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Sustainable materials with high insulation capacity obtained from wastes from hemp industry processed by wet-laid

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Abstract

This article reports on the revalorization of hemp waste from the textile industry, focusing on the development of new sustainable materials with high insulating properties. Wet-laid technology was used to manufacture nonwovens with different binding fibers, polylactic acid, and viscose fibers. The characterization of the acoustic insulating capacity was carried out using a Kundt tube, and the thermal insulating performance by measuring the heat transmission resistance (R) and thermal conductivity (λ). The results showed that the developed nonwovens have lower thermal conductivity values of about 0.027–0.034 W/(m K), were even lower than those of traditional thermal insulating materials, being the sample with 100 g/m^2 of areal density and with a composition of 80% of hemp, 10% of polylactide and 10% of viscose the one with the lowest thermal conductivity (0.027 W/(mK). Their acoustic absorption capacity was around 0.76 at a frequency of 6 kHz, in samples containing high hemp waste (>80 wt%). However, the heterogeneous, discontinuous, and high void density structure that contributes to excellent insulating properties, lead to a decrease in their mechanical properties. This demonstrated that these materials are suitable for substituting traditional materials in insulating applications. Additionally, antifungal tests were carried out. However, hemp nonwovens proved to be inefficient against fungal proliferation.

Keywords

Hemp fiber, wet-laid, nonwoven, insulating material, sustainable material, textile wastes

The concept of sustainability is becoming increasingly important in today's society. In recent years, the evidence of climate change, the problem of solid waste, the current use of fossil fuels, the depletion of oil, the lack (or very low) of renewable energies, and so on, are concerning to modern society.[1] Similarly, this has pushed the scientific community to research, optimize, and develop new materials to meet these social demands. In this sense, the so-called sustainable materials are becoming increasingly relevant given the possibility of becoming suitable substitutes for conventional, non-renewable, and environmentally unfriendly materials.[2] One of the main fields related to sustainable development is the use of residues, wastes, by-products, among others, as a source of environmentally friendly products with high added value. This approach can be positive from different standpoints. First, by upgrading a waste, it is removed from the production chain. Secondly, since it is a waste, it is usually a cost-effective material. Therefore, its new application will provide a clear economic advantage for the manufacture of a new product. In recent years, research focused on natural wastes, mainly lignocellulosic wastes coming from several sectors, have attracted great attention due to their sustainable character.[3,4]

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Within this ambit, the textile industry is one of the fields that uses and processes fibers and fabrics from natural origin. Since ancient times, several civilizations have made fabrics from plant fibers that grew in their environment, and one of these natural fibers is hemp. Textile archaeologists found the first traces of hemp fabrics in China (8000 BC) and Kazakhstan (4000 BC). Texts mention a hemp rope mixed with vegetation grains in the Gallo-Roman period (50 AD).[5] Nowadays, hemp fibers have become so important that in 2005 the "European Confederation of Flax and Hemp (CELC)" was created at the European level, with industries, universities, and research centers to promote the use of these fibers with proven environmental advantages. CELC confirms that hemp fiber is now recognized for its mechanical, thermoregulatory, antibacterial, and antifungal properties, which are suitable for insulation and the manufacture of technical fibers, composite materials, and lightweight concrete components, making it a worthy candidate for green building.[6,7] At present, hemp is a modern resource, a component of new bio-based materials that are at the forefront of innovation, with France being the largest producer in Europe.[5] In the USA a business volume of $600 million was generated in 2018. Hemp-derived products are usually found in industrial sectors such as textile, agriculture, automotive, food and drinks, paper, furniture, construction, recycling, and personal care, with a number of products close to 25,000.[5] Some of the most relevant properties are: low density, good stiffness, vibration absorption, thermal insulation, acoustic insulation, and 100% biodegradability.[8,9] The chemical composition of hemp is approximately 67–78% cellulose, 16–18% hemicellulose, 0.8% pectin, 3.5–5.5% lignin, and 0.7% wax.[10]

The textile industry processes hemp plant into fibers, fabrics, ropes, yarns, and household textiles. Hemp fiber has been widely used in natural fabrics because of its durability and versatility.[5] However, manufacturing of hemp fibers and fabrics produces significant amount of waste in the form of short fibers, which are removed from the production process.[11] As hemp is a natural fiber, it is possible to use its waste for other purposes. In this sense, an interesting application is the manufacture of nonwoven fabrics using wet laying technology, as it allows the incorporation of large quantities of waste (above 90 wt%). From a manufacturing point of view, wet-laid is a very efficient technology when it comes to using waste in the form of short fibers or particles, as it can handle large volumes of these wastes.[12–14] Moreover, from an economic point of view, its main advantage is the low cost of raw materials.

Nonwovens have been optimally used as thermal and acoustic insulation materials,[15,16] as their lightweight, void-filled structure gives them excellent insulating performance, enabling their use in various industrial fields such as automotive,[17] interior design,[18] transportation, and aeronautics, among others.[19,20]

The main objective of this study was the development of new sustainable materials for thermal and acoustic insulation by using natural waste obtained from the processing of hemp fibers in the textile industry. By using wet-laid technology, nonwoven structures with a high content of hemp waste and different proportions of binder fibers were studied. Optimization of the hemp waste/binder fiber content in relation to the characterization of the nonwoven determined the applicability of these new sustainable materials as insulating components.

Experimental

Materials

The hemp residue was provided by the French Institut Catholique d'Arts et Métiers (ICAM) and comes from the waste generated after the hemp fiber spinning process. As thermo-bonding material, thermoplastic polylactide (PLA) fiber especially designed for wet-laid applications was used. This PLA fiber was supplied by Trevira GmbH (Hattershein, Germany) with reference 260, a fiber length of 6 mm and a melting temperature of 127°C. This PLA fiber is crimped solid PLA (Ingeo) fibre for wetlaid applications, with a linear density or title of 1.7 dtex. Other binding fibers were also used to provide cohesion to the web. These binders were viscose fibers supplied by STW Fibers (Schwarzwälder Textil-Werke GmbH, Schenkenzell, Germany). They are artificial fibers that come from regenerated cellulose, with reference ZW gl 1.7/6T and a length of 6 mm and a linear density or title of 0.9 dtex. Figure 1 shows an optical image of the selected fibers.

Manufacturing of nonwovens by wet-laid

Table 1 shows the different formulations and codes of nonwovens with hemp fiber waste. The hemp fiber waste content ranged from 70–90 wt%, while different combinations of binding fibers were used to obtain nonwovens with different areal densities of 100 and 300 g/m^2. The nomenclature used for each wet-laid was: the corresponding areal density (100 or 300 g/m^2), hemp waste (H); poly(lactic acid) (PLA) fiber, and viscose (V) fiber. Three specimens were measured for the calculation of the final mass per unit area.

As it can be seen, there is some difference between the target (theoretical weight) and the actual weight.

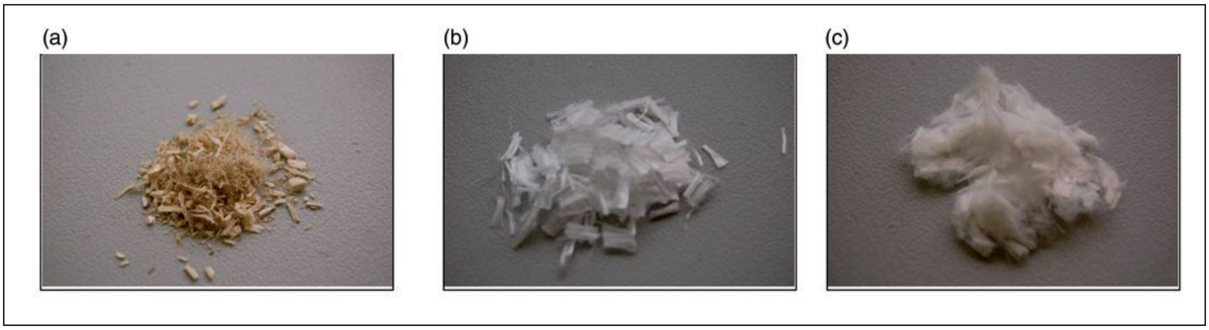


Figure 1. Images of the selected fibers for nonwovens, (a) hemp fiber, (b) polylactic acid fiber, and (c) viscose fiber.

Table 1. Composition and codes of the developed nonwovens from hemp fiber wastes by wet-laid process

Code	Composition (wt%) Hemp waste	PLA	Viscose	Theoretical mass per unit area t (g/m^2)	Final mass per unit area (g/m^2)
100/70H/20PLA/10V	70	20	10	100	129.8 ± 0.03
100/80H/10PLA/10V	80	10	10	100	122.4 ± 0.02
100/90H/5PLA/5V	90	5	5	100	117.7 ± 0.05
300/70H/20PLA/10V	70	20	10	300	447.8 ± 0.06
300/80H/10PLA/10V	80	10	10	300	410.8 ± 0.03
300/90H/5PLA/5V	90	5	5	300	397.1 ± 0.10
300/70H/30PLA	70	30	–	300	–
300/80H/20PLA	80	20	–	300	395.9 ± 0.03
300/90H/10PLA	90	10	–	300	380.1 ± 0.01

H: hemp waste; PLA: polylactide; V: viscose fiber.

This is directly related to the processing conditions by wet-laid. In general, the obtained weight is always higher than the theoretical weight. The actual weight is much more representative because it is a consequence of the processing parameters in the hydroformer.

Figure 2 shows the different stages carried out for the production of each of the nonwoven formulations based in hemp fiber wastes by means of the wet-laid technology.

Firstly, the different proportions of hemp waste, PLA, and viscose were weighted (Table 1). Then, they were poured into a pulper with water, in order to achieve adequate fiber separation and homogeneous fiber dispersion. Stirring in the pulper was carried out at 2400 rpm for 10 min with a fiber in water concentration of 10 g/l. The pulper was supplied by PILL Nassvliestechnik GmbH, (Reutlingen, Germany). In a second stage, the homogeneous mixture obtained in the pulper was poured into a dispersion tank with a 1200 l volume capacity. During this stage, the mixture was diluted with water to a fiber concentration of 1 g/l, and then stirred at lower rate of 200 rpm for 10 min, to maintain the fiber dispersion.

In the third stage, the water/fiber mixture was placed, with hydraulic pumps, onto a porous conveyor belt that acts as a filter that allows water to pass through, with an advance speed rate of 1 m/min, which allows the nonwoven web formation. The hydroformer machine was supplied by Pill Nassvliestehnik GmbH (Reutlingen, Germany) with a width for the web formation of 510 mm, and a tilt angle of 20° (Figure 3).

Once the nonwoven web was formed, it was transferred to a heated tunnel with a length of 3 m for thermal consolidation in a hot air thermal drying process. The drying module was supplied by Tacome S.A., mod. SDT-600 (Valencia, Spain). The nonwoven was maintained at 180°C during the drying process with a drying time of 7 min at 180°C. It is worth noting that this thermal process allows water removal as well as nonwoven consolidation since the temperature (180°C) allows partial or total melting of the low melting temperature PLA fiber (127°C).

Dimensional characterization

In order to determine the nonwoven thickness, UNE-EN ISO 9073-2 was used. A micrometre supplied by Sodemat TP (Ancenis, France) was used to obtain the thickness, with a pressure of 0.5 kPa. All measurements were carried out at a temperature of 20 ± 2°C and a relative humidity of 65 ± 4%. The areal density was

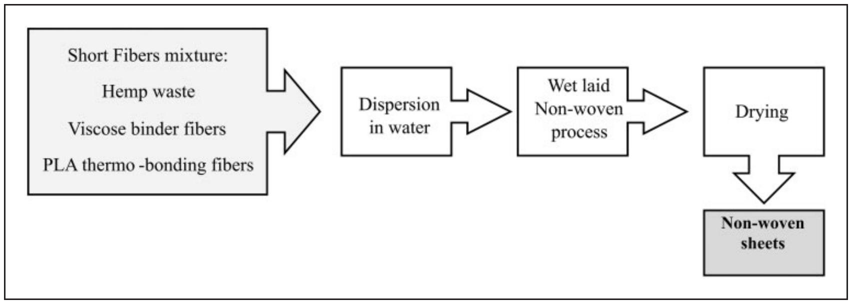


Figure 2. Schematic plot of the different stages of the wet-laid process with hemp fiber wastes. PLA: polylactide.

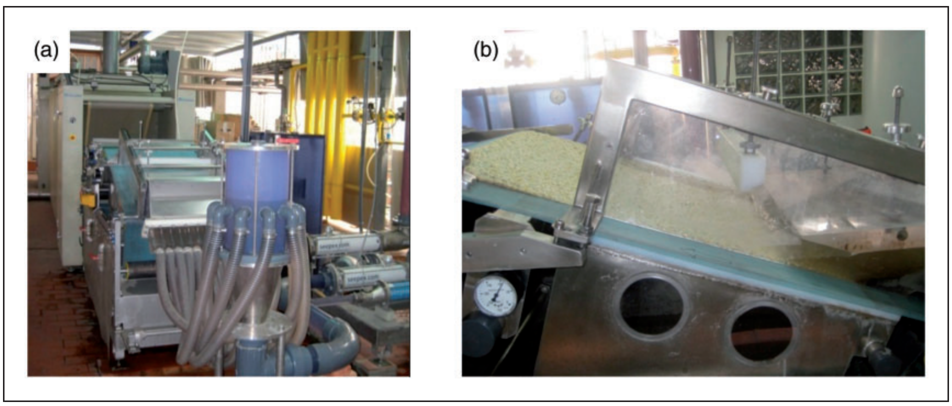


Figure 3. Images of the wet-laid equipment: (a) hydroformer and (b) detail of the conveyor belt on which, the nonwoven web is formed.

obtained according to the guidelines of UNE-EN 29073-1:1993. All measurements were taken at least 10 times in order to give reliable data. Moreover, to study the structural and surface characteristics of the nonwoven webs, a Leica stereomicroscope, model MZ 12.5, (Heerbrugg, Switzerland) was used.

Mechanical characterization

The mechanical characterization of the nonwovens was carried out by tensile tests, following UNE-EN ISO 29073-3:1993. An Instron dynamometer, model 4501 (Instron, Barcelona, Spain) was used to obtain tensile properties. The distance between clamps was set to 200 mm and a crosshead speed rate was 100 mm/min. Transversal and longitudinal tests were carried out on five samples in order to obtain reliability.

Thermal and acoustic insulation characterization

The acoustic absorption coefficient, α_n, was obtained following the guidelines of UNE-EN ISO 10534-2:2002. An impedance tube or Kundt tube, type 4206, supplied by Brüel & Kjaer (Naerun, Denmark) was used. This device works in a frequency range between 50–6000 Hz. Three circular samples with a diameter of 100 mm were used for the test to work at low frequencies, and three samples with a diameter of 29 mm were used to evaluate the acoustic properties at high frequencies.

The heat transmission resistance or thermal resistance (R), and thermal conductivity (λ), were obtained according to UNE-EN 12667:2002, in a heat flow measurement device, model HFM 436/3/1E Lambda, supplied by NETZSCH (Selb, Germany). Three different samples were tested, and the obtained results were averaged.

Antifungal characterization of nonwovens

To evaluate the resistance of the hemp-based nonwovens against fungus proliferation, different methodologies following UNE-EN 14119:2004 (A1 method, B1 method, and B2 method), and American Association of Textile Chemists and Colorists (AATCC) 30-2004. II and III methods were carried out. The results were evaluated qualitatively and consisted of visual inspection of the surface after the recommended incubation period. The results were given according to a rating scale shown in Table 2.

Morphological characterization

The morphology of hemp nonwovens was carried out with a scanning electron microscope, model FEI

Table 2. Scale for a qualitative assessment of the fungal growth

Observation	Growth degree	
There is no growth		0
There is growth, but only visible in a microscope		1
There is growth and it is visible for the human eye (macroscopic)	Visible, covered until 25%	2
	Visible, covered until 50%	3
	Considerable, covered more than 50%	4
	Strong, 100% covered	5

Quanta 200 from FEI Company (Oregon, USA) working at an acceleration voltage of 25 kV. Before surface characterization, samples were metalized with a sputtering of Au/Pd from Emitech mod. SC7620 (Quorum Technologies Ltd, UK).

Results and discussion

Surface morphology and tensile properties of hemp-based nonwovens

Figure 4 shows the appearance of the developed nonwovens. It is worth noting that for the nonwoven with reference 100/90H/5PLA/5V the obtained web was poorly consolidated and showed poor cohesion due to the large amount of hemp fiber. In a similar way, the nonwoven coded as 300/70H/30PLA showed PLA fiber aggregation and, subsequently, the cohesion was not homogeneous. These two nonwovens were discarded for further characterization since they did not offer good cohesion and homogeneity.

Evaluation of the surface morphology of the nonwoven samples allowed a detailed analysis of the structure formed between the hemp waste and the binding fibers. This structure is important to understand the mechanical properties of the different nonwovens. Figure 5 gathers the SEM images of the nonwovens with 80 wt% hemp waste. As it can be seen, all of them were characterized by a high discontinuity in their structure. Short hemp fibers can be clearly identified by their high diameter and woody appearance, as they are natural lignocellulosic fibers (marked by white arrows). It should be noted that since hemp fiber comes from industrial waste, there was quite a difference between the size of hemp fibers. As the three samples shown in Figure 3 have the same amount of hemp waste, all three images show a homogeneous layout of those fibers, which indicates good dispersion during the formation process of the nonwoven. In addition, the long binding fibers of PLA and viscose are clearly visible, forming an entanglement/web/framework that catches the short hemp fibers. The thermoplastic nature of PLA fibers, with a melt temperature of 127°C, led to partial melting during the drying stage at 180°C. The bonding fibers favor the consolidation of the nonwoven as they form thermo-bonds between the fibers (marked with a white circle). It was also observed that, although the drying step was carried out at 180°C, the waiting time of the nonwoven in the drying unit was relatively short and, therefore, the PLA fibers did not fully melt.

However, the main morphologic issue of nonwovens was the high void density and a lack of continuity in their structure, which would be responsible for their insulation properties and poor mechanical performance. The wet-laid technology employed, provides nonwovens with randomly dispersed hemp fibers and as a result the material does not show continuity.[12,13] It is noteworthy that a high content of hemp waste was used, in the order of 70–90% wt%; in contrast a very small amount of binding fibers are contained, so there will not be a continuous phase or matrix with fully embedded hemp fibers. In fact, wet-laid allows reaching this high fiber content in contrast to other conventional manufacturing techniques such as injection moulding, extrusion among others.[21] The consolidation of the nonwoven was mainly due to the lignocellulosic fibers of the hemp waste being "trapped" between the longer binding fibers[22] and, in some cases, thermal bonding of the PLA fibers due to their partial melting. These binder fibers formed a network that allows the consolidation of the nonwoven with a high load of hemp waste. However, the lack of stable bonds between fibers and residue allowed to foresee a low resistant response since load transfer is not allowed.

The inhomogeneity and the discontinuity observed in the hemp-based wet-laid nonwovens, was also responsible for some anisotropic behaviour, that can be clearly observed in Table 3, which gathers the main mechanical properties obtained from tensile tests. The maximum tensile force and, the elongation at the maximum tensile force are different in the longitudinal or transversal direction to web formation during the wet-laid process.

The tensile performance of the low areal density nonwoven samples, 100 g/m^2, showed a strong influence of the hemp waste loading. Both maximum force

Figure 4. Visual appearance of the hemp fiber based nonwovens with different binding systems.

and elongation were higher for the samples with less hemp waste content, and decreased with increasing natural fiber content. This is due to the sample with 70wt% of hemp waste being the one with more binding fibers, 30 wt%, which allowed more intensive consolidation/cohesion in the nonwoven structure, providing higher tensile strength and higher elongation at break. Nonwovens with 90 wt% hemp waste showed low values for the maximum force of about 2 N (very poor mechanical properties), that were discarded for further characterizations, since most of them broke while handling. This almost nonexistent mechanical resistance was due to the lack of compaction and the high discontinuity in the structure, because of the shortage in binding fibers (only 10 wt%) contained in the corresponding nonwoven formulations, as can be observed in their transversal SEM images (see Figure 6(a)).

Regarding the high areal density nonwovens with a nominal value of 300 g/m^2 and the PLA and viscose binding fibers, a major resistant response was observed in the transversal direction to the advance axis during web forming. The maximum tensile force decreased with increasing hemp waste content. The nonwoven with 70 wt% hemp waste showed maximum longitudinal and transversal forces of 26 N and 35 N, respectively, and they decreased to 13 N and 26 N for the nonwoven containing 90 wt% hemp fiber. This resulted in a decrease of 50% for the maximum longitudinal force and 26% for the transverse force. This tendency was also observed in elongation values which, for the same hemp fiber contents, decreased approximately by 36%. As has been previously mentioned, this effect is due to the higher content in lignocellulosic fibers in nonwovens, the lower the content is in binding fibers,[23] responsible for giving consistency to the material, so the mechanical performance notably decreases. High volumes in hemp fibers (90 wt%) did not allow PLA (5 wt%) and viscose (5 wt%) fibers to entangle all hemp fibers and the resulting material contains high void density. As expected, high areal density nonwovens provide better properties compared to their low areal

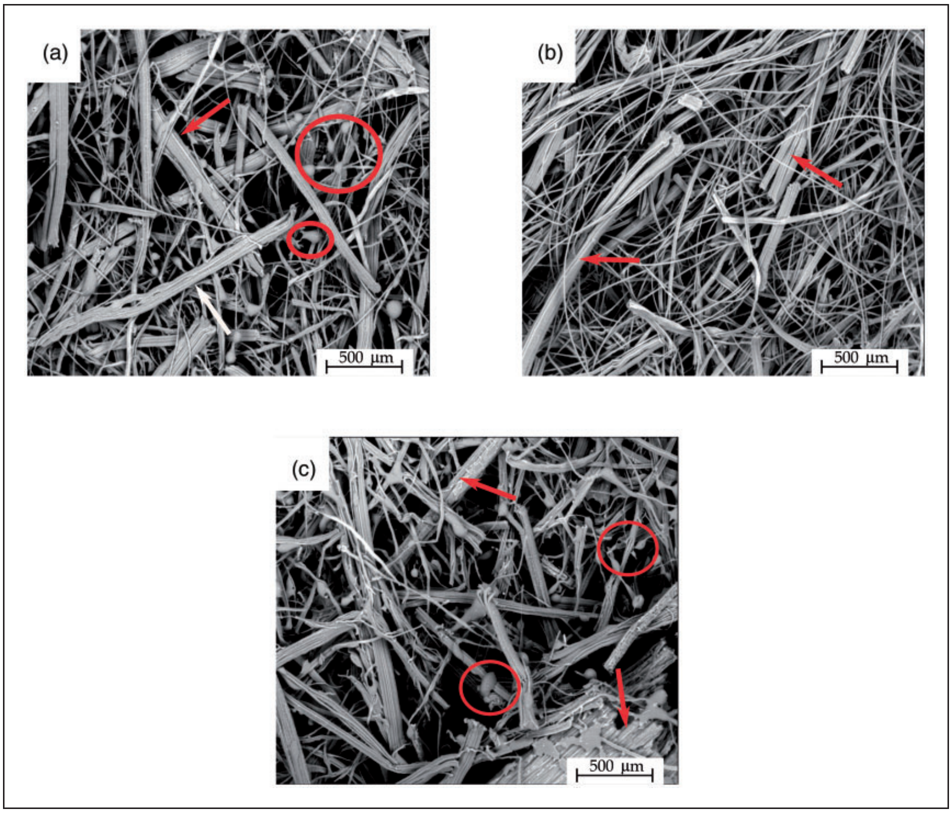


Figure 5. Scanning Electron Microscopy (SEM) images of the superficial morphologies of the samples: (a) 100/80H/10PLA/10V, (b) 300/80H/10PLA/10V and (c) 300/80H/20PLA (100×).

Table 3. Mechanical properties of hemp waste nonwovens obtained by wet-laid, measured in the longitudinal and transversal direction to the web formation direction

Code	Direction of application of the force	Maximum force (N)	Elongation at maximum force (%)	Average thickness (mm)
100/70H/20PLA/10V	Longitudinal	20 ± 0.04	3 ± 0.11	1.99 ± 0.13
	Transversal	13 ± 0.05	3.4 ± 0.11	
100/80H/10PLA/10V	Longitudinal	11 ± 0.02	2.2 ± 0.10	1.56 ± 0.15
	Transversal	11 ± 0.14	2.8 ± 0.18	
100/90H/5PLA/5V	Longitudinal	2 ± 0.39	1 ± 0.33	1.61 ± 0.12
	Transversal	2 ± 0.18	1 ± 0.18	
300/70H/20PLA/10V	Longitudinal	26 ± 0.23	2.2 ± 0.12	4.55 ± 0.18
	Transversal	35 ± 0.6	2.2 ± 0.11	
300/80H/10PLA/10V	Longitudinal	23 ± 0.23	2 ± 0.19	4.44 ± 0.07
	Transversal	29 ± 0.04	2 ± 0.10	
300/90H/5PLA/5V	Longitudinal	13 ± 0.16	1.6 ± 0.24	3.55 ± 0.11
	Transversal	25 ± 0.05	1.4 ± 0.72	
300/70H/30PLA	Longitudinal	0.5 ± 0.03	—	3.95 ± 0.07
	Transversal	0.6 ± 0.04	—	
300/80H/20PLA	Longitudinal	12 ± 0.39	1.6 ± 0.19	3.93 ± 0.06
	Transversal	18 ± 0.17	1.4 ± 0.22	
300/90H/10PLA	Longitudinal	19 ± 0.17	1.4 ± 0.03	3.12 ± 0.12
	Transversal	20 ± 0.10	1.2 ± 0.16	

H: hemp waste; PLA: polylactide; V: viscose fiber.

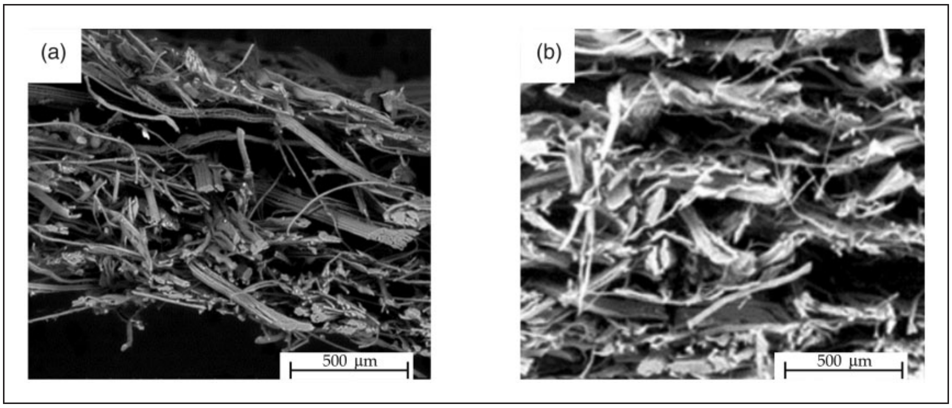


Figure 6. Scanning electron microscopy (SEM) images of the morphologies of the transversal sections of the samples: (a) 100/90H/5PLA/5V and (b) 300/70H/30PLA (160×).

density counterparts. The reason for this was a significant difference in nonwoven thicknesses (Table 3). Nonwovens with a nominal areal density of 100 g/m^2 gave an average thickness of 1.7 mm, while nonwovens with a nominal areal density of 300 g/m^2 offered higher thickness, with an average value of about 3.9 mm.

On the other hand, samples with high areal density and only thermo-bonding PLA fibers, showed different tensile behavior. The sample with higher content in PLA (30 wt%) showed a null tensile strength, namely 0.5 N longitudinal maximum force and 0.6 N transversal force. For this reason, 300/70H/30PLA nonwoven was excluded from further characterization. This behavior was caused by a noticeable lack of homogeneous dispersion of PLA fibers, as can be observed in its cross-section image (Figure 6(b)). The resulting nonwoven was characterized by the lack of bonding between the fibers and poor consolidation. Interestingly, nonwovens with 20 wt% and 10 wt% showed tensile mechanical behavior similar to that of the nonwoven with PLA/viscose as the binding system. This is observed both in the maximum force, with values between 12–20 N, and in the elongation, increasing from 1.2% to 1.6%. These results support the possibility of replacing an artificial binding fiber such as viscose, with a biodegradable fiber such as PLA as binder in nonwovens with waste hemp fibers.[24]

Table 4. Results of average thermal conductivity, λ, and average thermal resistance, *R*, of the nonwoven materials of hemp residue

Code	Thermal conductivity λ (W/(m K))	Thermal resistance R ((m^2 K)/W)
100/70H/20PLA/10V	0.029 ± 0.001^a	0.040 ± 0.001^a
100/80H/10PLA/10V	0.027 ± 0.001^b	0.038 ± 0.001^a
300/70H/20PLA/10V	0.032 ± 0.000^c	0.151 ± 0.002^b
300/80H/10PLA/10V	0.032 ± 0.000^c	0.149 ± 0.001^b
300/90H/5PLA/5V	0.029 ± 0.000^d	0.108 ± 0.002^c
300/80H/20PLA	0.028 ± 0.000^d	0.110 ± 0.001^c
300/90H/10PLA	0.034 ± 0.000^e	0.144 ± 0.001^d

H: hemp waste; PLA: polylactide; V: viscose fiber.
[a–e]Different letters in the same column indicate a significant difference among the samples ($p < 0.05$).

Thermal insulation of hemp-based nonwovens obtained by wet-laid

Thermal insulation properties of nonwovens with high content of hemp waste, processed with wet-laid technology, were obtained. Results are reported in Table 4. Thermal conductivity (λ) is one of the most important parameters for determining the thermal insulating properties of a material. This parameter depends on a number of factors such as morphology, density, or homogeneity.[25] The quantification of the thermal conductivity (λ) of the nonwovens with natural hemp waste does not depend on the thickness of the material, as it is an intrinsic property. Therefore, the values obtained were within a narrow range of 0.027–0.034 W/(m K) and were not affected by the areal density of either 100 or 300 g/m^2. This narrow change in λ value seems to indicate that it does not depend on the hemp fiber content nor on the binding fiber content. Therefore, they do not seem to affect the thermal insulating properties of the studied nonwovens noticeably. However, the thermal insulating capacity of the nonwovens with high natural fiber content was remarkable. The λ values were even lower than those presented by the hemp fiber between 0.040–0.060 W/(m K).[6,25,26] The internal structure of the nonwovens analyzed above (Figures 5 and 6), which had a high density of voids, hinder the heat transfer process as there is no continuity in the internal structure of the material. Xue et al.[27] reported that those voids full of air make the heat flow transmission process more difficult. It must be taken into account that the thermal transmission coefficient of air is <0.02 W/(m K).[6,27,28] Thus, nonwovens with lignocellulosic hemp fibers have high efficiency as heat-insulating materials.[29] The thermal conductivity values obtained for all nonwovens were in the range 0.027–0.034 W/(m K) range. These values were even lower than those of traditional thermal insulation materials. For instance, cork has a λ value of 0.038 W/(mK), while glass wool and rock wool give a λ of 0.037 W/(m K) and 0.033 W/(m K), respectively. Similar values have been reported for expanded polystyrene.[5,11,18,30] Therefore, the obtained hemp nonwovens are potential substitutes for traditional materials used in thermal insulation applications.[31] Some possible applications could be textile elements for improved military cold weather clothing, hand wear, and sleeping bags, where a minimum heat transfer is essential.[32] Another potential field of application would be the automotive industry, where these materials could be used for covering seats, door panels, or headliners. The Technical Building Code considers a material to be "thermally insulating" when it has a thermal conductivity below 0.060 W/(m K). All nonwoven formulations developed based on hemp fiber have lower λ values, so they can be categorized as thermal insulators.

The thermal resistance (R) values, however, showed clear differences between hemp waste nonwovens as a function of areal density. The lowest thermal resistance was observed in low areal density nonwovens (nominal areal density of 100 g/m^2), with values close to 0.04 (m^2 K)/W. These values were almost tripled for nonwovens with an areal density of 300 g/m^2. Hemp fiber nonwovens bonded with PLA and viscose fibers showed some dependence of R on the hemp waste content. Nonwovens with 70 wt% and 80 wt% of this hemp fiber showed similar R values of 0.151 and 0.149 (m^2 K)/W, respectively. The R value of the nonwoven with 90 wt% hemp fiber decreased to 0.108 (m^2 K)/W, which resulted in a loss of thermal insulation efficiency of about 28%, for nonwoven thicknesses of about 4 mm. However, nonwovens with 90 wt% hemp fiber and thermally bonded with PLA fiber, gave an R value of 0.144 (m^2 K)/W, which is representative of higher thermal insulation ability. Nevertheless, no significant influence of the type of binder fibers (PLA-viscose) on the thermal insulation performance was observed, as it was demonstrated by the statistical analysis.

These results suggest that the formation of the nonwoven by the wet-laid method promotes the existence of a large amount of air trapped inside the structure. This decreases its thermal conductivity and increases its thermal insulating efficiency, which corroborates the potential applications mentioned above for military and automotive industries.

Acoustic insulation of hemp fiber nonwovens

Figure 7 shows the variation of the acoustic absorption coefficient as a function of sound frequency for a single layer of the nonwovens obtained.

The graph shows that all analyzed nonwovens with high hemp fiber loading showed low acoustic absorption coefficient values at low sound frequencies (under 600 Hz), with small variations in the range 0.003–0.006. In this low frequency range, the difference in hemp fiber content as well as the binding system did not affect the acoustic absorption coefficient values, as the differences were very low. In addition, an increase in α_n was observed with increasing frequency. In the mid-frequency range, between 600 Hz and 2 kHz there was a significant increase in the acoustic absorption coefficient which reached values between 0.003–0.12. For these medium sound frequencies, nonwovens with the higher areal density (nominal value of 300 g/m^2) showed the highest acoustic absorption coefficients. Following the same analysis, at higher sound frequencies in the range of 2–6 kHz, a rapid increase in acoustic absorption capacity was detected. Figure 7 shows a small increase in α_n for low areal density nonwovens of about 100 g/m^2, although from a quantitative point of view they tripled their value ($\alpha_n = 0.09$) compared to low frequencies. Hemp nonwoven fabrics with a nominal areal density of 300 g/m^2 showed a more noticeable increase in the acoustic absorption coefficient because they are thicker samples of about 4 mm. In comparison, the acoustic absorption coefficient obtained at 6 kHz, was higher in the samples with PLA and viscose as the binding system.

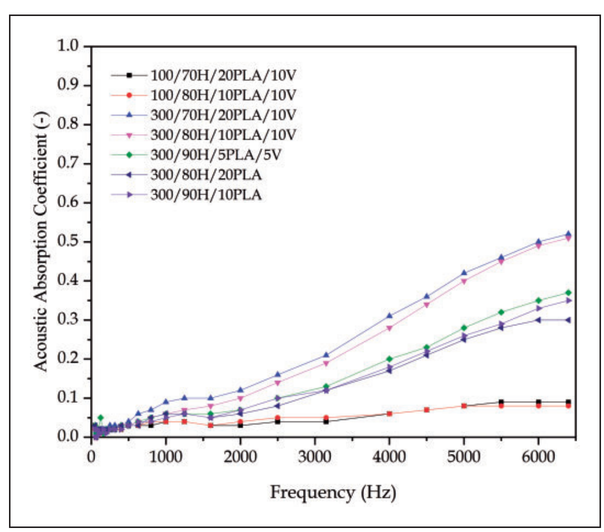


Figure 7. Acoustic absorption coefficients comparison (α_n) referring to sound frequency, in a single layer of hemp fiber nonwovens obtained by wet-laid with different binding systems and hemp fiber content.

Hemp nonwovens coded as 300/70H/20PLA/10V and 300/80H/10PLA/10V exhibited the highest acoustic absorption capacity for a single nonwoven layer with hemp fiber, with values around 0.5 at a frequency of 6 kHz. This sound absorption capacity at high frequencies decreased around 35% for the rest of the 300 g/m^2 nonwoven formulations. Bhuvaneshwari et al.[33] reported the acoustic insulation effect of hemp fibers.

To corroborate the effect of the thickness on acoustic absorption, Figure 8 illustrates the same analysis for three stacked layers of each nonwoven. The variation of the acoustic absorption coefficients as a function of sound frequency was the same as that observed for a single nonwoven layer. At low-medium sound frequencies, the absorption capacity was lower in all samples. For high frequencies, above 1000 Hz, a rapid increase was observed. As for their quantification, the materials with the lowest acoustic absorption were again those with low areal density: 100/70H/20PLA/10V and 100/80H/10PLA/10V, since they had a lower thickness (5.9 and 4.7 mm respectively with three stacked layers of the corresponding nonwovens). For mid frequencies around 2 kHz, the acoustic absorption coefficient doubled, reaching values of approximately 0.07 and 0.08, respectively, compared to 0.03 and 0.04 obtained with a single nonwoven layer. Regarding high frequencies, at 6 kHz, the three-layer samples tripled the acoustic absorption capacity relative to their single-layer counterparts.

For high areal density nonwovens of 300 g/m^2, the absorption coefficients were higher for three-layer samples than for single-layer samples over the entire frequency range studied for all materials. Graphically, it

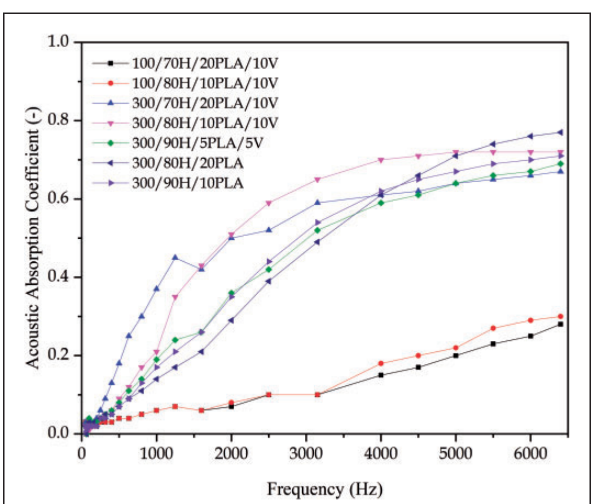


Figure 8. Acoustic absorption coefficients comparison in function of sound frequency, for three nonwoven layers of hemp fiber nonwovens obtained by wet-laid with different binding systems and hemp fiber content.

can be seen how the highest values were obtained in the high frequency region at 6 kHz, with very little change in the 0.66–0.76 range. This indicated that neither the hemp waste content (wt%) nor the content of the binding system significantly affected the acoustic absorption. The highest values of acoustic absorption coefficient were registered for high areal density nonwovens with three layers and high hemp fiber content of 80 wt% and 90 wt%. In particular, it is worth mentioning these nonwovens: 300/80H/20PLA and 300/80H/10PLA/10V with acoustic absorption coefficients of 0.76 and 0.72 respectively at 6 kHz. Considering that a acoustic absorption coefficient =1 would be considered as a total acoustic insulator, nonwoven hemp waste can be considered good acoustic insulating material. Balciunas et al.[6] reported that the structure of open-pore materials, which present a continuous communication air path with an external surface, is responsible for the good acoustic absorption properties. Nonwovens possess this kind of structure, as they have a high void density.

This acoustic absorption capacity over a wide frequency range of hemp fiber nonwovens was compared with the α_n values of traditional materials used in acoustic insulation and/or acoustic absorption applications. Table 5 lists the relationships between the acoustic absorption coefficient and the thickness of the hemp fiber nonwovens, as well as typical values for traditional materials such as polyurethane foam, wood panel, polyester wool, cork, rock wool, glass wool, glass window, and plasterboard.[6,18,34] The ratio of acoustic absorption coefficient to thickness was more useful for comparing efficiency or yield in sound absorption capacity, as it takes into account the total thickness of the material under consideration, and thickness is a critical parameter in sound absorption.

The results in Table 5 demonstrate the effectiveness of these hemp waste fiber nonwovens obtained by wet-laid technology for applications as acoustic insulating materials. The relationship between the acoustic absorption coefficient and the thickness of the hemp-based nonwovens, compared to the values of traditional materials currently used in commercial applications, show a significant advantage of hemp fiber nonwovens in the frequency range studied. Nonwovens developed with a high fiber content from hemp waste are worthy competitors to traditional materials used in acoustic absorption applications,[33] such as noise protection in car interiors regarding luggage compartments, floor-carpet underlay mats, A-B-C pillars, among others,[35] as well as in applications in the construction industry (green building initiatives), in sound absorbing surfaces. Moreover, being based on hemp fiber waste, they represent a sustainable solution to acoustic absorption.

Table 5. Comparison of the acoustic absorption coefficient to thickness ratio (α_n/mm) of hemp fiber nonwovens obtained by wet-laid with different binding systems and hemp fiber content, and some acoustic insulating commercial materials

	Acoustic absorption coefficient to thickness ratio (α_n/mm) for different frequencies (Hz)						
Code	125	250	500	1000	2000	4000	6000
100/70H/20PLA/10V	0.010^{a}	0.010^{a}	0.015^{a}	0.020^{a}	0.015^{a}	0.03^{a}	0.045^{a}
100/80H/10PLA/10V	0.064^{b}	0.013^{b}	0.02^{b}	0.025^{b}	0.025^{b}	0.038^{b}	0.051^{b}
300/70H/20PLA/10V	0.004^{c}	0.006^{c}	0.009^{c}	0.02^{c}	0.026^{b}	0.07^{c}	0.11^{c}
300/80H/10PLA/10V	0.004^{c}	0.004^{d}	0.007^{d}	0.13^{d}	0.022^{c}	0.063^{d}	0.11^{c}
300/90H/5PLA/5V	0.014^{d}	0.006^{e}	0.008^{e}	0.017^{e}	0.02^{d}	0.056^{e}	0.10^{d}
300/80H/20PLA	0.005^{e}	0.005^{f}	0.007^{f}	0.015^{f}	0.015^{e}	0.043^{f}	0.076^{e}
300/90H/10PLA	0.003^{f}	0.006^{g}	0.009^{g}	0.15^{g}	0.021^{f}	0.056^{g}	0.102^{f}
Polyurethane foam	0.003^{f}	0.004^{h}	0.010^{h}	0.024^{h}	0.026^{g}	0.030^{h}	0.030^{g}
Wood panel	0.029^{g}	0.022^{i}	0.017^{i}	0.008^{i}	0.011^{h}	0.011^{i}	0.011^{h}
Polyester wool	0.001^{h}	0.004^{j}	0.005^{j}	0.008^{i}	0.011^{h}	0.020^{j}	0.026^{i}
Cork	0.001^{h}	0.004^{j}	0.010^{k}	0.009^{j}	0.011^{h}	0.008^{k}	0.009^{j}
Rock wool	0.001^{h}	0.004^{j}	0.015^{l}	0.026^{k}	0.029^{i}	0.030^{l}	0.030^{k}
Glass window	0.070^{i}	0.050^{k}	0.036^{m}	0.026^{k}	0.014^{j}	0.008^{m}	0.006^{l}
Glass wool	0.006^{j}	0.010^{l}	0.015^{n}	0.020^{l}	0.026^{k}	0.030^{n}	0.030^{m}
Plasterboard	0.029^{k}	0.010^{l}	0.005^{o}	0.004^{m}	0.007^{l}	0.008^{o}	0.009^{n}

H: hemp waste; PLA: polylactide; V: viscose fiber.
$^{a\text{–}o}$Different letters in the same column indicate a significant difference among the samples ($p < 0.05$)

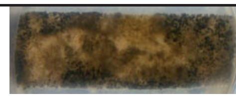
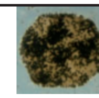
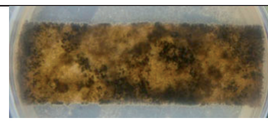
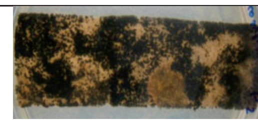
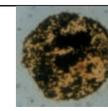
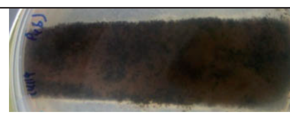
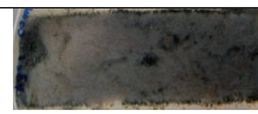
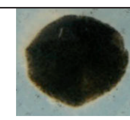
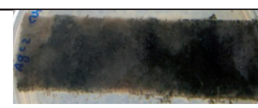
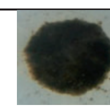
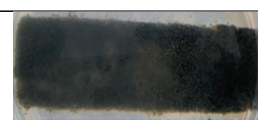
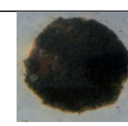
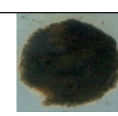
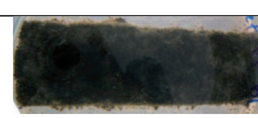
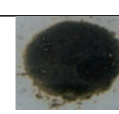
Characterization against fungal growth

Microscopic fungi can grow on textile products when environmental conditions are suitable for their proliferation. The action of microscopic fungi on natural textiles can cause deterioration, since their lignocellulosic nature serves as a nutritive substance for fungi and, therefore, favors their growth. Metabolic products of fungi may cause discoloration or biochemical deterioration.[36]

According to several studies, hemp fiber possesses intrinsic antifungal properties.[5,37,38] Standardized microbiological characterization tests were used to investigate whether the nonwovens with high hemp fiber content showed this property or not. Different standards were used to evaluate the antifungal resistance of the developed nonwovens. Table 6 shows, comparatively, the results obtained with different strains of microorganism and different growing media after two weeks of incubation at $29 \pm 1°C$. The microorganisms corresponding to the A.1 method, *Aspergillus niger, Chaetomium globosum, Trichoderma virens, Paecilomyces variotii*, and *Penicillium pinophilum* in a mineral salt medium proliferated homogeneously on the surface of the nonwovens. This indicated that the nonwovens were not resistant to attack by these microorganisms and that they possessed nutritious substances that favored their growth. The fungal growth formed a biofilm on the sample surface, with growth degree=5. The same behavior was observed for *Aspergillus niger, Chaetomium globosum, Trichoderma virens, Paecilomyces variotii*, and *Penicillium pinophilum* when a growing medium containing glucose and mineral salts was used, according to the B.1 method. The results obtained suggested that none of the hemp-based nonwovens developed showed resistance to these fungi, as the degree of attack was also =5 in these cases. These low antifungal properties exhibited by hemp waste nonwovens were corroborated with the B.2 method test, using a growing medium inoculated with *Aspergillus niger* spores. All of the samples analyzed showed a superficial fungal film covering the entire surface of the materials studied.

The study against fungi was completed with a test to assess potential antifungal capacity. Table 7 shows the comparative results obtained for all samples. The Method II, corresponding to a *Chaetomium globosum* inoculated medium, after 2 weeks of testing, showed a high degree of growth on the surface of all samples. Regarding the *Trichophyton rubrum* microorganism (Method III), 100/70H/20PLA/10V and 100/80H/10PLA/10V were those that performed best against this microorganism. Microscopic growth of the fungus was observed on the surface of the samples. This growth was not macroscopically visible. The rest of the samples did not provide good antifungal performance, as a massive macroscopic growth of the fungus was observed, covering the surface of the tested nonwovens with degrees of growth =4 and 5. These results demonstrated the low resistance of hemp fiber waste nonwovens to fungal growth, as well as their low antifungal behaviour. If used in applications as insulating

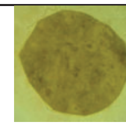
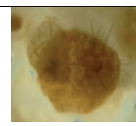
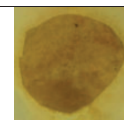
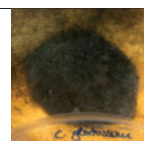
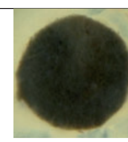
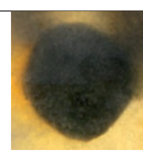
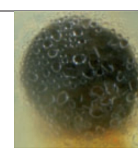
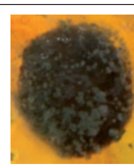
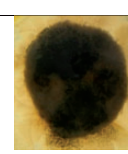
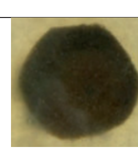
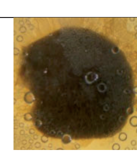
Table 6. Surface appearance of fungal growth of hemp fiber nonwovens obtained by wet-laid with different binding systems and hemp fiber content. Standard method A.1, B.1, and B.2

Sample	Method A.1	Method B.1	Method B.2
100/70H/20PLA/10V	 Degree of growth 5	 Degree of growth 5	 Degree of growth 5
100/80H/10PLA/10V	 Degree of growth 5	 Degree of growth 5	 Degree of growth 5
300/70H/20PLA/10V	 Degree of growth 5	 Degree of growth 5	 Degree of growth 5
300/80H/10PLA/10V	 Degree of growth 3	 Degree of growth 5	 Degree of growth 5
300/90H/5PLA/5V	 Degree of growth 5	 Degree of growth 5	 Degree of growth 5
300/80H/20PLA	 Degree of growth 5	 Degree of growth 5	 Degree of growth 5
300/90H/10PLA	 Degree of growth 5	 Degree of growth 5	 Degree of growth 5

H: hemp waste; PLA: polylactide; V: viscose fiber.

materials in environmental conditions that favor proliferation of fungi, they would not perform optimally. The wide variety of microorganisms tested showed a rapid growth on the surface of all nonwoven samples tested. In order to improve fungal behavior, it could be interesting to add antifungal elements into the composition of these nonwovens in future works, such as potassium iodide.[39]

Table 7. Surface appearance of fungal growth on hemp fiber nonwovens obtained by wet-laid with different binding systems and hemp fiber content. Standard American Association of Textile Chemists and Colorists (AATCC) 30, Method II and III

Sample	AATCC 30- Method II	AATCC 30- Method III
100/70H/20PLA/10V	 Degree of growth 5	 Degree of growth 1
100/80H/10PLA/10V	 Degree of growth 5	 Degree of growth 1
300/70H/20PLA/10V	 Degree of growth 5	 Degree of growth 4
300/80H/10PLA/10V	 Degree of growth 3	 Degree of growth 5
300/90H/5PLA/5V	 Degree of growth 5	 Degree of growth 4
300/80H/20PLA	 Degree of growth 4	 Degree of growth 4
300/90H/10PLA	 Degree of growth 4	 Degree of growth 4

H: hemp waste; PLA: polylactide; V: viscose fiber.

Conclusions

This work has reported the technical feasibility of the development of sustainable insulating materials, through the valorization of hemp waste from the textile industry, by wet-laid technology. Hemp nonwovens developed with high hemp content have proved to be worthy competitors to traditional materials used in thermal and acoustic insulation. In addition, it is important to note that they are sustainable materials due to the high content in hemp waste. The following points describe the most relevant findings of this work concerning the properties of the nonwovens.

- Their low thermal conductivity values between 0.027–0.034 W/(mK), were lower than those of traditional thermal insulating materials, such as rock wool or expanded polystyrene. Specifically, the sample with 100 g/m^2 of areal density and with a composition of 80% of hemp, 10% of PLA, and 10% of viscose presents the lowest thermal conductivity (0.027 W/(mK).
- The highest values of acoustic absorption coefficient were shown by the three-layered nonwovens with high areal density and high content of hemp waste (80 wt% and 90 wt%). Specifically, nonwovens with 300 g/m^2 of areal density, containing 80 wt% hemp fiber and 20 wt% binding fibers, exhibit the best acoustic absorption coefficient, with values at high frequencies (6 kHz) of 0.76 and 0.72 for 20 wt% PLA thermo-bonding fiber and 10 wt% PLA+10 wt% viscose binding system.
- The internal structure of the developed nonwovens provides thermal and acoustic insulation, competing with even the best insulating materials on the market.
- In addition, the results obtained showed the possibility of replacing the artificial binder fiber, such as viscose, with a biodegradable fiber such as PLA, in nonwoven formulations of hemp fiber waste. Thus, obtaining a sustainable and fully biodegradable material with very interesting technical properties.

These findings suggest that hemp nonwovens are perfect candidates for applications in a wide range of fields, but especially in the automotive and military industries where their acoustic and thermal insulation allows for the construction of nonwoven elements for cars and textiles for the military forces in order to improve comfortability and reduce energy consumption costs. However, more research is needed regarding the antifungal behavior of the materials developed here, maybe trying to introduce additional antifungal elements into their composition.

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