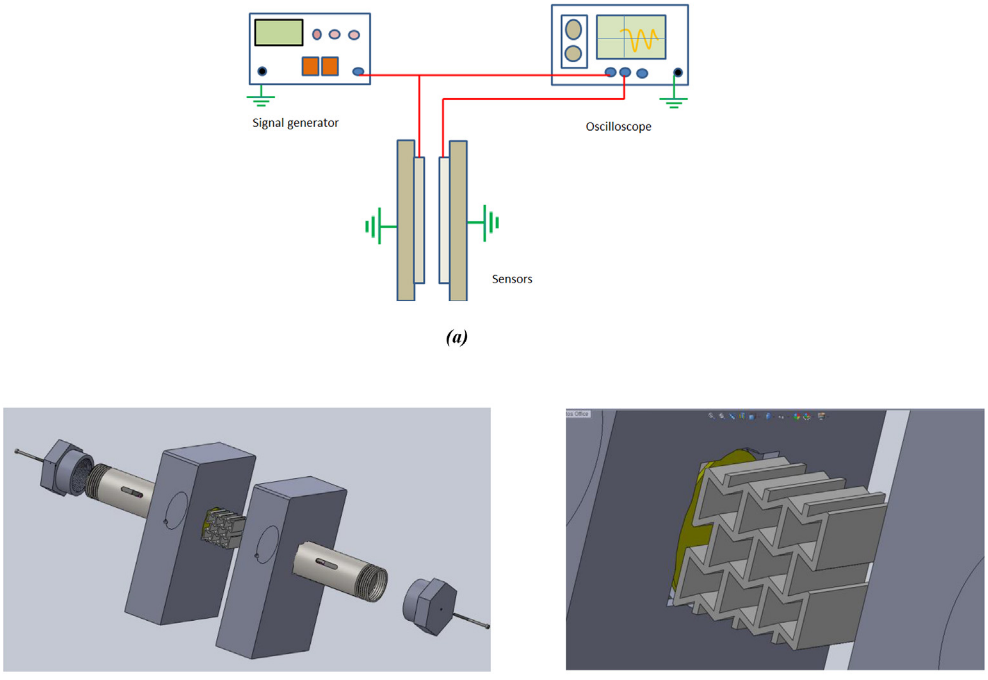

Fig. 2. Instrumentation of the acoustic experiment. a) Acoustic system, b) Detailed sensor device, c) Tested sample stuck between sensors.

where $\boldsymbol{\alpha}$ is called absorption coefficient.

Separating and integrating (1). With limits, initial intensity $\boldsymbol{I_0}$ and output intensity $\boldsymbol{I_s}$, and for thickness $\boldsymbol{x = 0}$, and final thickness **x**.

$$\int_{I_0}^{I_s}\frac{dI}{I}=\int_0^x-\alpha dx\rightarrow[\ln I]_{I_0}^{I_s}=[-\alpha x]_0^x\rightarrow\ln I_s-\ln I_0=-\alpha x$$

$$\ln\frac{I_s}{I_0}=-\alpha x\rightarrow e^{\ln\frac{I_s}{I_0}}=e^{-\alpha x}\rightarrow I=I_0e^{-\alpha x}$$

Thus, Eq. (2) establishes that the wave intensity falls exponentially ($\boldsymbol{e^{-\alpha x}}$) with the thickness of the composite:

$$\boldsymbol{I=I_0e^{-\alpha x}} \quad (2)$$

2.4. Acoustic characterization

For the evaluation of sound absorption coefficient, an electroacoustic experiment was implemented (Fig. 2-**a**). This system incorporates a signal generator that produces different wave trains, a digital oscilloscope Tektronix TBS1164® and an own designed device as sensor. This last is shown in Fig. 2-**b,c.**

In the assembled system, a sine wave voltage was induced through the signal generator, at 5 kHz frequency. Input and output signals were recorded and characterized in the oscilloscope. The wave signals obtained from four voltages were evaluated in each material, the maximum of the sine wave (Vmax) was selected because it presents less variability in the values obtained experimentally (see details of used frequencies, **Section S3**).

3. Results and discussion

Fig. 3**a** shows the results obtained for y, (natural logarithm of the quotient of the output (V_o) and input (V_i) voltages), as a function of x (sample thickness). The fit function for the data of each material was obtained using the method of least squares. **Table S3** shows correlation coefficients close to −1, indicating a good linear and inverse relationship between the variables. As it can be appreciated, all materials show a clear diminish in the y value as the thickness of the samples increases. The slope of these graphics is associated with the absorption coefficients (α), which are depicted in Fig. 3**b**.

Regarding the capacity of sound absorption, the results show that the behavior exhibited by auxetic specimens, is superior to that shown by hexagonal structures at the evaluated frequency (5 kHz, audible range). This agrees to other published work [12], in which auxetic foams, with a negative Poisson ratio, showed their better sound absorption coefficients when frequencies in audible range (lower frequencies below 1500 Hz) were used, (see details **Section S3**). At the same time, results demonstrate that keratin reinforcements (particularly rachis cell structure) contribute to acoustic properties, because it was found that all composites are superior to conventional honeycomb and auxetic plain PLA samples (Fig. 3b-1).

On the other hand, the barb fiber does not have the same structure than rachis possess, instead of cell structure a solid fiber is found; however, it also increases the absorption coefficient of the matrix Fig. 3b-2. (See more details **Section S4**). Reduction of free space in cavities is caused by presence of keratin materials through 3D printed cells. This increases the resistance to air flow and improves the sound absorption. Additionally, modified keratin materials are better dispersed, therefore composites (auxetic or hexagonal) with modified keratin (rachis or fiber) show higher α values than composites with unmodified keratin at all concentrations. Besides, the increase in the interconnectivity of the cavities, also achieved by both types of reinforcement, but mainly because of rachis, plays an important role in sound absorption. Table 1 shows absorption coefficients for all composites, as it can be appreciated the highest values are for those composites with rachis and the lowest are for plain PLA.

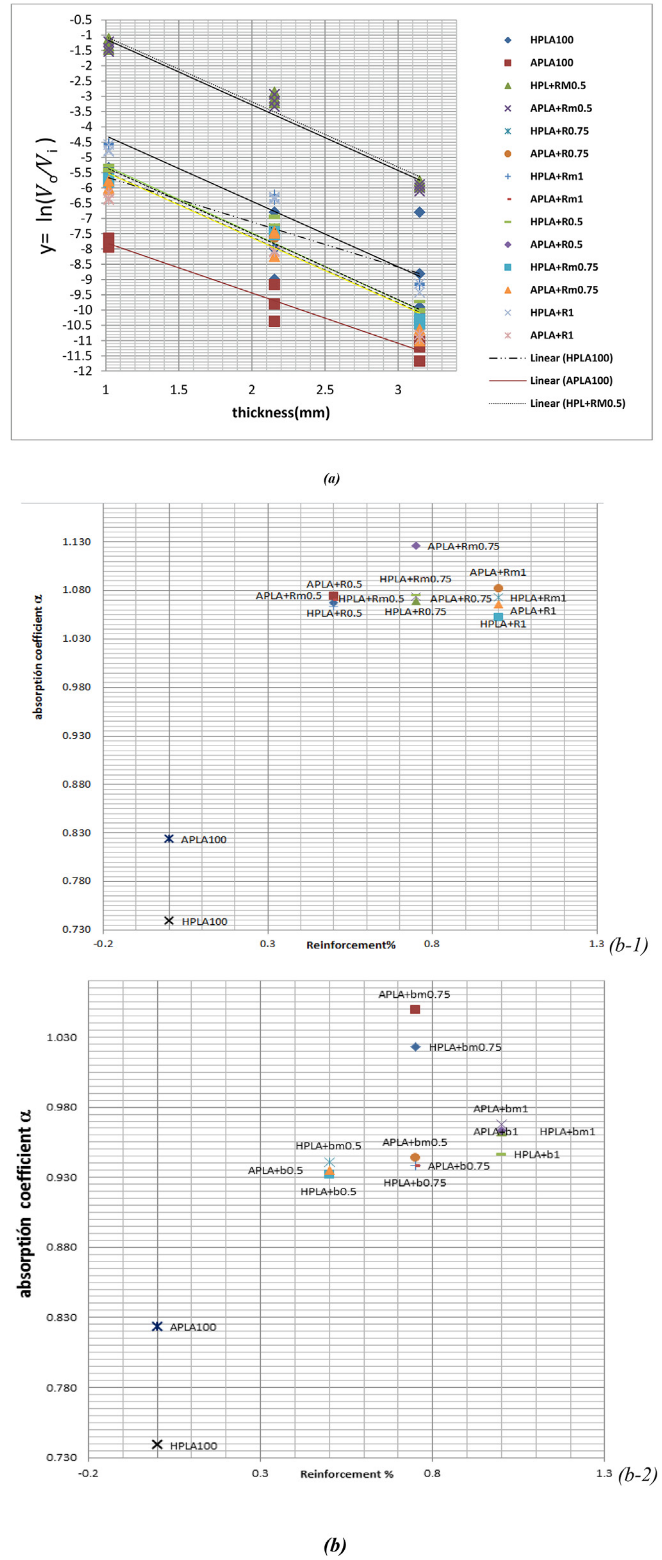

Fig. 3. Absorption coefficient: (a) Natural Logarithm of the output and input voltage ratio for auxetic (A) and hexagonal (H) PLA-Keratin composites (b) Slope values as function of reinforcement percent for systems: a) Polylactic acid-Barb, b) Polylactic acid-Rachis.

Table 1
Absorption coefficients (α) of composites at Vmax in potential difference.

Reinforcement (%)	sample	α (1/mm)
0.75	**APLA + Rm0.75**	**1.126**
1	**APLA + Rm1**	**1.082**
0.75	HPLA + Rm0.75	1.075
0.5	**APLA + Rm0.5**	**1.075**
0.75	**APLA + R0.75**	**1.073**
1	HPLA + Rm1	1.073
0.5	**APLA + R0.5**	**1.070**
0.75	HPLA + R0.75	1.069
0.5	HPLA + Rm0.5	1.067
1	**APLA + R1**	**1.065**
0.5	HPLA + R0.5	1.063
1	HPLA + R1	1.053
0.75	**APLA + bm0.75**	**1.049**
0.75	HPLA + bm0.75	1.023
1	**APLA + bm1**	**0.968**
1	**APLA + b1**	**0.963**
1	HPLA + bm1	0.963
1	HPLA + b1	0.947
0.5	**APLA + bm0.5**	**0.944**
0.5	HPLA + bm0.5	0.941
0.75	**APLA + b0.75**	**0.938**
0.75	HPLA + b0.75	0.936
0.5	**APLA + b0.5**	**0.935**
0.5	HPLA + b0.5	0.932
0	**APLA100**	**0.823**
0	HPLA100	0.739

4. Conclusions

The auxetic structures reinforced with keratin represent a novelty. The results respond not only to the auxetic structure, but also to the interactions between keratin materials and matrix. An adequate dispersion of keratin produces synergic effect between the auxetic geometry and the reinforcement, improving notably the sound absorption. Rachis structure plays an important role in acoustic coefficient values. These kinds of materials could find important applications in automotive and aeronautic industries, due to the versatility of the involved parameters.

CRediT authorship contribution statement

Vicente Amaya-Amaya: Methodology, Validation, Formal analysis, Investigation, Writing - original draft, Visualization. **Miguel de Icaza-Herrera:** Methodology, Validation, Formal analysis, Investigation, Visualization, Conceptualization. **Ana Laura Martínez-Hernández:** Conceptualization, Resources, Writing - review & editing, Supervision, Project administration, Funding acquisition. **Gonzalo Martínez-Barrera:** Conceptualization, Writing - review & editing, Supervision, Project administration, Funding acquisition. **Carlos Velasco-Santos:** Conceptualization, Resources, Writing - review & editing, Supervision, Project administration, Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.matlet.2020.128757.

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