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**“Wet-laid technology implementation in revalorization of costal algae
wastes in textile nonwoven industry”.**

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Abstract:

The wet-laid process with three different lengths of *Posidonia oceanica* algae wastes (base) and PLA (binder) fibres has been used to obtain nonwovens to be used as a reinforcement of composite materials processed by hot-press and injection molding. *Posidonia oceanica* collected directly from drifts contains large amounts of sand, salt and stones of a variety of sizes which make processing difficult. In addition, in a raw state it is too large to be processed on

an experimental wet-laid plant. Thus, the tasks of cleaning, drying and crushing algae wastes have been carried out in order to develop several non-woven to select the most suitable Posidonia waste length. Main physical property (surface mass) has been determined by standardized test. Tensile strength have been determined on longitudinal (preferential deposition direction in hydroformer station) and transversal directions that has revealed that slight anisotropy is obtained with the wet-laid nonwovens and a better tensile strength is obtained in the preferential deposition direction. In general the nonwovens made by medium length of the algae wastes get the higher value of tensile strength. As well acoustical analysis has been carried out and this has revealed that using a larger number of layers, better acoustic absorption is obtained. It is possible to obtain eco-friendly composites as the total content of natural raw material is higher than 70 wt%, and the binder used is a bio-based PLA thermoplastic material.

Key words: nonwoven, wet-laid, Posidonia oceanica, algae wastes, PLA, hot-press moulding, injection moulding

1. Introduction

Algae and seaweed accumulations on beaches and along our coasts are an environmental nuisance; this biomass emits unpleasant odours, promotes mosquitos and their rotting contribute to increase the high mortality in shellfish beds as they turn into rubbish. City Councils of the coastal areas are required to remove them if they want to remain their touristic conditions and their Blue Flag Beach category (FEE-Foundation for Environmental Education). It is important to remark that sand is also removed when algae residues are collected so, year by year the beach goes back and must be regenerated applying new sand. For this reason, currently the most widely adopted practice is to leave the algae residues in the coasts in winter and collect them in summer when tourists make massive use of the coastal areas. Generally, this actuation takes place within the Integrated Management System where all the marine accumulations are managed as urban solid wastes and are deposited in a landfill and/or incinerated.

In the last years, a growing interest on the use of biobased materials or materials from renewable resources has been detected. This interest has also arrived to technical sectors such as building, automotive, transportation, etc.

industries due to increasing environmental concerns, potential biodegradability and use of overall eco-friendly materials. Some of the requirements of these technical sectors are focused on comfort and this means that base materials must provide good insulation behaviour, both thermal and acoustic (Chen et al., 2011, Parikh et al., 2006). Mineral and synthetic polymer wools are interesting substrates which are able to offer easy handling and good insulation properties due to the internal structure, therefore their use in building industry and other technical sectors has been generalized (Debnath et al., 2010, Lin et al., 2009, Lou et al., 2008, Tai et al., 2010); nevertheless, new materials from renewable resources are being demanded since they could offer similar behaviour than traditional heat and sound absorbers, so that, important efforts on the development of natural (plant or animal derived) materials are being done.

The wet laid is a processing technique highly used in the paper and textile industry for nonwoven formation. In the case of nonwoven textiles, it is possible to obtain nonwoven structures based on different base components both raw and waste materials. So that it is possible to mix a base natural fibre with a binding fibre to provide cohesion after a thermo-bonding process (Fedorova et al., 2007, Kim et al., 1999, Rawal et al., 2010). The wet-laid process, which uses highly diluted fibre-water dispersions, is an eco-friendly process since,

although it consumes high water amounts, all the water is recirculated since it only acts as the fibre carrier component so that, almost all water is recovered in the hydroformer station in which nonwoven formation occurs (Safavi et al., 2009).

The main aim of this work is the revalorization of costal algae wastes in textile nonwoven industry using the wet laid technology. To provide high cohesion on nonwovens, different binder fibres have been used: Lyocell fibre and PLA fibre. The main physical properties such surface mass are determined by standardized test. In addition, mechanical preform on longitudinal (preferential formation direction in the hydroformer station) and transversal directions is evaluated by determining tensile strength and elongation at break in terms of the binder fibre content. The potential of these nonwovens as sound absorber materials is determined with standardized tests using an impedance tube to determine the absorption coefficient.

Within the last five years AITEX has been involved in several R&D projects regarding the revalorization of solid wastes by means of using wet-laid technologies. For instance, AITEX has been the technical coordinator of WET-COMP LIFE+ Project (<http://wetcomp.aitex.net/>), being this initiative related to

the use of textile wastes as raw materials for the manufacturing of composites. It has been possible to develop composites with good mechanical properties based on textile wastes in rate close to 60-70% wt. The wet-laid non-woven obtained using textile wastes as raw materials were employed as reinforcement of composites in LFT-D and compression moulding processes. On another hand, AITEX is coordinating nowadays the SEA-MATTER LIFE+ Project (<http://www.seamatter.com/>). The main objective of SEA-MATTER is to demonstrate and validate the reuse of coastal algae and seaweed accumulations as raw materials in composites industry, specifically as acoustic panels in buildings.

2. Materials and methods

2.1. *Materials*

Posidonia oceanica wastes from the Mediterranean coast was used as base fibres for nonwoven formation. Tasks of collecting, cleaning and crushing have been carried out to obtain different lengths (1 mm, 1,4 mm and 4,40 mm).

Table 1 here

Lyocell fibres were commercial reference LYOCELL gl 1,7/4 supplied by STW Fibres (SchwarzwälderTextil-werke, Schenkenzell, Germany) have been used to allow the web formation. The binder fibre to give cohesion to the nonwoven was a PLA (polilactic acid) commercial grade PLSTD-013NRR-0650 1.3 dpf x 1/4" Staple Fibres supplied by Minifibres, Inc. The main properties of both fibres are summarized in Table 2.

Table 2 here

2.2. Methods

Formation of Posidonia oceanica wastes nonwovens by the Wet-Laid Process

In a first stage, Posidonia oceanica algae wastes, Lyocell and PLA fibres were weighed and immediately they were poured into a pulper with a maximum capacity of 35 L. This pulper was supplied by PILL Nassvliestechnik (PILL Nassvliestechnik GmbH, Reutlingen, Germany). The fibre concentration in the water dispersion was 10 g/L. To obtain optimum fibre separation, vigorous agitation at 2,300 rpm was maintained for 10 min. After this initial stage, the water dispersion is transferred to a larger polyethylene tank (1,200 L maximum

capacity) in which, the fibre dispersion is diluted up to 1 g/L with less aggressive agitation at 170 rpm for a total time of 15 min. Then once the fibres are appropriately dispersed in aqueous solution the water-fibre mixture is moved to the hydroformer station by using hydraulic pumps. The hydroformer station has been supplied by PILL NASSVLIESTECHNIK GmbH and it is constructed in stainless steel with different polycarbonate windows to observe the process evolution. The maximum width is 510 mm, the take-off angle is 20 ° and the conveyor speed can vary between 1 and 10 m min⁻¹. Two different tanks are used to ensure optimum dispersion conditions; when one of these two tanks is used, the other is empty and vice versa. The water-fibre dispersion is pumped to the hydroformer station, but before reaching to the forming strip, the water-fibre dispersion is diluted once again up to a final concentration of 0.33 g/L. Then the water-fibre dispersion is dropped onto the porous forming strip which acts as a filter media in which water is removed by vacuum and fibres are deposited.

Table 3 here

Once the nonwoven is formed, additional thermo-bonding process must be carried out in order to melt PLA fibres to embed *Posidonia oceanica* wastes

thus increasing nonwoven cohesion. A first drying stage was carried out initially in a drying oven SDT-600 by Tacome (Tacome S.A., Ontinyent, Spain) at a fixed temperature of 195°C for 15 min. After this stage, the dried nonwoven is subjected to a calendaring process in a CL-600 calender supplied by Tacome (Tacome S.A., Ontinyent, Spain). The surface temperature of the roller was maintained to 200°C and the linear pressure over the nonwoven was fixed to 0.124 MPa m (124 N/mm). Finally, the continuous nonwoven was rolled in a roller machine EN-600 supplied by Tacome.

Figure 1 here

Composite Formation by Hot-Press and Injection Moulding of Posidonia oceanica wastes Nonwovens

The formation of engineering composites using hotpress moulding have been carried out with a Robima S.A. (Valencia, Spain) 10 Tn press with temperature control Dupra S.A. (Dupra, Castalla, Spain). The composites were manufactured using aluminum plates with a hollow space of 20x20 cm². Eight sheets sizing 20x20 cm² were cut and were placed between the plates in order to consolidate the composite. Then a constant force of 4 Tn was applied at

160°C for 60 min and then proceeded to cool the mould for 20 min at room temperature to appropriately unmould.

Figure 2 here

The formation of engineering composites using injection moulding have been carried out with a vacuum bag and polyurethane foams were combined with the *Posidonia oceanica* wastes non-woven material, and an EPOXY resin was used.

Figure 3 here

Determination of surface Mass

Surface Mass of the nonwoven materials has been measured on 1dm² shapes obtained by cutting nonwovens with a circular shape former. Five different samples were cut and weight in a precision balance GR-200-EC and average values of surface mass were calculated. Temperature and relative humidity have been maintained at 20±2°C and 65±4% respectively.

Mechanical Characterization

Tensile properties (both longitudinal and transversal) of nonwovens have been determined by following the guidelines of the ISO 29073-3:1993 with an Instron dynamometer model 4501 (Instron, Barcelona, Spain). The clamp distance was set to 200 mm and the crosshead rate was 100 mm/min⁻¹. At least five samples were tested and average values for tensile strength and elongation at break were calculated. Temperature and relative humidity have been maintained at 20±2°C and 65±4% respectively.

Acoustic insulation characterization

Acoustic insulation properties have been evaluated on nonwovens with *Posidonia oceanica* wastes, by measuring the absorption coefficient of 1 layer and 10-layers s by following the guidelines of the ISO 10534-2:2002 standard with an impedance tube built to the referenced standard. A double channel Symphonie analyzer with FFT has been used with 1/2 inch and 1/4 inch microphones. The experimental frequency has varied in the 100 - 6400 Hz.

3. Results and discussion

3.1. *Characterization of Posidonia oceanica wastes nonwovens*

The surface mass values (Table 4) of the *Posidonia oceanica* wastes nonwovens shows variability between the samples even all of them have been developed with the same wet laid process parameters because the raw material used are wastes and due to the random deposition of fibres during nonwoven formation. The most variability showed is the samples formed with the higher length of algae wastes.

Table 4 here

With regard to the mechanical properties of individual nonwovens as a function of the *Posidonia oceanica* waste length, Table 5 shows a summary of the main mechanical parameters: tensile strength and elongation at break for longitudinal and transversal directions. It can be said that slight anisotropy is obtained with the wet-laid nonwovens and a better tensile strength is obtained in the preferential deposition direction. This fact could be important for further processing of these nonwoven structures by hot-pressing moulding of stacked nonwoven substrates

in order to obtain high isotropic composite materials. The samples with the poorest performance in this respect are those of coarse-grain G3 type (sample POSWS-G3). The best-performing sample is the medium-grain G2 type (POSWS-G2). Regarding the elongation at break, values are different between the samples but there are no differences between longitudinal and transversal. Therefore we can conclude that the best-performing sample is the medium-grain G2, type POSWS-G2.

Table 5 here

The increasing use of nonwovens in technical applications requires, in most cases, good acoustic insulation properties. For this reason, acoustic insulation behaviour of *Posidonia oceanica* wastes nonwovens has been evaluated in a Kindt's tube (Figure 4).

Figure 4 here

The acoustic absorption coefficient measurement was conducted using a single-layer nonwoven sample (Graph 1, Graph 2 and Graph 3) and 10-layers sample (Graph 4, Graph 5 and Graph 6) and the following graphs demonstrate

that when the analysis is performed on the 10-layer sample the acoustic absorption values increase and Graph 5 show the higher acoustic absorption of 10 layers of POSWS-G2 samples. Therefore these nonwoven structures show attracting properties for technical applications as acoustic absorbers.

Graph 1 here

Graph 2 here

Graph 3 here

Graph 4 here

Graph 5 here

Graph 6 here

4. Conclusions

The use of the wet-laid technique to obtain *Posidonia oceanica* wastes-based nonwovens with different algae wastes length has been validated. It can be said that the thermobonding process is useful to give enough cohesion to nonwovens thus enabling them for handling without breakage and subsequent processing operations by hot-press and injection molding which requires adaptability to different shapes.

Mechanical characterization of nonwovens shows that the nonwoven made using medium length of algae wastes has the better behavior. On the other hand, the wet-laid process on a hydroformer is characterized by a preferential deposition direction so that, slight anisotropy on nonwoven sheets can be detected as mechanical performance on transverse direction are lower than those obtained on longitudinal (preferential) direction.

The acoustical insulation properties (measured through the absorption coefficient) are better using 10 layers of nonwoven and showing higher absorption the POSWS-G2 nonwovens in the low and high frequencies range.

In general terms we can conclude that the wet-laid technique is useful to obtain nonwovens from *Posidonia oceanica* wastes. Although some anisotropy is detected, due to intrinsic preferential deposition direction, mechanical performance is enough to ensure good handling. In addition to this, interesting acoustic insulation properties can be obtained by stacking different sheets thus allowing the use of these materials as technical substrates for sound absorption insulation applications.

5. Acknowledgements

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fiber dispersion for wet-laid nonwoven. *Fiber. Polym.* 10, 231-236.

7. Figure legends

Figure 1.- Images of AITEX wet-laid pilot plant.

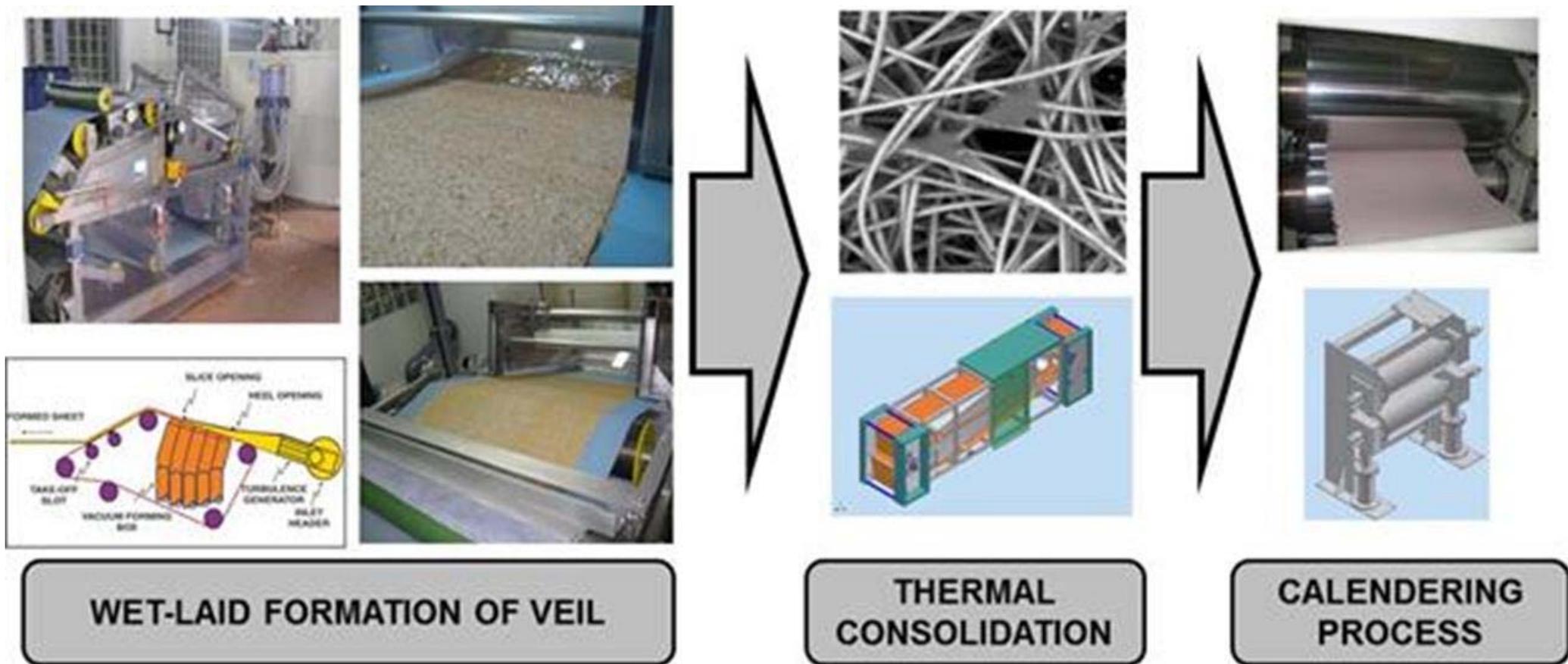


Figure 2.- Composites developed using hotpress moulding process

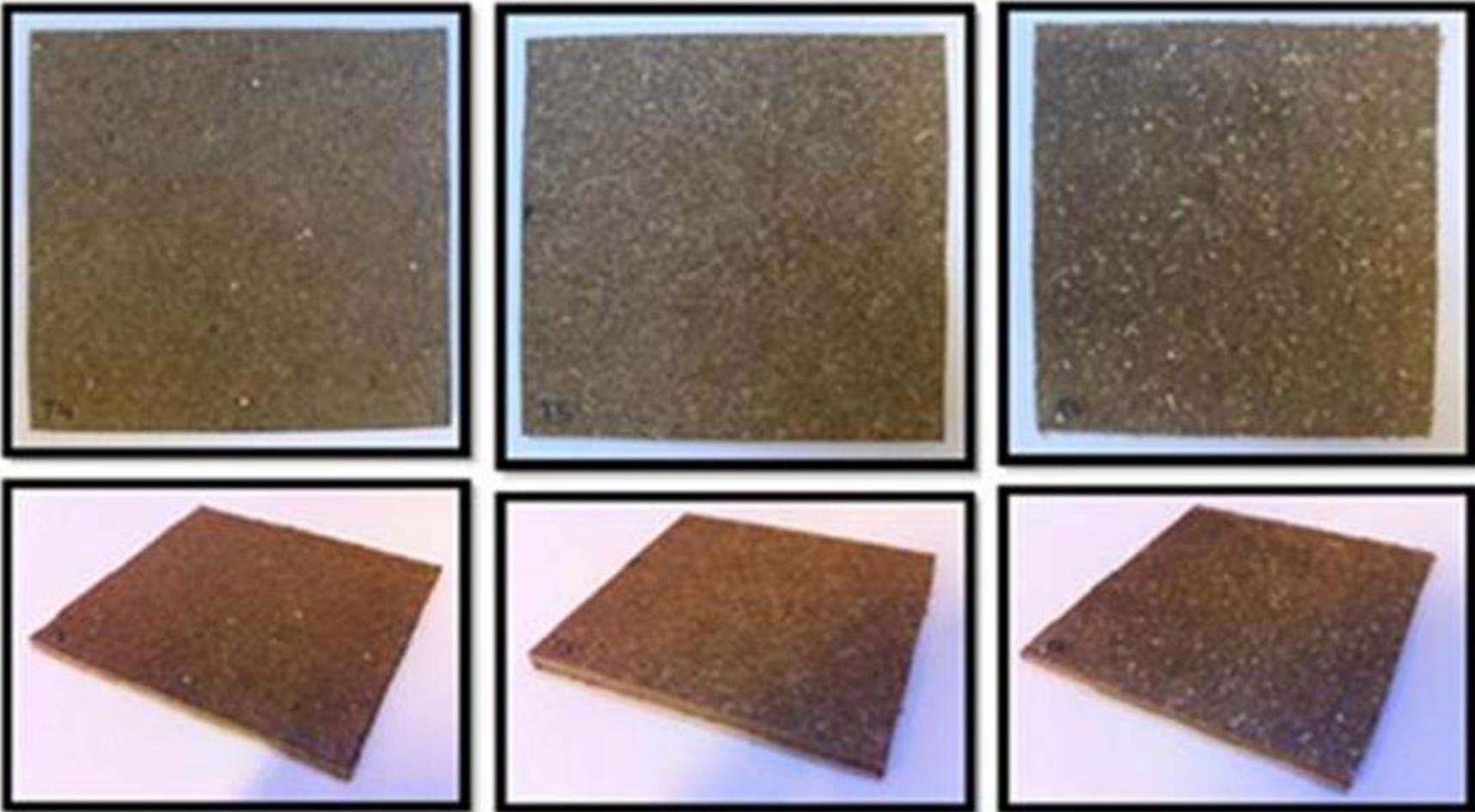


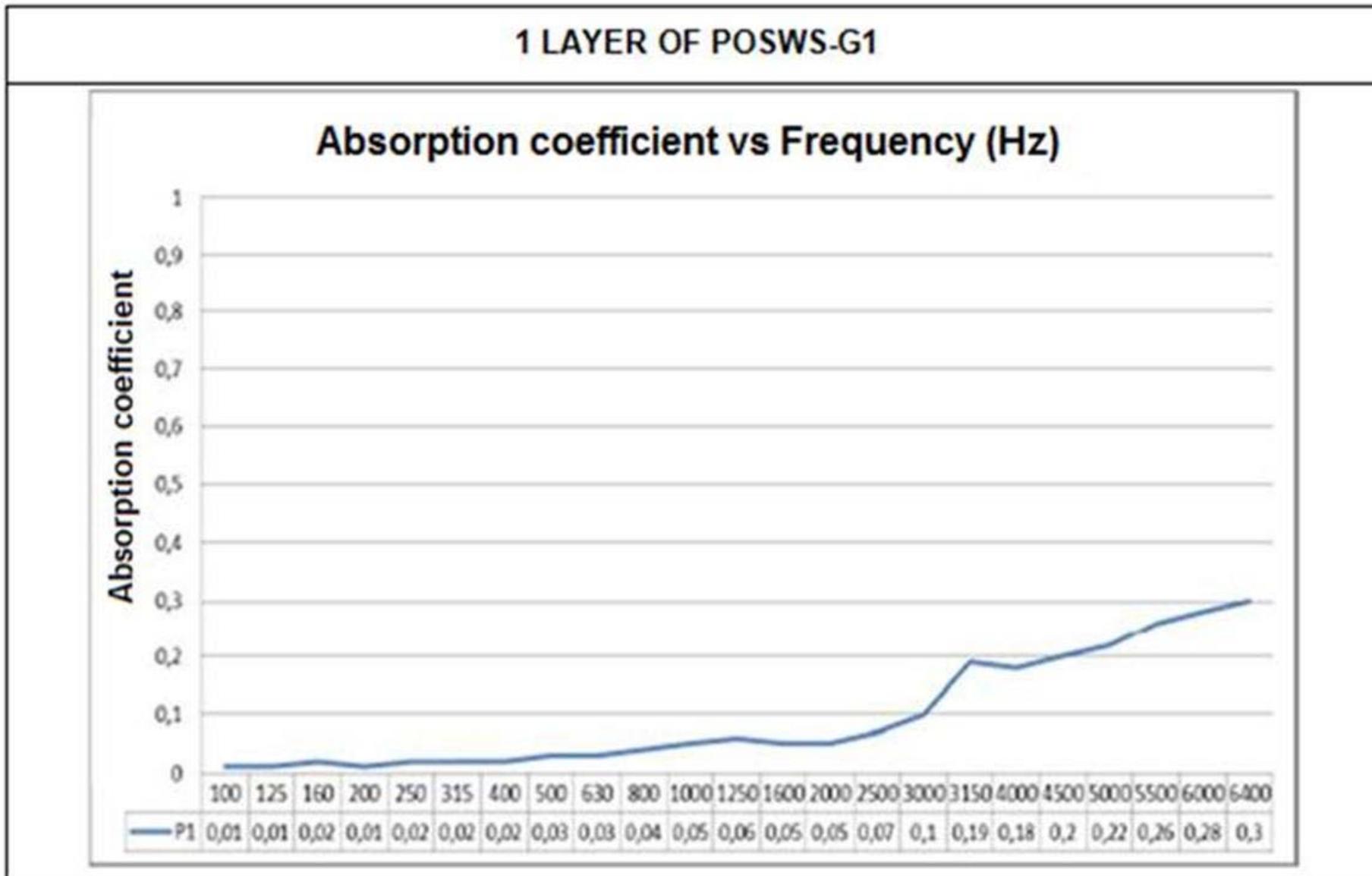
Figure 3.- Composites developed using injection moulding process



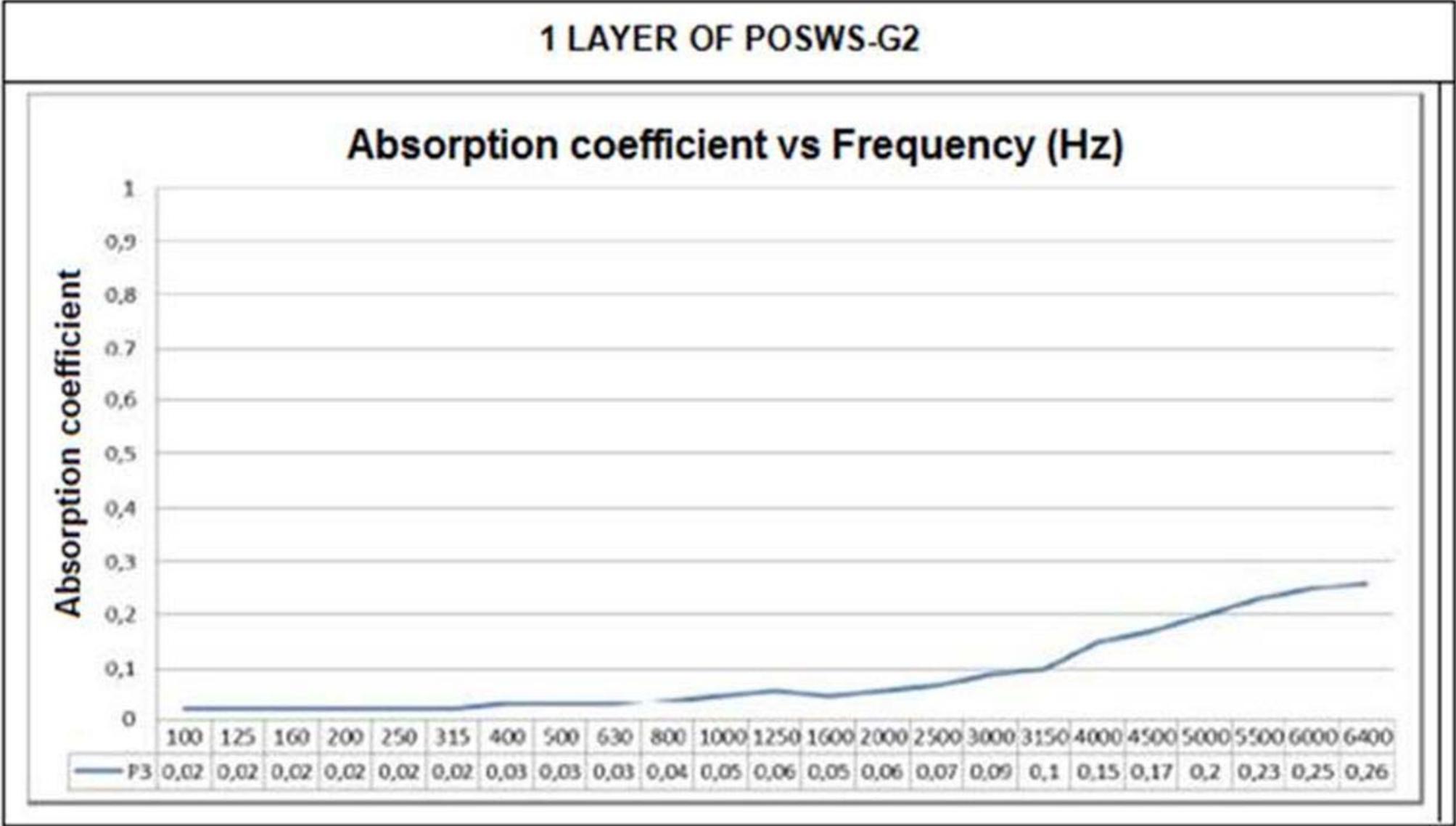
Figure 4.- Image of AITEX Kundt Tube



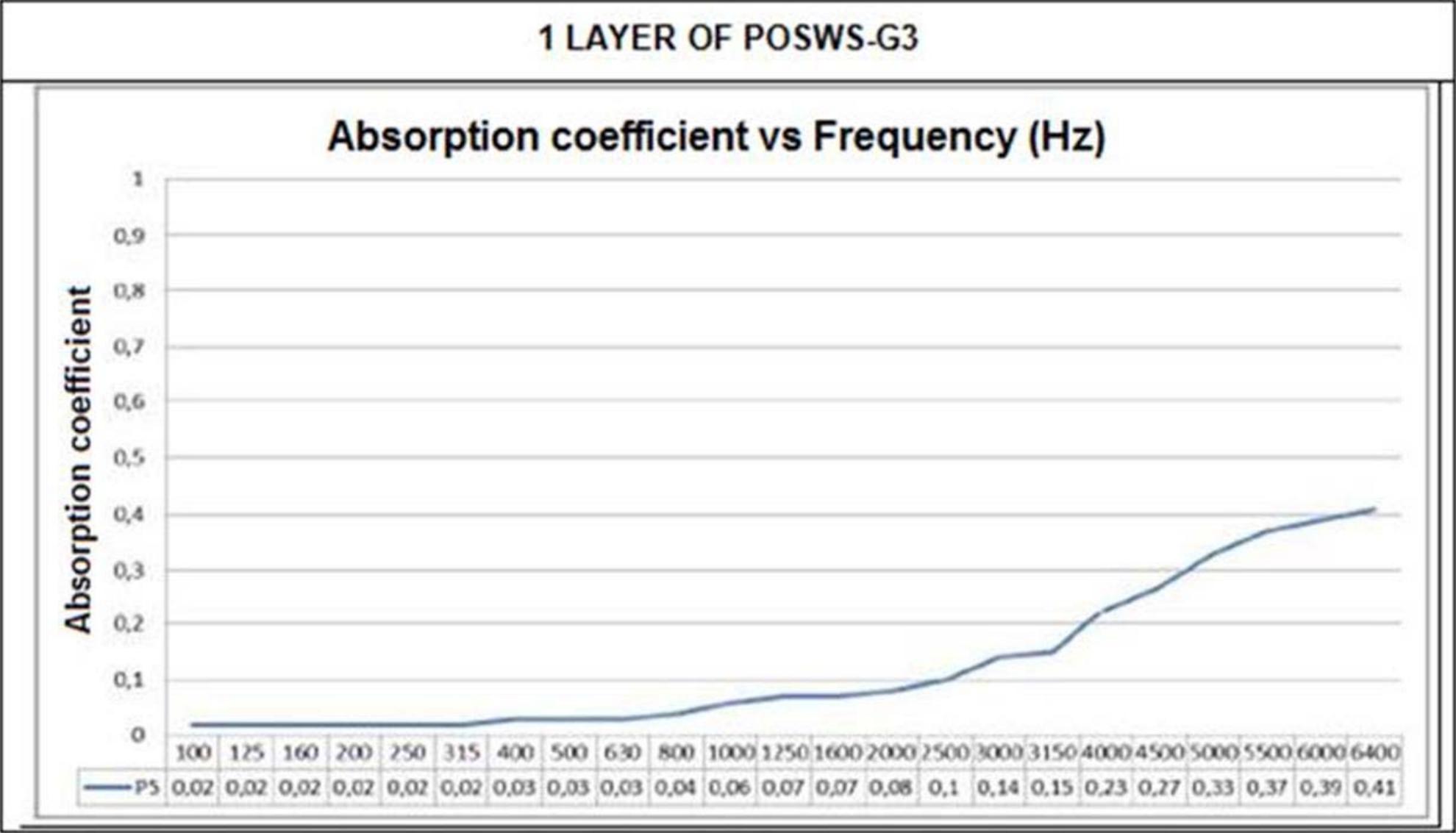
Graph 1.- Representation of the acoustic absorption coefficient at different frequencies for 1 layer of POSWS-G1



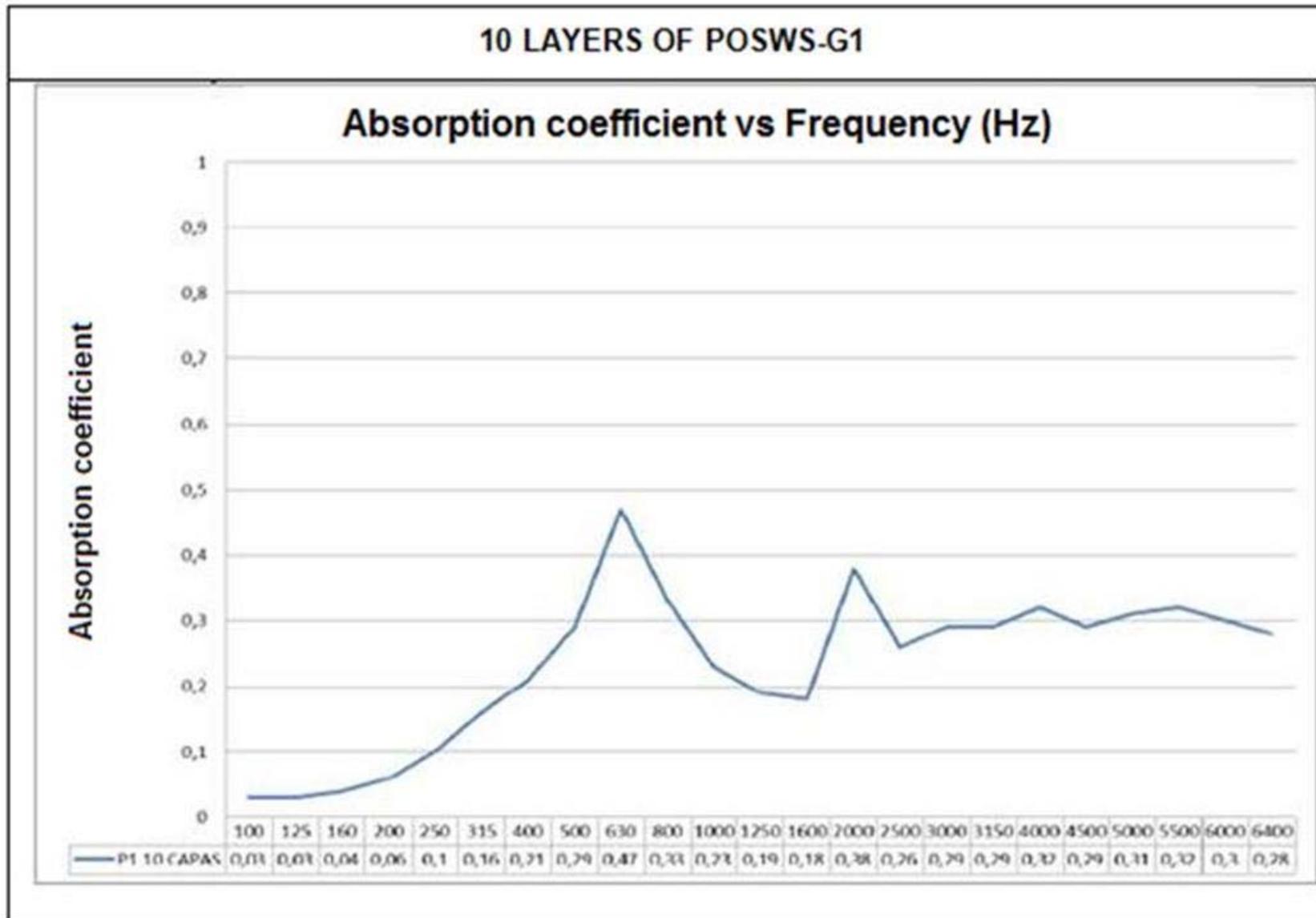
Graph 2.- Representation of the acoustic absorption coefficient at different frequencies for 1 layer of POSWS-G2



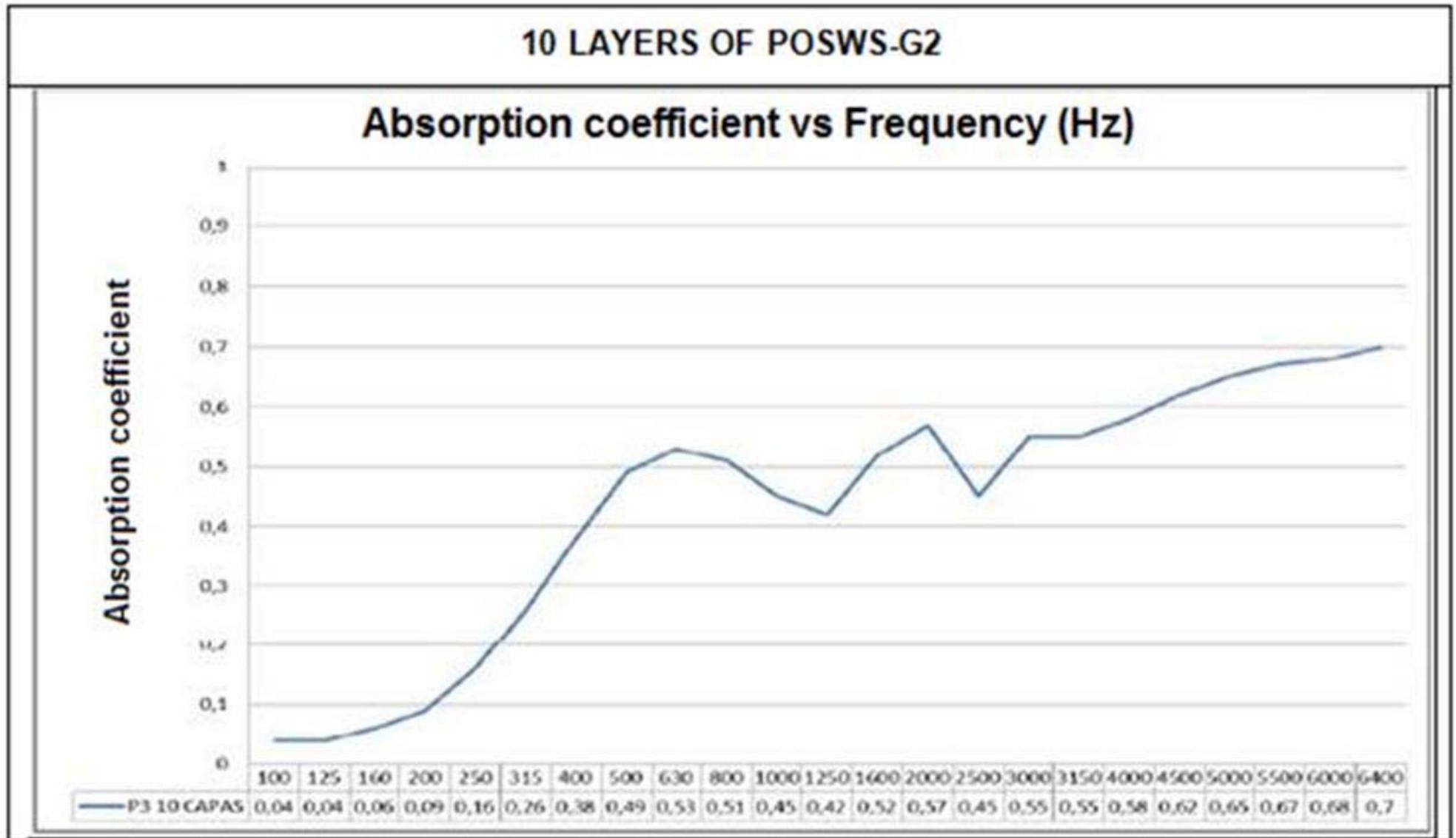
Graph 3.- Representation of the acoustic absorption coefficient at different frequencies for 1 layer of POSWS-G3



Graph 4.- Representation of the acoustic absorption coefficient at different frequencies for 10 layers of POSWS-G1



Graph 5.- Representation of the acoustic absorption coefficient at different frequencies for 10 layers of POSWS-G2



Graph 6.- Representation of the acoustic absorption coefficient at different frequencies for 10 layers of POSWS-G3

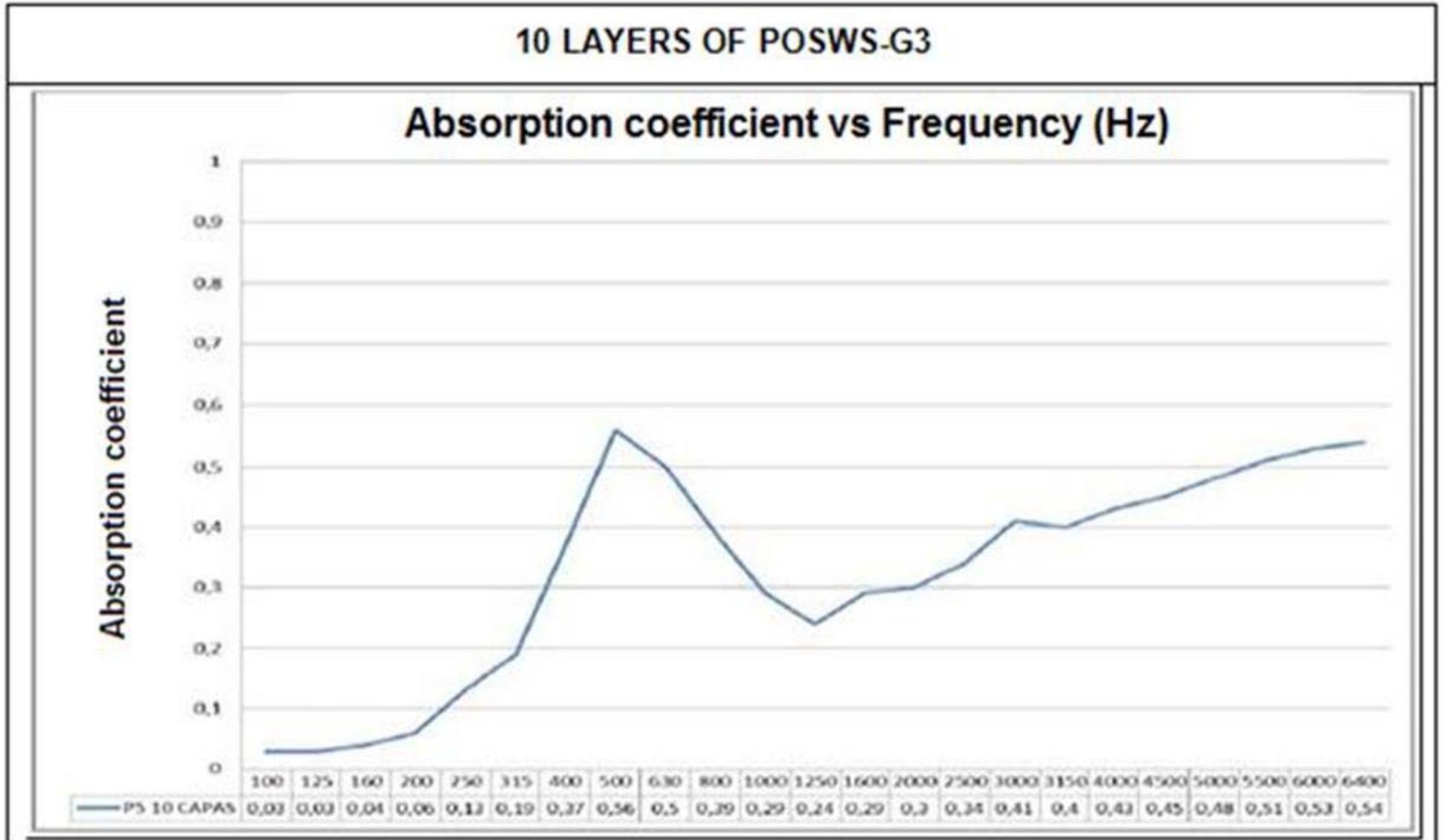


Table 1.- Different average length of *Posidonia oceanica* wastes

Reference	Description	Average length (mm)
G1	Fine grain <i>Posiconia</i> wastes	1 mm
G2	Medium-grain <i>Posidonia</i>	1,4 mm
G3	Coarse-grain <i>Posidonia</i>	4,40 mm

Table 2.- General characteristics of Lyocell and PLA fibres used to give cohesion to the nonwoven (PLA) and to allow the nonwoven formation (Lyocell) by the wet-laid process.

Property	Lyocell Fibre	PLA Fibre
Cut length (mm)	4	6
Fibre coarseness	1,7 dtex	1,3 dpf
Melting point (°C)	---	168/170
Degradation point (°C)	190	---

Table 3.- Samples of *Posidonia oceanica* wastes nonwoven

Reference	Material	Composition % (p/p)
POSWS-G1	Posidonia oceanica waste fibre G1	70
	Lyocell fibre	20
	PLA fibre	10
POSWS-G2	Posidonia oceanica waste fibre G2	70
	Lyocell fibre	20
	PLA fibre	10
POSWS-G3	Posidonia oceanica waste fibre G3	70
	Lyocell fibre	20
	PLA fibre	10

Table 4.- Mass per unit area results

Reference	Mass per unit area (g/m²)
POSWS-G1	235.48
POSWS-G2	258.27
POSWS-G3	165.75

Table 5.- Tensile strength and Elongation at break

Reference	Direction	Maximum force (N)	Elongation at break (%)
POSWS-G1	Longitudinal	63	2.8
	Transversal	30	2.8
POSWS-G2	Longitudinal	79	4.0
	Transversal	51	5.4
POSWS-G3	Longitudinal	30	3.0
	Transversal	27	3.2