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Durability, recyclability and life cycle of green insulating panels hemp shiv based

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ABSTRACT

There is currently a growing need for sustainable and environmentally friendly materials. Previous studies demonstrate that a promising candidate is the use of hemp shiv and recycled cardboard for insulating applications. This study focuses on the durability, recyclability and life cycle of the material, in order to validate the real eco-friendly grade. To validate the durability of the material sample protected with two different vegetable coating (colophony and gum arabic) and the use of citric acid to enhance cellulose crosslinking were tested with different life span up to 17 months in ambient conditions. Thermal insulation properties were assessed measuring the heat transfer through the material. Acoustic insulation properties were evaluated using a Kundt tube within a frequency range of 100–6500 Hz. Mechanical tests, including compression, shear and bending, were performed to assess the material's strength. Results demonstrated that there is not noticeable reduction of the properties over the time tested, however, there is a fluctuation of the properties depending on ambient conditions (moisture manly). The 100% of the composite material could be reused as a raw material for remanufacture new panels. Nevertheless the life cycle analysis demonstrate that it is necessary to reduce the energy consumption during the fabrication to obtain a negative CO_2 emission.

1. Introduction

To effectively combat climate change, it is imperative to prioritize the creation of energy-efficient buildings, thereby minimizing the environmental impact of climate control systems. Additionally, the utilization of renewable, eco-friendly materials holds significant promise in reducing carbon emissions across all stages of material production, utilization and end of life. Developing novel biocomposite insulation materials presents an exciting avenue for achieving these goals, but ensuring their durability is essential for their eventual commercial viability. In this trends, several research have developed new eco-friendly insulating materials by using by-product of harvest plants [1–4]. However, for long-term applications, the main problem with these materials is their biodegradability. The longevity of biocomposites can be compromised by various ways of biological reduction, such as mold growth, as well as environmental factors including temperature and humidity fluctuations [5]. To study the durability of sustainable biobased composite the main aging test are: fungi resistance, fatigue, thermo aging, water aging and natural weathering [6]. The most common aging test for building materials involves immersion in alkaline water [7]. Novel hemp-based materials are typically manufactured using non-green binder materials, such as epoxy formaldehyde or mortar [8–10]. In these cases, water immersion serves as an appropriate aging test. However, for more sustainable materials where the binder is also natural, such as in some green materials, immersion in water is not feasible. For these cases, there is currently no standardized aging test for building materials. Despite

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the importance of long-term durability testing for these materials, existing literature on the subject is limited. There are different approach to reduce the degradation of the green materials over time, the most important are chemical treatment and to apply a superficial coating.

The main material proposed in this paper is hemp shiv, to produce a green insulation material for building sector. In previous publication, it was validate its mechanical, chemical and insulation properties. In the case of building sector, the common life cycle of a modern building is 100 year, consequently, its important to study how the environment could degraded it.

Investigating the water absorption characteristics of biocomposites is crucial, given the weak water resistance of biomaterial. For biocomposites used outdoors in construction applications, their water absorbency is a critical factor affecting their mechanical properties and dimensional stability [11]. In the case of hemp shiv, it also presents a low stability against the contact with water, its Young's modulus is reduced by 50% after immersion in water [12]. Moreover, using a 100% vegetable material increases the risk of mold growing in high humidity conditions [13]. Mold growth can be relieved by increasing the pH of the material, as is the case with hempcrete, which has an antifungal properties [14]. If the material is in contact with cement matrix the issue arises from the alkaline hydrolysis resulting from the production of $Ca(OH)_2$ during cement hydration. The calcium adsorption is pH dependent, the pectin contained in fibers can react with calcium ions in an alkaline environment [15]. Moreover, a reduction in compressive strength has been observed in hempcrete when using $Ca(OH)_2$ treated hemp shive compared to untreated ones [16,17]. To incorporate a material that is rich in Al_2O_3 and SiO_2 can mitigate the reduction of the hemp fibers. This material should have the ability to consume $Ca(OH)_2$ during mortar hydration [18]. Alternatively, the fiber can undergo physical or chemical treatment.

To prevent the degradation of the biomaterials, a solution is to introduce a coating in order to protect the material against the environmental effects. To increase the water resistance of these materials, surface coatings such as NaOH, silane and epoxy have been studied, which reduce the moisture absorption of the fibers [19–22]. For a more sustainable solution a vegetal resin is proposed. By coating a bamboo particle with pine resin, it is obtain a hydrophobic material, in addition, bamboo particles become a stable material under ambient conditions [23]. Nevertheless, the pine resin worsen the fire resistance properties. Gum arabic also presents good results as a coating in cases of humidity and fire, although it could not be used in direct contact with water, since gum arabic is soluble in water, so when the layer is in direct contact protective coating is removed until the material is uncoated [24]. Although these coatings increase the durability of the material, no studies have been found that investigate how the insulating properties behave when these coatings are added. The coating would not only have the function of protecting the material, but also of stabilizing the interior moisture. Since the moisture has a great impact on the insulating properties.

Also, it is crucial to ensure compatibility between the hemp shiv and the vegetal binder to achieve optimal results of the final composite material. Recycled cardboard fiber has demonstrated remarkable insulation capabilities and can be easily disintegrated to achieve desired bonding and shape through cellulose crosslinking. Citric acid, has been identified as a potential additive to enhance specific properties of paper products, including wet tensile, compressive strength and life span. As the curing temperature increases, the bonding percentage improves, starting with a temperature above the melting point of the acid [25]. The addition of citric acid has been found to significantly enhance the durability of cardboard boxes, increasing their lifespan by up to four times. This improvement is attributed to the reduction of OH groups, which enhances the resistance to moisture of the cellulose bonding [26,27].

Finally, to study if a material is truly an eco-friendly material is necessary to evaluate the ecological assessment [28,29] and to check the emissions and contaminant produced since the gathering of the raw material until the end of life. A life cycle analysis is methodology to evaluate how eco-friendly is a material by taking into account the electricity consumption, CO_2 emissions, transportation, use of contaminant materials, water by-product, transport, ecological impact of the material in the use phase and how to process the residues after its use. The life cycle analysis is an objective method to evaluate how ecological is a material and also to establish comparison between materials taking into account all the phases that are needed to use it. In the case of rockwool, the most common material for insulation in building, the produce an emission of 4.6 kg of CO_2 for every functional unit [30] and 5 kg of CO_2 eq. in the case of EPS [31].

This study proposes a comprehensive examination of the durability under ambient conditions, recyclability, and life cycle of a novel eco-friendly biocomposite material made with hemp shiv and recycled cardboard fiber previously published [1]. While previous research published has primarily focused on developing green materials for civil applications, there remains a crucial need to assess the long-term viability of these materials to corroborate that these materials can be use as real substitutes for traditional materials in the construction sector. The research delves into the evolution of acoustical and thermal insulating properties and mechanical properties (compression, shear and bending) over time in ambient indoor conditions using two green coatings (gum arabic and colophony) and the application of citric acid as a crosslinking agent to enhance the durability of paper fiber. Thermal and acoustical insulation and mechanical properties are systematically evaluated across different lifespans, providing insights into the material's performance over time. Moreover, the material will undergo a recycling process to validate the viability of the procedure by study the properties of the recycled material. Finally, to ensure that the final solution developed is truly an eco-friendly material, a life cycle analysis is conducted to assess the CO_2 emissions, water consumption, raw material and other contaminant emission produced by the material.

2. Materials

The green composite material is formed by a combination of hemp shiv and recycled cardboard fiber. Hemp shiv, Fig. 1, is a lightweight material with high insulation properties. It is the woody portion of the hemp plant's trunk, is a material that is often overlooked and considered a low-value by-product or waste of hemp cultivation.

Fig. 1. Hemp shiv size distribution [1].

The cardboard fibers are used to bind the hemp shiv into a stable material. Additionally, the new porosity formed in combination with the hemp shiv, due to the crosslinking of the cellulose present in both materials, increases the final insulation properties. Cardboard fibers are softwood fibers known for their higher resistance and longer length compared to hardwood fibers [32].

Nevertheless, since is a vegetable material a coating is needed to protect it from moisture. In this case, colophony or gum arabic. The coating of colophony was prepared using acetone in a 1:1 weight relation at 50 °C and gum arabic was dissolved in water in a ratio of 3:2 based in weight at 90 °C. Citric acid is employed to activate the hydroxyl groups (OH) present in the cellulose molecules; improving the durability of the samples. For more, details about the materials and fabrication method check previous publications [1].

3. Methodology

3.1. Fabrication method

The recycled cardboard fibers are produced by introducing cardboard in a pulper (5% consistency) to disintegrate the fibers at a temperature of 70 °C and a rotational speed of 1250 rpm for 60 min. Then the two materials are mixed (in water) in a proportion of 70% hemp shiv and 30% recycled cardboard fiber based in their dry weight. An electric mixer at a speed of 900 rpm for 3 min was used to prepare the material. If the addition of citric acid is required, the recommended ratio is 1 g of acid for every 20 g of the dry mixture [25]. The water used in this process can be reused for subsequent samples, promoting water conservation and minimizing resource consumption.

The method employed to remove excess water involves allowing it to drip through a mesh due to gravity. Subsequently, the sample is left exposed to the air for a period of 3 h, in this case, a test sample measuring 35 × 25x3 cm is produced using 300 g of dry material mixed in 10 L of water. Obtaining a theoretical dry density of 115 kg/m^3.

After forming the sample, it is placed in an oven to ensure thorough drying at 170 °C for 48 h to activate the citric acid reaction. To properly compare between sample all the samples were dried at 170 °C. Once the sample is completely dry, the coating (colophony or gum arabic) is applied to the surface using a brush. The coating is applied at a rate of 0.3 kg/m^2 to the two main surfaces leaving the 4 lateral surfaces without coating. This configuration is proposed in order to accelerate the reduction of the properties in the durability test. The two main degradation issues the material could face are the weakening of cellulose bonding, accelerated by significant internal moisture fluctuations, and fungal growth [1]. Since the coating reduces water diffusion and prevents fungal growth, leaving some surfaces uncoated amplifies these effects and accelerates the durability process. The sample is then left to cure for 48 h under ambient conditions. The sample is stored in laboratory conditions for the durability process, Fig. 2.

3.2. Recycling method

Samples were aged for 17 months. After this they were tested mechanically, before being subjected to a recycling process. The recycled materials were used as raw materials to remanufacture new samples.

Two different methods were used to recycle the samples. In the first method (RM, manual) the samples were soaked in water for 2 h and then mixed with an electrical mixer for 5 min. After this process, the remanufacture process remained as in the previous section, the only differences is the treatment of the raw material. In the second method (RP, pulper) the samples were disintegrated in the pulper at 1250 rpm for 60 min, using water at 70 °C, and then the raw material was processed as in the previous section.

In the samples remanufactured using the second method, it was observed that the previous coating, which was not removed, began to produce foam (Fig. 3). This foam was generated by the colophony and the gum arabic. The recycled fibers and shives were

Fig. 2. Durability samples storage in laboratory conditions (right) and recycled samples (left).

Fig. 3. Foam produced in the pulper by the recycled samples (left) and cleaned recycled raw material (right).

cleaned with water to eliminate the foam and resin from the previous coating, resulting in samples with no resin in the interior. In contrast, in the samples remanufactured using the first method, the energy introduced during mixing was lower, so in that case, the foam was not produced, and the previous coating remained present in the interior of the samples, affecting to the bonding of the fibers.

When the sample is remanufactured, a new coating is also applied, Fig. 2. The recycled material undergoes testing for thermal and acoustic insulation and mechanical properties to compare and validate the effectiveness of the recycling process.

3.3. Test method

3.3.1. Durability and recyclability test

The durability test conducted were perform to compare the degradation of the main properties of the insulation material. These durability test encompassed the evaluation of the material's acoustic and thermal insulation properties, as well as its mechanical response under compression, shear, and bending in aged samples (0, 3, 6, 9, 12 and 17 months). The test carried out are:

Acoustic Insulation

The acoustic properties were assessed using a two-microphone impedance tube, specifically the Brüel & Kjaer type 4206, in accordance with EN 10534 [33]. This standard test method involves the use of a tube, two microphones, and a digital frequency analysis system to measure the impedance and absorption of acoustical materials. The measurements were conducted within the frequency range of 100–6500 Hz. Cylindrical samples, with a radius of 1.5 cm and a thickness of 2 cm, were subjected to a plane sound wave, and the sound pressures were simultaneously measured at two microphone positions. By comparing the absorbed acoustic energy to the total incident energy, the normal incidence sound absorption coefficient (α) was determined.

Thermal Insulation

For assessing thermal insulation properties, a square test specimen measuring 10 cm in width and 3 cm in thickness is positioned within a thermal chamber. The objective is to measure the temperature difference between the two surfaces of the specimen using two K-type thermocouples as sensors, following the guidelines specified in the EN 12664 [34] standard in order to calculate the thermal conductivity (λ) which represents the ability of a material to conduct heat when a temperature gradient exists perpendicular to a unit cross-sectional area.

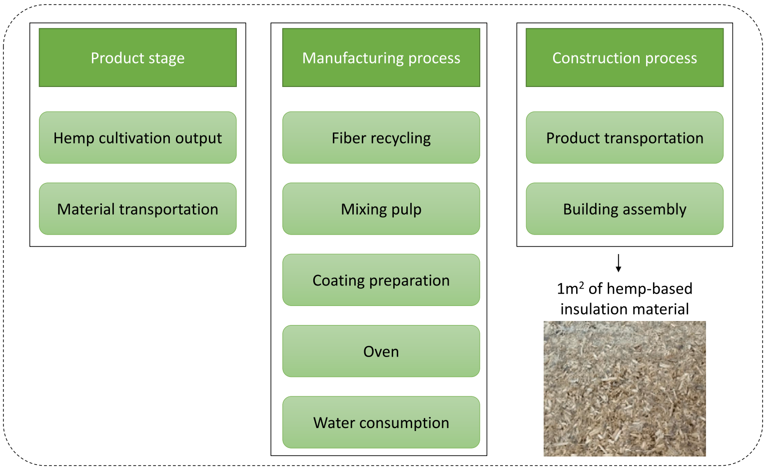

Fig. 4. System boundary for conducting the life cycle assessment of 1 m^2 of hemp-based insulation material.

Compression

For the compression test, prismatic specimens with dimensions of 100 × 100 × 30 mm were prepared. These specimens were subjected to compression testing at a constant test speed of 5 mm/min with an electromechanical universal testing system of 10 kN. The purpose of the test was to evaluate the material's resistance to compression and determine its ability to withstand the applied loads. The deformation of the specimens was monitored during the test, and the resistance of the material was assessed based on its ability to withstand strain up to 10% of its original dimensions, as specified by the EN 826 standard [35].

Shear

The shear test was performed using a static puncture test (CBR test). Cylindrical specimens with a radius of 10 cm and thickness of 3 cm were prepared for this test. The specimens were placed on a cylindrical support with a radius of 6 cm, and a puncture with a radius of 3 cm was used. The test procedure followed the regulations outlined in ISO 12236 [36]. The shear test was conducted using an electromechanical universal testing system of 10 kN, with a constant test speed of 5 mm/min.

Bending

The bending test was conducted using 250 × 50 × 30 mm samples. A 3-point test configuration was employed, with a distance of 200 mm between the supports. The span to depth ratio of the three point bend test was approximately seven, corresponding to an inter-laminar shear test geometry but the failure mode was thought to be typical of a three point bend test and it was also confirmed by testing a 900 × 400 × 50 mm sample. The test was performed at a constant speed of 5 mm/min using an electromechanical universal testing system of 10 kN following the EN 12089 standard [37].

3.3.2. Life cycle

In this section, the objective is to examine whether the use of the novel material aligns with a sustainable development model. To corroborate it, a life cycle analysis was performed. However, since the material is in a development phase. An industrial-scale manufacturing process has not been developed to correctly calculate its life cycle. In that study, a simplifications are assumed in order to estimate the performance of the composite material.

The methodology followed the guidelines that prescribe how to conduct a life cycle analysis of materials (ISO 14040:2006 [38] and ISO 14044:2006 [39]). In order to use more specific methodology, the life cycle analyze followed the guidance outlined in the PCR (Product Category Rules) for insulating materials made of foam plastics [40]. The analysis emphasized on examining until the material's use, nevertheless, the end-of-life phases and recycling have also been considered based on the research developed in this study.

The material being studied is the insulating panel made up of 70% hemp shiv, 30% recycled cardboard fibers and a coating of gum arabic. This material is fabricated under laboratory conditions. To simplify the analysis, it is important to note that resources related to the construction of the factory, waste generated from personal protective equipment (PPE) like gloves, packaging, have not been included in the assessment, since its contribution to CO_2 emissions is negligible compared to the electricity consumption of manufacture or transportation phase. In the case of secondary raw material like the coating it only have included its CO_2 emissions as input in the product stage. The secondary waste from the supply of raw materials has been negligible. In order to perform the analysis the PCR defines that the declared functional unit for coated insulating materials is 1 m^2 (considering 5 cm thick panel), Fig. 4.

Table 1
Nomenclature structure of the samples.

Coating	Crosslinking	Time
		M0 (0 month)
		M3 (3 month)
C (Colophony)	AC (Citric acid)	M6 (6 month)
GA (Gum arabic)	Ø	M9 (9 month)
		M12 (12 month)
		M17 (17 month)

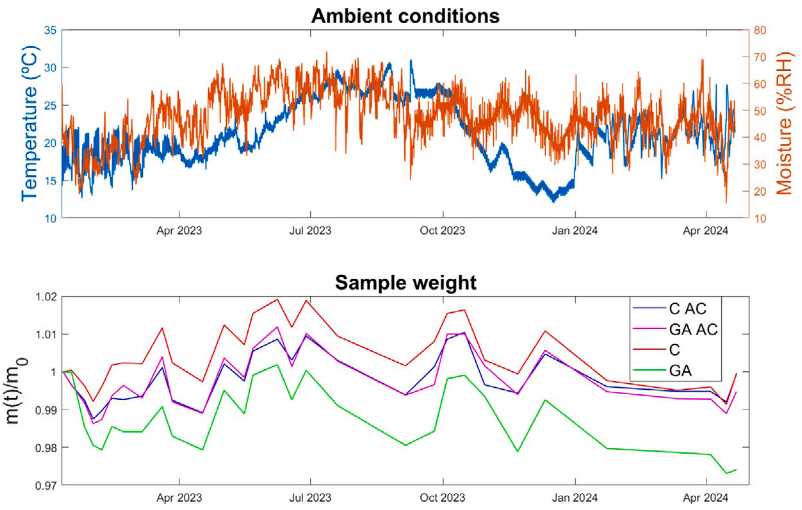

Fig. 5. Temperature and moisture in the laboratory (up) and sample weights across durability process (down).

4. Results and discussions

4.1. Durability test

The durability testing method involves subjecting the material to natural indoor cycles under ambient conditions. A range of test specimens were prepared and exposed to different duration of ambient conditions. These specimens were then tested to evaluate the potential property reduction. In Table 1, the 4 configurations of the samples are presented with the different durability span.

Prior to presenting the test outcomes, it is essential to outline the temperature and moisture conditions to which the samples were subjected and their impact on weight over time, Fig. 5. As depicted in the two graphs, the ambient humidity percentage is the primary factor influencing weight due to moisture absorption by the test specimens.

It can be observed that both types of coatings follow the same trend, although there is a difference in their initial weight. This is because gum arabic takes longer to eliminate the water used to produce the liquid resin than acetone in colophony. As a result, its initial weight is higher due to the additional water content it retains during the first two weeks. Since the figure is expressed relative to the initial weight of each sample, this difference reflects the initial conditions. However, as the results of the Moisture Buffer Value (MBV) test presented in previous studies [1], the variability of weight is slightly higher in the case of gum arabic. In a FTIR analysis conducted previously to the composite suggest that the acid does not enhance cellulose crosslinking but may offer a slight improvement in moisture resistance [1]. In other words, reduction over time may be less than in cases where citric acid was used. The durability test could also serve to corroborate that statement.

4.1.1. Acoustic insulation

From the results shown in Fig. 6, it can be seen that in both cases involving citric acid, the reduction across the different time steps tested (M0-M17) is smaller (0–0.2 dB/dB) compared to the cases without acid (0.2–0.4 dB/dB). In the acoustic case, the crosslinking of cellulose does not play a significant role, so this reduction corresponds to the reduction of hydrogen bonds in the cellulose molecule, as confirmed in the FTIR test [1]. In the case of commercial materials, rockwool and EPS the acoustic absorption obtained is 0.95–0.99 and 0.4–0.15 dB/dB respectively [1]. The absorption obtained in the insulating materials confirm its viability as a acoustic insulation.

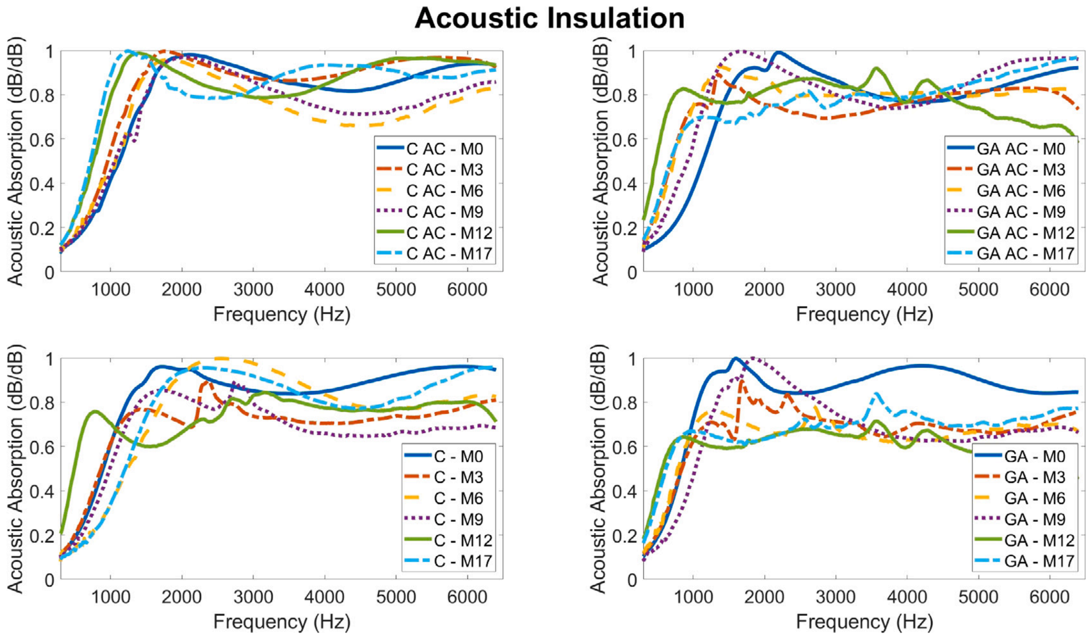

Fig. 6. Variation of acoustical insulation properties over 17 month of indoor ambient conditions for different samples: (a) colophony-citric acid (b) gum arabic-citric acid (c) colophony (d) gum arabic.

4.1.2. *Thermal insulation*

Based on the results presented in Fig. 7, similar to the acoustic case, cellulose crosslinking does not play a significant role. However, the ambient humidity level is a critical factor, capable of causing variations of up to 50% in the results. Examining Fig. 5, It is noticeable that humidity levels during months 6, 9, and 17 are higher compared to the initial months, resulting in a reduction in thermal properties. This is reflected by an increase in thermal conductivity, as shown in Fig. 7

When comparing the results between months 6/9/17 and months 0/3/12 for each case, we observe an increase in thermal conductivity ranging from 40%–45% in both cases, whether acid was used or not. This suggests that the addition of acid does not enhance thermal properties and the more influence factor for the thermal insulation properties are the internal moisture of the sample. In the case of 12 months the results are in the same range to 0 months leading to a not reduction of the properties in 1 year of life. Moreover fungi propagation did not appear in the material even at 17 months cases.

For commercial materials such as rock wool and EPS, thermal insulation works on the same principle. Their high porosity traps small air bubbles within the material, which are primarily responsible for the insulating properties. These materials achieve thermal conductivity values ranging from 0.35 to 0.37 for rock wool and 0.33 to 0.40 for EPS. Similar values are obtained in the green composite material due to its high porosity.

4.1.3. *Mechanical tests: compression, shear and bending*

Figs. 8, 9, and 10 illustrate the reduction of mechanical properties in the test samples caused by moisture cycles. Notably, the mechanical properties at month 0 are inferior to those at month 3. This discrepancy arises because the initial tests were conducted just three days after applying the coating. After the coating is applied, the sample increase its internal moisture and need a few weeks to stabilize, which can be illustrated by the weight measured in Fig. 5. This lingering moisture within the sample seems to have a more substantial impact on mechanical properties compared to insulating properties. Consequently, reduction have been compared with the properties at month 3 for each characteristic.

These figures confirm a reduction in the mechanical strength samples in the month with elevate moisture ambient (M6 & M9). Nevertheless, like in the thermal test, these properties are highly influenced by ambient humidity, when the levels of ambient moisture are lower the mechanical properties increase recovering its initial strength. It is worth noting that despite the reduction, these samples still maintain superior mechanical properties compared to EPS (0.5 MPa compression Young modulus, 30 kPa shear strength and 100 kPa bending strength [41] . In this application, the samples will already be in use, and there is no need for them to have a structural component as long as the material does not disintegrate. Furthermore, these results align with previous findings in mechanical properties and FTIR analysis [1]. It was confirmed that the inclusion of acid does not enhance the cross-linking of cellulose in the samples since it is not appreciable an increase in the maximum strength or a lower reduction in the durability process.

Taking into account that every sample have the same dry weight, the difference in grammage (mass/area) corresponds to a higher moisture content in the sample, which in turn reduces the mechanical properties. To compare the influence of moisture in

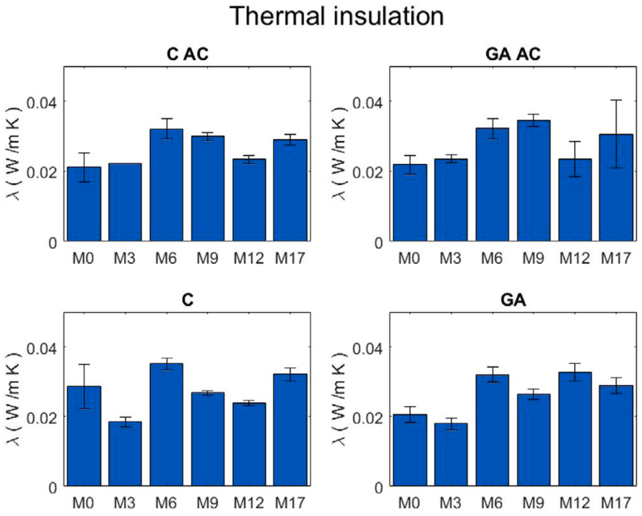

Fig. 7. Variation of thermal insulation properties over 17 month of indoor ambient conditions.

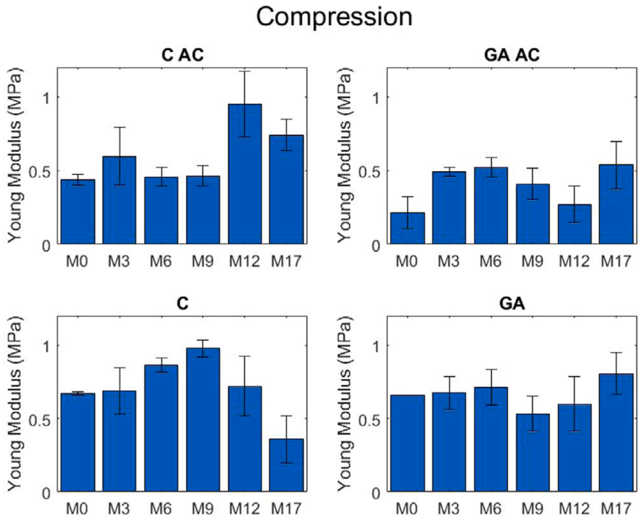

Fig. 8. Variation of compression elastic modulus over 17 month of indoor ambient conditions.

the samples, Fig. 11 displays the grammage of each sample (normalize to 3 cm of thickness) alongside their compression Young modulus. The figure illustrates that if the grammage increases, the mechanical properties of the samples decrease taking into account the comparative between samples of the same configuration.

However, there are some exceptions that can be explained by the manual manufacturing. In these cases, the increase in grammage is due to a higher content of hemp shiv and cardboard fiber, rather than moisture. Although the same amount was used for each panel, the test specimens were cut from a larger panel. In this larger panel, due to the imperfections of manual manufacturing, it is possible that the fibers were not distributed completely uniformly. As a result, some of the cuttings used for the tests may have slightly different grammages. In such cases, higher grammage actually increases the mechanical properties.

To validate that the durability process is accelerated by not coating all the sides of the sample it was proposed to conduct the mechanical test into samples (C-M0) that are full coated. The samples were introduced into a climatic chamber with different moisture (30–50%–70%) until the internal moisture was stabilized (1 week). For all the samples the internal moisture was measured with Sartorius moisture analyzer.

Comparing the results in Table 2, the variability of the compression Young modulus due to ambient moisture is lower than in the durability test samples. Notably, the internal moisture of the samples is lower than the ambient moisture, confirming the effectiveness of the coating. In terms of mechanical properties, the reduction in the compression Young modulus is less than 2% for samples exposed to 30%–70% moisture. These results are significant because the internal moisture variation is smaller than in the durability test samples, as the fully coated specimens provided effective protection against environmental conditions. The material will maintain its mechanical and insulation properties during the rainy months.

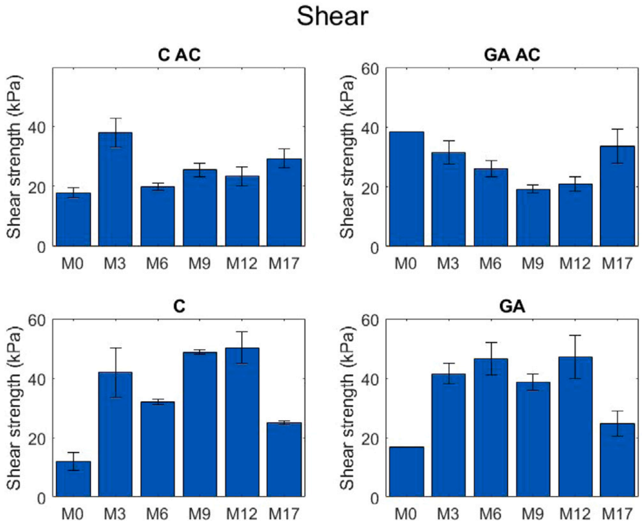

Fig. 9. Variation of shear strength over 17 month of indoor ambient conditions.

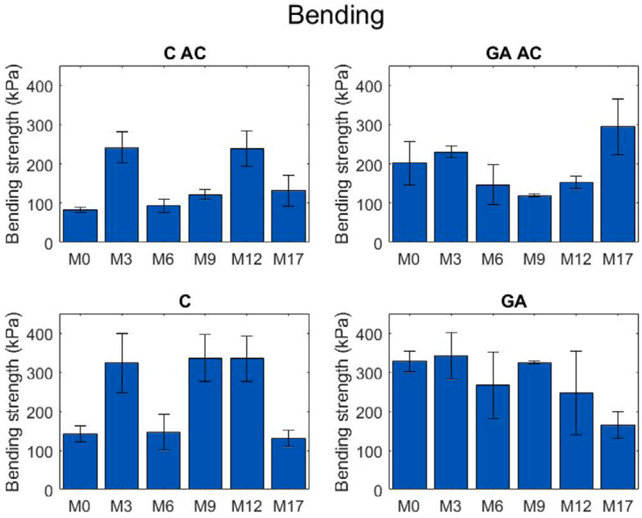

Fig. 10. Variation of bending strength over 17 month of indoor ambient conditions.

Table 2
Mechanical properties for different moisture content in C-M0 samples.

Moisture applied (%)	Internal moisture (%)	Compression Young modulus (kPA)
30	4.8	803.2 (35)
50	7.3	792.0 (37)
70	9.7	789.3 (47)

4.2. Recyclability test

For the two cases of recycled samples the acoustic and thermal insulation properties, Fig. 12 and Fig. 13a, are similar to the original durability samples (M17), validating the viability of recycling the composite materials for insulation applications. In Table 3, the different configuration of the recycled samples with the label are presented.

For the cases of recycled material with manual method (RM), the remanufactured process produces an unstable material, specially, in the cases with colophony, obtaining lower mechanical strength, Fig. 13. The samples with gum arabic could be disintegrated and mixed completely. In the case of colophony, the superficial layer could not be properly disintegrated because colophony is not soluble in water. As a result, small solid pieces of colophony, approximately 0.5 cm in length, were introduced

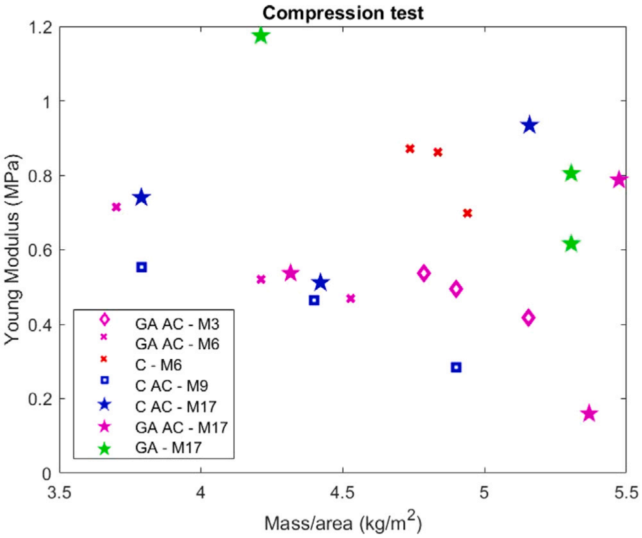

Fig. 11. Influence of moisture in compression test.

Table 3
Nomenclature structure of the recycled samples.

Coating	Crosslinking	Method
C (Colophony)	AC (Citric acid)	RM (manual)
GA (Gum arabic)	∅	RP (pulper)

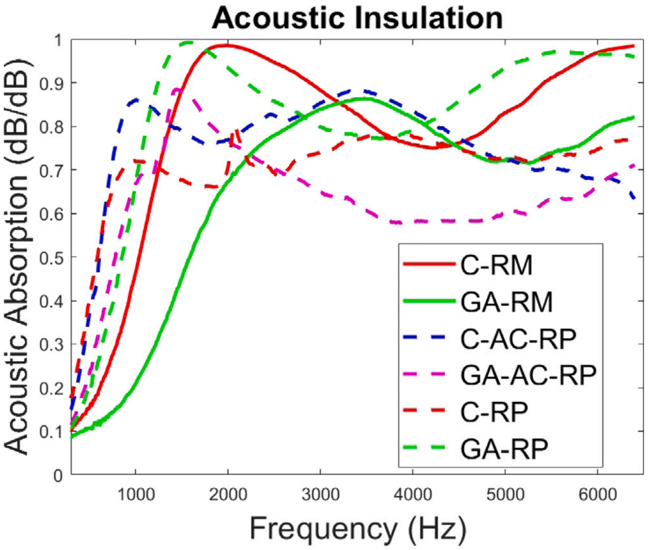

Fig. 12. Acoustic insulation properties of recycled samples.

into the mix. These pieces obstructed the binding of the fibers, leading to the production of a less stable material. In the RM method, the energy applied was insufficient to break all the fiber bonds, and the residual resin present in the raw material hindered the formation of new bonds between the hemp shiv and cardboard. Consequently, these samples exhibited instability due to the reduced number of bonds.

Nevertheless, the samples remanufactured with the pulper method (RP), obtained similar properties to the non-recycled cases, obtaining better results in the samples coating with colophony (acoustic: 20%, thermal: 5%, compression: 20%, shear: 5% and bending: 10%,). The resin is eliminated from the raw material and all the fiber are disintegrated which facilitate the crosslinking of all the fibers.

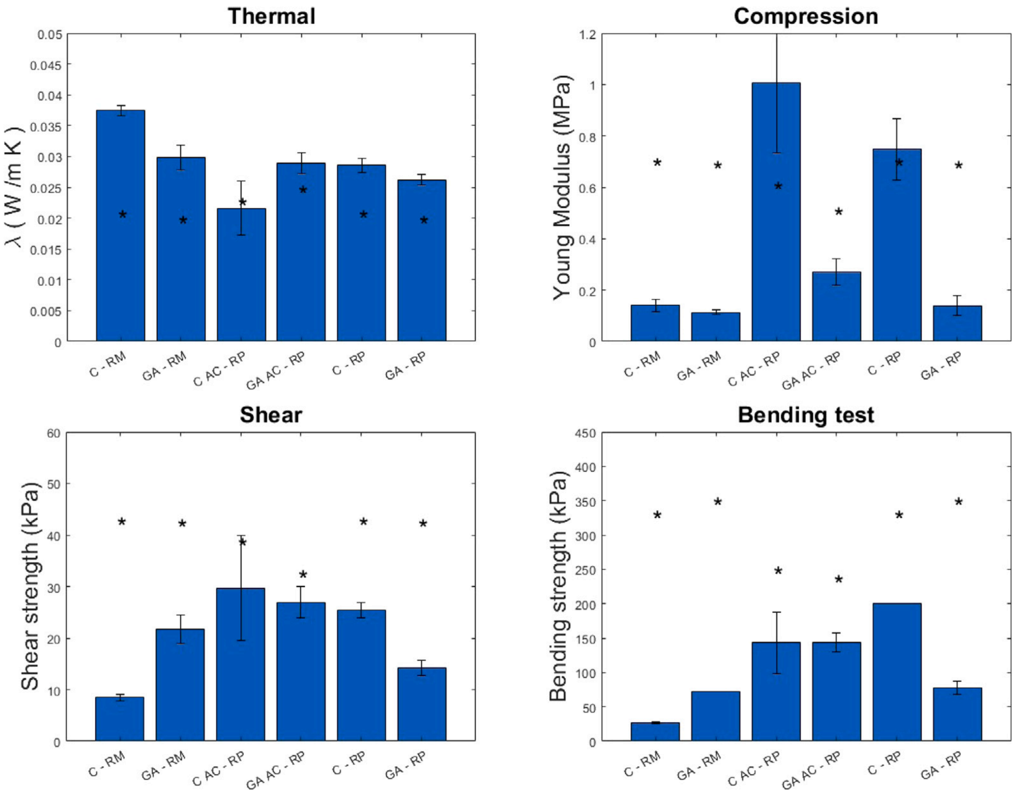

Fig. 13. Thermal insulation properties (up left), compression young modulus (up right), Shear strength (down right) and bending strength (down left) of recycled samples compared to the original samples (asterisk).

To corroborate it, the samples remanufactured with the RM method were evaluated by microscopy, it was conducted using an Olympus BH-2 optical microscope, which was equipped with a x4 objective. Since the colophony is not soluble in water, some fibers and shiv remained coated after the recycling process, which prevent them to produce new bonding. In the case of gum arabic, the resin present in the water start to produce bonding with the cardboard fiber, which resulted in a lower quantity of croslinking between hemp and cardboard, Fig. 14. Obtaining samples with lower mechanical properties. Therefore, the distribution of the fiber was not uniform producing areas where the materials present insufficient quantity of fiber to bond the hemp shiv. The unbounded block still have the initial internal porosity, therefore, the insulating properties were not affected and it only affect to the mechanical stability of the material. So based in the results of the RM method, the number of the bond in the samples could be reduced in every recycling process, limiting the number of reuses. A solution to this problem could be the use of new recycled cardboard fiber to decrease the hemp shiv-cardboard ratio, in order to increase the crosslinking of the samples.

The samples remanufactured with RP method, presented similar mechanical properties to the original samples. Nevertheless, it is worth to note that the samples also contains resin in the interior. The sample was cleaned but it could not be assured that all the resin was removed.

4.3. Life cycle

The life cycle analysis have been performed taking into account the considerations explained in the methodology, for a functional unit of 1 m^2 of the hemp insulation panel (5 cm thick). The recyclability of the material show good sight and the scenario that the material could be totally recycled by processing it as raw material in the pulper (RP method) could be considered. However, it still requires further studies to determinate the final end of life or the maximum number of recycled process that the material can withstand without affecting to its properties. Based on the results presented in this document, a conservative hypothesis will be considered, assuming that the material can be recycled once.

- Product stage
  Raw material supply. At this stage, emissions from the collection of raw materials and their transportation to the manufacturing site are considered, following the simplifications outlined in the methodology. The emission associated to the production and transport of the coating (gum arabic) are 0.45 kg of CO_2 [42]. On the other hand, cardboard fiber is a recycled and 0Km material, so in this case, the inputs and outputs are treated as zero, except for the treatment of the fiber in the pulper in the

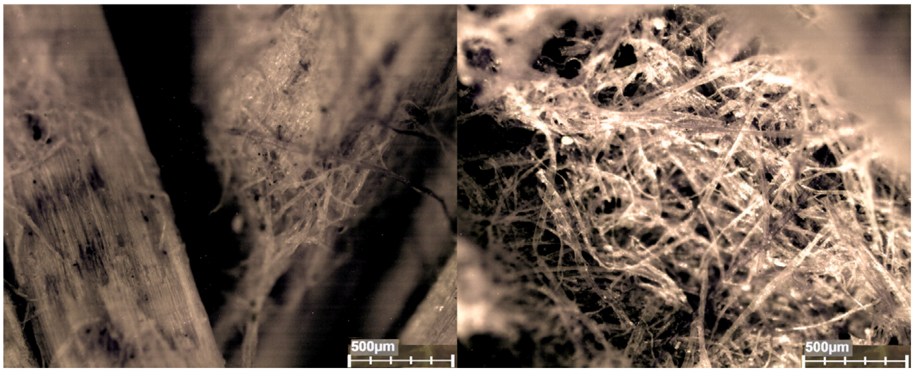

Fig. 14. Sample coated with colophony with low grade of bonding (left) and sample with a accumulation of cardboard fiber (right).

manufacturing process. Regarding hemp shiv, some inputs include water, fertilizer, and other products used in cultivation, while outputs include CO_2 emissions, chemical products, etc. Based in all the stages of the cultivation process and the CO_2 absorbed by the plant, producing 1 kg of hemp shiv results in the absorption of 0.315 kg CO_2 [43,44], which correspond to a 1.25 kg of CO_2 for a functional unit (4 kg of hemp shiv).

Manufacturing. In the manufacturing stage, the electricity consumption in the laboratory factory has been taken into account. This includes the disintegration of the recycled cardboard fiber, the mixing of raw materials, the preparation of resin, and the electricity consumed by the oven during the curing process. The pulper machine consumes 0.3 kWh, and it is required for 1 h to produce the material for one functional unit. The preparation of the coating consumes 0.1 kWh for 15 min. However, the oven is the most energy-consuming component, using 0.5 kWh for 48 h for one functional unit. In total, the energy consumption amounts to 24.3 kWh for manufacturing one functional unit. Assuming that the emission are 273 g $CO_{2\ eq}$/kWh [45]. This energy consumption is associated with 6.6 kg of equivalent CO_2. On the other hand, the production of the material requires the use of 100 liters of water. However, this water can be reused for each functional unit. Nevertheless, a 10% loss as been taken into account for evaporation in the oven, considering the losses of water during the reusing process, the need to change the water between several cycles, and the cleaning process of the machines. Moreover, to produce the gum arabic used for coating the functional unit, 0.8 L of water is needed. In that case, a consumption of 10.8 L of water is considered.

- Construction process stage

Transport from the gate to the site. In this stage, the fuel consumption of the transport truck to carry the panels from the factory. In this way, the amount of equivalent CO_2 for the functional unit of the panel is estimated, taking into account that, for simplification, other inputs such as worn tire rubber, vehicle maintenance oil, etc., are not considered. In the PCR document, the specific quantity of kilometers used to estimate CO_2 emissions is not specified. Therefore, for the purpose of this analysis, and given that the material promotes a local economy approach (km 0), a transportation distance of a maximum of 300 km have been assumed. Since the panels are a lightweight material, the limitation is the maximum volume. An average truck can carry 50 m^3, assuming a 90% of capacity, it can transport up to 2000 panels (1000 × 500 × 5 mm), obtaining a 3.000 kg payload. On average, a truck carrying 10,000 kg of payload is assumed to consume 40 liters of fuel per 100 km traveled and 30 liters without payload [46]. with the data provided, it will be use a consumption of 0.33 L/km with 3t of payload.

Based on this, the functional unit is calculated to be 6.5 kg of raw material. Therefore, for the functional unit, a fuel consumption of 0.15 liters is considered, resulting in the production of 1.18 kg of CO_2 equivalent emissions. Assuming an emission of 1.25 kg of CO_2 for 1 L of diesel [47].

Assembly. In this stage, the inputs and outputs are null because no machinery is required for the installation of the insulating panel, and it can be installed manually by the operator. Nevertheless, it should be noted that based on the durability test performed, applying the coating will be necessary if the material is cut, in order to completely cover all the surface.

Use stage. In this stage, it has been assumed that the material does not require any repairs. Given that it does not need energy or consume other materials to operate, the inputs and outputs for this stage are considered as zero.

Considering all the simplifications made in this analysis, the life cycle of a functional unit of 1 m^2 of the green insulating panels, it is necessary to consume 6.5 kg of raw material and 10.8 L of water, resulting in emissions of 6.98 kg of equivalent CO_2 (6,6 kg of equivalent CO_2 to manufacture it, mostly oven drying), Fig. 15. In the case of commercial materials, the rockwool, the commercial material most used in this application, produce an emission of 4.6 kg of CO_2 eq.[30] and 5 kg of CO_2 eq. in the case of EPS [31]. Even thought, in laboratory conditions, the CO_2 emissions are higher than commercial materials, the new composite material could have a considerable impact in the emissions of the building sector. Since rockwool is manufactured in ovens over 1500 °C. The analysis highlights that the curing stage in the oven is the phase with the highest emissions, accounting for 75% of the total emissions. This phase could potentially be improved in a large-scale industrial factory by implementing more efficient machinery, ultimately achieving a material with very low CO_2 emissions, obtaining a more sustainable material than the actual commercial materials.

Lastly, under optimal conditions, the material could potentially be reused as a raw material to produce other green insulating materials, extending the material's life cycle. Considering that the material could be recycled, in its second life the water consumption

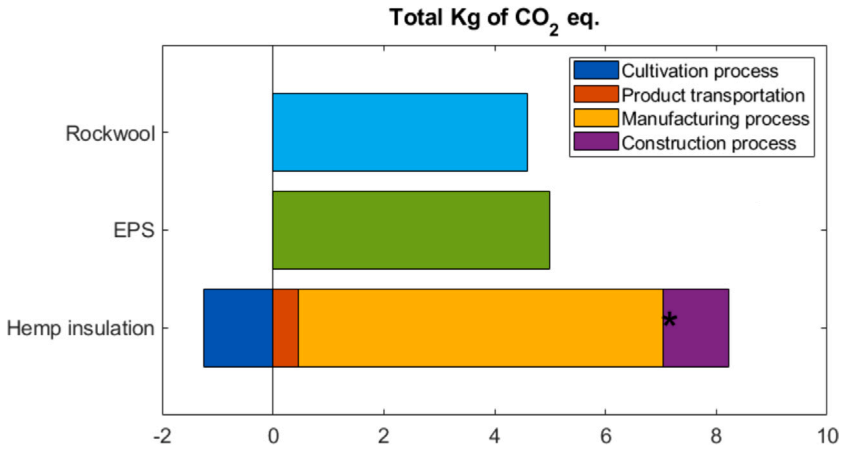

Fig. 15. Kg of CO_2 eq. emissions in every stage compared with commercial materials.

and CO_2 emissions will be the same since its produced by the manufacture and transportation, however, the consumption of the raw material is 0; obtaining a double life use. Nevertheless, it is necessary further research to determine how many times could the materials be recycled or a maximum number of years until its degradation.

5. Conclusions and further research interests

In this study, an experimental investigation was conducted to explore the durability, recyclability and the life cycle of an insulating bio-material based on hemp shiv, recycled cardboard fiber, two different vegetal coatings (colophony and gum arabic) and citric acid as a cellulose crosslinking agent. The results obtained are as follows:

- For each sample, the material did not show a degradation of properties after 17 months in ambient conditions.
  The experimental tests demonstrate that the inclusion of citric acid does not lead to a significant improvement in cellulose crosslinking between hemp shiv-recycled cardboard, only affects in cardboard-cardboard. Nevertheless the material can withstand a 17 months durability without presenting degradation in the properties. The results present a variation of the properties depending on the ambient conditions, however, it do not show a degradation of the properties. While citric acid enhances the bonding of the cardboard fiber, the key factor affecting its long-term properties is the prior moisture fluctuations. Thus, the most effective way to extend its lifespan is by applying a coating that protects the material from water diffusion. The samples coating by all the surface only present a reduction of 2% of its compression young modulus when the ambient moisture its raise from 30 to 70%.
- The 80%–100% of the composite material can be recycled, depending on the method and composition used.
  When it is introduced in water, the resin bonds the cardboard fiber producing a non-homogeneous material with a low grade of crosslinking, moreover, in the case of colophony it cannot be fully disintegrated. When the resin is removed hemp shiv and cardboard can be introduced in a pulper to produced new cellulose bonding which lead into a full recover of its original properties.
- A functional unit of the green insulating panel is associated with 6.98 kg of equivalent CO_2.
  The total carbon footprint of the panel is currently positive. However, with a more efficient curing process, emissions could potentially be reduced.
  The life cycle analysis conducted has been simplified to provide a basic technical characterization of the material in a laboratory manufacture. A more comprehensive study with more energy efficient industrial plant and the utilization of specialized software will be necessary for validation at each stage. However, this approach serves as a valuable method to validate the environmental advantages of replacing synthetic commercial materials with a green insulating panel made from hemp shiv. Moreover, if citric acid is not used, a manufacture process with a low temperature of sun dry curing could be performed in order to reduce the electricity consumption.

While the new bio-composite material, based on hemp shiv and eco-friendly binding materials, shows potential as a substitute for conventional inorganic insulating materials in building construction, further research is needed validate its performance in real building conditions and the final ecological assessment. Continued exploration and advancement of this material will be crucial for its successful application as a sustainable building material.

CRediT authorship contribution statement

Borja Martínez: Writing – original draft, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Ernest Bernat-Maso:** Writing – review & editing, Validation, Supervision, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Lluís Gil:** Writing – review & editing, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization.

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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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Data availability

Data will be made available on request.

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