UNIVERSITAT POLITÈCNICA DE CATALUNYA
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Hemp crop waste for green solutions in construction: physical and mechanical characterization

PhD. THESIS

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Solutions: Development of Hemp Stalk-Based Green Insulation Material," which is currently undergoing the review process.

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Abstract

The primary goal of this thesis is to unlock the potential of hemp stalk, an agricultural byproduct that currently lacks practical applications. The overarching aim is to promote the use of eco-friendly materials, reduce waste, emphasize recyclability, and embrace the principles of a circular economy, all while combating climate change by adopting more sustainable materials in lieu of synthetic ones.

To achieve these objectives, an extensive review of existing literature served as an initial step to explore the untapped potential of hemp stalk. This thorough exploration identified five possible applications: acoustic insulation materials, thermal insulation materials, particleboard, coffered ceiling, and parquet flooring. Subsequent research delved into identifying suitable plant-based binders and environmentally responsible manufacturing techniques.

Following this material-focused phase, an initial investigation was carried out by experimenting with various materials and manufacturing methods to ascertain which of the five applications held the most promise. The results were particularly encouraging for insulation materials, although coffered ceiling also displayed significant potential. The insulation materials utilize recycled cardboard as a binder and employ manufacturing methods with minimal energy consumption during mixing and drying, resulting in low carbon emissions and sustainable materials.

Once the material was selected, efforts were made to enhance its properties. This involved incorporating a plant-based coating (comprising arabic gum and colophony) to provide protection and introducing a cross-linking agent (citric acid) to bolster durability and material properties. These additions were meticulously assessed through a battery of tests related to thermal and acoustic insulation, mechanical characteristics, water resistance, fire resistance, and chemical analysis, such as FTIR spectroscopy.

To validate the material's performance, durability tests were conducted, exposing samples to humidity cycles over 0, 3, 6, and 9 months, alongside a scaled-down life cycle analysis. The end result was a material boasting superior thermal insulation properties compared to commercial alternatives, albeit with slightly lower acoustic insulation performance. Notably, citric acid was found to marginally enhance the material's durability. Furthermore, it was determined that different coatings could be selected depending on the application environment, thereby optimizing specific properties.

Resum

L'objectiu principal d'aquesta tesi és revelar el potencial dela canemuixa, un subproducte agrícola que actualment manca d'aplicacions pràctiques. L'objectiu general és promoure l'ús de materials respectuosos amb el medi ambient, reduir els residus, fer èmfasi en la capacitat de reciclatge i abraçar els principis d'una economia circular, tot mentre es combat el canvi climàtic mitjançant l'adopció de materials més sostenibles en lloc dels sintètics.

Per assolir aquests objectius, es va dur a terme una exhaustiva revisió de la literatura existent per explorar el potencial encara no aprofitat de la canemuixa. Es van identificar cinc possibles aplicacions: materials aïllants acústics i tèrmics, taulells aglomerats, cassetons i parquet. La investigació posterior es va centrar en identificar aglutinants d'origen vegetal adequats i tècniques de fabricació respectuoses amb el medi ambient.

Després, es va dur a terme una investigació inicial experimentant amb diversos materials i mètodes de fabricació per determinar quina de les cinc aplicacions tenia més potencial. Els resultats van ser especialment esperançadors pels materials aïllants, tot i que els cassetons també van mostrar un gran potencial. Els materials aïllants utilitzen cartró reciclat com a aglutinant i empren mètodes de fabricació amb un consum mínim d'energia durant la mescla i l'assecat, la qual cosa dóna lloc a baixes emissions de carboni i materials sostenibles.

Un cop seleccionat el material, es van fer esforços per millorar-ne les propietats. Això va implicar la incorporació d'un revestiment d'origen vegetal (goma aràbiga i colofònia) per proporcionar protecció i la introducció d'un agent de reticulació (àcid cítric) per reforçar la durabilitat i les propietats del material. Aquestes addicions van ser avaluades a través d'una sèrie de proves relacionades amb l'aïllament tèrmic i acústic, les característiques mecàniques, la resistència a l'aigua, la resistència al foc, i l'espectroscòpia FTIR.

Per validar el rendiment del material, es van dur a terme proves de durabilitat que van exposar mostres a cicles d'humitat durant 0, 3, 6 i 9 mesos, juntament amb un anàlisi del cicle de vida a petita escala. El resultat final va ser un material amb propietats d'aïllament tèrmic superiors a les alternatives comercials, tot i que amb un rendiment lleugerament inferior en l'aïllament acústic. Cabe destacar que l'àcid cítric millorava marginalment la durabilitat del material. A més, es va determinar que es podrien seleccionar diferents revestiments segons l'entorn d'aplicació, optimitzant així propietats específiques.

Resumen

El objetivo principal de esta tesis es revelar el potencial de la cañamiza, un subproducto agrícola que actualmente carece de aplicaciones prácticas. El objetivo general es promover el uso de materiales respetuosos con el medio ambiente, reducir los residuos, hacer hincapié en la capacidad de reciclaje y abrazar los principios de una economía circular, todo ello mientras se combate el cambio climático mediante la adopción de materiales más sostenibles en lugar de los sintéticos.

Para alcanzar estos objetivos, se llevó a cabo una exhaustiva revisión de la literatura para explorar el potencial aún no aprovechado de la cañamiza. Se identificaron cinco posibles aplicaciones: materiales aislantes acústicos y térmicos, tableros aglomerados, casetones y parquet. La investigación posterior se centró en identificar aglutinantes de origen vegetal adecuados y técnicas de fabricación respetuosas con el medio ambiente.

Tras esta fase centrada en el material, se llevó a cabo una investigación inicial experimentando con diversos materiales y métodos de fabricación para determinar cuál de las cinco aplicaciones tenía más potencial. Los resultados fueron especialmente alentadores para los materiales aislantes, aunque los encofrados también mostraron un gran potencial. Los materiales aislantes utilizan cartón reciclado como aglutinante y emplean métodos de fabricación con un consumo mínimo de energía durante la mezcla y el secado, lo que resulta en bajas emisiones de carbono y materiales sostenibles.

Una vez seleccionado el material, se realizaron esfuerzos para mejorar sus propiedades. Esto implicó la incorporación de un revestimiento de origen vegetal (goma arábiga y colofonia) para proporcionar protección e introducir un agente de reticulación (ácido cítrico) para reforzar la durabilidad y las propiedades del material. Estas adiciones fueron evaluadas a través de una batería de ensayos relacionados con el aislamiento térmico y acústico, las características mecánicas, la resistencia al agua, la resistencia al fuego y la espectroscopia FTIR.

Para validar el rendimiento del material, se llevaron a cabo pruebas de durabilidad que expusieron muestras a ciclos de humedad durante 0, 3, 6 y 9 meses, junto con un análisis del ciclo de vida a pequeña escala. El resultado final fue un material con propiedades de aislamiento térmico superiores a las alternativas comerciales, aunque con un rendimiento ligeramente inferior en el aislamiento acústico. Destacablemente, se descubrió que el ácido cítrico mejoraba marginalmente la durabilidad del material. Además, se determinó que se podrían seleccionar diferentes revestimientos según el entorno de aplicación, optimizando así propiedades específicas.

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Chapter 1

Introduction

1.1 The importance of hemp nowadays

Hemp (*cànnabis sativa L.*), a versatile and environmentally friendly plant, has been cultivated for thousands of years, primary for its fibers and seeds. Currently hemp is mainly produced for industrial or food purposes. Hemp is a multi-functional crop with diverse applications across several fields. The cultivation of hemp has played a crucial role throughout human history, providing numerous applications for each part of the plant.

Amidst this extensive range of applications, the immense potential lying within hemp stalks has been largely overlooked. At present, hemp stalks are merely considered waste by-products of hemp cultivation. In light of the urgent need to adopt eco-friendly practices and minimize waste, this research will focus on unlocking the hidden value of hemp stalks through reuse and recycling.

As the world faces an ever-increasing challenge of waste accumulation, hemp cultivation compounds this issue with its rapid growth and potential for up to four harvests annually, leading to a surplus of hemp stalks. Conventional disposal methods, such as landfills and incineration, only exacerbate pollution, climate change, and the depletion of precious natural resources. As responsible stewards of our planet, this research endeavors to seek alternative solutions, reducing our ecological footprint and embracing sustainable practices to make the most of this valuable resource.

Hemp stalks, a material rich in cellulose and lignin, present a unique opportunity for resource utilization. By harnessing this eco-friendly material, we open the door to a diverse array of sustainable products that can effectively replace less environmentally friendly alternatives. This, in turn, fosters a circular economy and significantly reduces our overall environmental impact. The extraordinary properties of hemp stalks make them ideal candidates for recycling initiatives, propelling us towards a greener and more responsible future.

The key properties of hemp stalks include:

- High insulating properties
- Low density
- Affordability
- Recyclable material
- Environmentally friendly material

During the 21st century, hemp stalks have garnered significant attention within the scientific community, leading to a notable rise in related publications, as depicted in Figure 1.1. This figure demonstrates the growing trend of scientific interest, showcasing the increased number of published articles. Nevertheless, this review extends its scope encompassing articles from various editors and taking into account factors such as the year of publication, methodology, and citation count for each article.

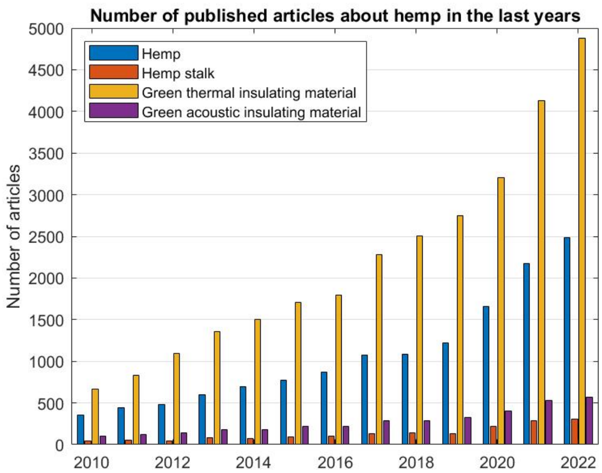


Figure 1.1: Number of articles published about hemp and green insulation materials

Energy consumption has shown a continuous upward trend, leading to a proportional surge in greenhouse gas emissions. Specifically, in the building sector, a substantial 45% of the total energy usage can be attributed to climate conditioning efforts [3]. Furthermore, in Europe, approximately 36-38% of all CO_2 emissions are associated with the building sector [4]. This trend is exacerbated each year as the changing climate provoke a higher energy consumption to maintain optimal indoor temperatures. Moreover, effective noise management is essential to create comfortable living conditions within buildings. High noise levels in residential environments can have negative effects, such as sleep disturbance and long-term health issues including psychiatric problem, cardiovascular disease and hypertension [5]. Consequently, there is a pressing need for studies aimed at enhancing the energy performance and improve the acoustic comfort of buildings. Additionally, in the coming years, there will be a substantial surge in demand for green buildings, defined as structures designed, constructed, or operated in a manner that minimizes negative environmental impacts while also creating positive ones [6]. Through this type of technologies it is possible to reduce the carbon footprint of human activities and to achieve the objectives set in the Paris agreements; to aim a “net-zero” emissions by 2050 [7].

A technique that can be explored in this field of research is the improvement of the performance of insulating materials based in green materials [8, 9] with a positive carbon footprint [10]. A biomaterial or eco-friendly material are materials has been designed, produced, and utilized in a manner that minimizes its negative impact on the environment throughout its entire lifecycle. Eco-friendly materials are characterized by their sustainable sourcing, minimal energy consumption during production, reduced emissions, and biodegradability or recyclability at the end of their useful life. These materials aim to conserve natural resources, mitigate pollution, and promote ecological balance while meeting specific functional or performance requirements. These materials have a reduced carbon footprint compared to traditional materials and reduce the amount of waste generated and decreasing the need for raw materials. The use of eco-friendly materials is an important step towards achieving a more sustainable future. The European Industrial Hemp Association (EIHA) states that the hemp industry is an economically viable and socially responsible enterprise, as it contributes to restoring ecological balance and achieving decarbonization goals for a promising sustainable economy [11].

Considering that it is a plant-based material, this research aims to add value to hemp stalk by incorporating it into construction applications where it can replace synthetic materials. This will help to revalorize hemp cultivation by increasing the value of a part that is currently considered a low value by-product. Consequently, it will reduce the use of synthetic materials that generate CO_2 during their production, and replacing them with a plant-based material.

To further enhance the value of green materials, the developed materials are intended to be recyclable. This way, once they reach the end of their useful life, they can be reused as raw materials, contributing to a circular economy that prolongs the lifespan of different materials. The properties of the hemp stalk make it a suitable material to manufacture more sustainable materials for the construction and urban furniture industry. Furthermore, by integrating local communities and industries into this process, we can create new economic opportunities and foster a greener, more resilient future.

Furthermore, in the literature, the utilization of hemp or other agricultural waste in combination with plant-based resins for various construction applications has been documented. Remarkably, no studies have been found that confirm the efficacy when paired with recycled cardboard or when employing manufacturing methods that do not necessitate the use of pressure.

In conclusion, the proposed study aligns seamlessly with the objectives of waste reduction, recycling, and sustainability. By unlocking the untapped potential of hemp stalks and utilizing their properties to create environmentally friendly green composite materials for construction, a promotion of the circular economy can also be achieved. This approach facilitates significant progress in conserving our planet’s valuable resources and alleviating the strain on the environment. Embracing this innovative endeavor collectively paves the way towards a greener and more responsible future.

1.2 Objectives

The main goal of this thesis is to produce a new a composite material made form hemp stalk and to the understanding of its various properties according aplicattions in construction and urban furniture. This aims to provide a solution for hemp stalk, a waste material, so that it can replace synthetic materials in specific applications where the full potential of hemp stalk can be utilized, thereby increasing sustainable materials.

To achieve this, it is necessary to study the different factors that influence the characteristics of the material. The study focuses on the following specifics objectives:

- OBJECTIVE 1. Develop a new green composite material using hemp stalk

  The primary objective of this research is to explore the potential of hemp stalk as a base material for developing a new green composite material. This involves conducting a thorough study of hemp stalk to understand its properties and characteristics. Additionally, various potential materials will be investigated to identify suitable binding matrices that can effectively combine with hemp stalk to create a stable and durable composite material.

- OBJECTIVE 2. Manufacture of the composite material and study of its properties

  Once the material study is complete, the focus shifts to the manufacturing process of the composite material. Different combinations of hemp stalk and binding matrices will be fabricated, and their properties will be thoroughly analyzed. Mechanical tests, insulation properties analysis, fire behavior studies, and durability assessments will be conducted to characterize the performance of each composite material. In order to select the most suitable combination that exhibits the desired properties for specific applications.

- OBJECTIVE 3. Applications of the composite material and solutions for construction

  This objective centers on studying the potential applications of the newly developed composite material in the field of construction. Different areas within construction will be explored to identify the most promising applications for the green composite material. Furthermore, studies on industrial-scale manufacturing processes will be carried out to assess the feasibility of large-scale production and its integration into construction practices. The overarching goal is to present viable, environmentally conscious remedies for construction through the utilization of green composite materials. To verify the ecological friendliness throughout all stages of the novel material, a comprehensive life cycle analysis will be undertaken.

The three objectives are interconnected and not sequential; instead, they complement each other in the research development process.

1.3 Methodology

This section will explain the methodology followed throughout the thesis, including the path taken and decisions made to achieve the established objectives. Figure 1.2 illustrates the flowchart of the study.

1.3.1 State of the art

Initially, the study will focus on conducting an extensive literature review aimed at identifying the key properties of hemp stalk. Furthermore, it will explore various construction industry applications where these characteristics can be effectively utilized, while also assessing the viability of green materials for these purposes. Consequently, different vegetal binders and their potential limitations in each application will also be studied. During this phase of the thesis, five applications have been identified for conducting preliminary studies.

1.3.2 Initial exploration of various material options and the selection of the most promising one

This thesis endeavors to unlock the potential of hemp-based materials for sustainable applications within the construction and urban furniture industries. The thesis centers on harnessing the distinctive qualities of hemp to create eco-friendly materials and explores five distinct applications. The primary step is to initiate a preliminary investigation to validate material properties and evaluate its feasibility in each application. This includes compatibility tests with chosen binders and fabrication methods. Following the identification of suitable green binders and fabrication techniques in alignment with the specific prerequisites outlined in the literature, a comprehensive material characterization is conducted to determine the key properties for each application.

In this characterization phase, the applications are categorized based on the chosen binder and fabrication method. The initial stage involves the selection of a mixing rule to evaluate the bonding potential between hemp stalk and the green binder, a combination that has received limited study thus far.

The study includes the creation of initial material samples, followed by manual agitation to assess their adhesion and stability. By progressively increasing the hemp stalk content in each sample, we can ascertain the maximum bonding capacity. Once this maximum bonding capacity is determined, test specimens are prepared for characterization tests, with a focus on identifying the most promising scenarios for the key properties relevant to each application.

To maintain a manageable scope, a decision-making process based on the characterization results is conducted based on the comparison with commercial materials, leading to the selection of one application for an in-depth study. If other promising materials are identified, future studies for those applications will be proposed.

1.3.3 Characterization and durability test of the selected material

Once the application is selected, a property enhancement process is undertaken, incorporating both mechanical and chemical methods. This optimization process involves conducting several tests to validate the results and determine the material's characteristics. Additionally, a long-term study of the material properties is conducted.

After the initial investigations, commercial-sized demonstrators will be manufactured to explore the feasibility of large-scale industrial production. An experimental investigation will then be undertaken to further optimize the material's properties, including applying a vegetable coating and enhancing cellulose crosslinking. This comprehensive study will involve various tests, including microscopy, FTIR-ATR analysis, and assessments of mechanical strength (compression, shear, and bending), fire resistance, moisture resistance, and water resistance. The specific characterization tests will depend on the selected application.

To ensure the long-term viability of the organic materials used, a durability test will be conducted to verify the material's performance over time. This step is essential to assess the material's stability and reliability throughout its lifespan. By addressing these aspects comprehensively, this thesis seeks to contribute valuable insights into the potential of hemp-based materials.

The material will be subjected to environmental cycles in a laboratory environment with precise monitoring of temperature and humidity conditions. This approach entails conducting periodic characterization tests to evaluate any possible degradation in the material's properties over time.

Throughout this thesis, durability cycles spanning 0-3-6-9 months have been conducted. Morever, the durability process will be extended to 12 and 17 months outside the thesis to corroborate the data.

1.3.4 Life cycle

In the preceding stages of the thesis, eco-friendly materials are employed to create a sustainable product. The purpose of conducting a life cycle analysis is to substantiate the eco-friendly attributes proposed in the thesis across all phases of the material's lifespan, confirming a reduced carbon footprint and more efficient utilization of resources through recycling.

The stages considered in this study encompass:

- Material and component acquisition
- Factory production
- Distribution and sale
- Usage
- Final disposal/Recycling

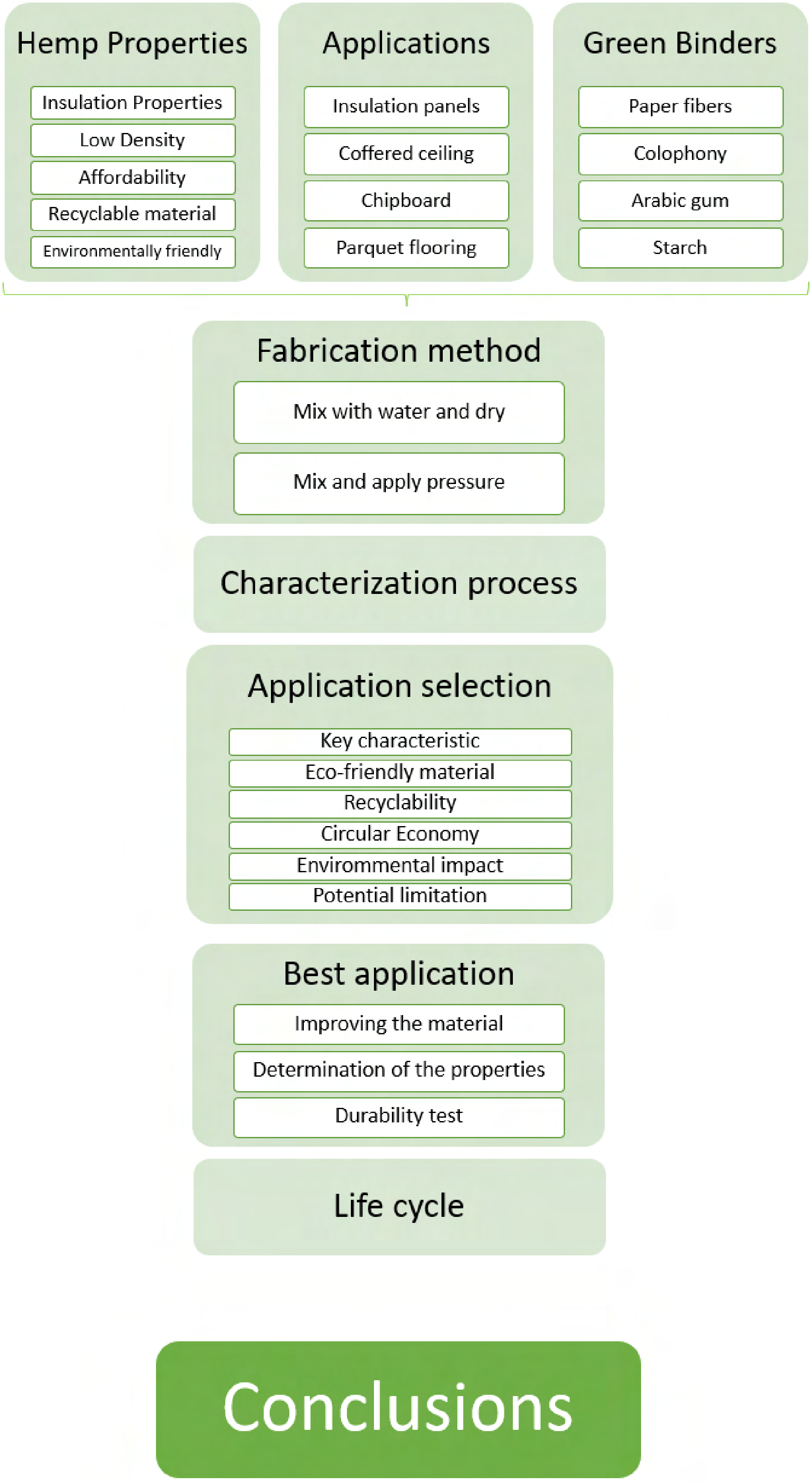


Figure 1.2: Flowchart

Chapter 2

State of the art

The following section provides an introductory overview of the historical background, cultivation practices, and diverse applications of hemp. The objective is to highlight the significant attributes of hemp that can be utilized in the production of an environmentally friendly composite material, specifically focusing on hemp stalk as a waste byproduct of hemp cultivation. This material holds the potential to contribute to a circular economy by ensuring recyclability.

To achieve this objective, a thorough examination of the properties of hemp stalk will be conducted, considering the specific requirements of various applications. Furthermore, the exploration will extend to manufacturing techniques and the optimization of plant-based composite materials, emphasizing their advantages over synthetic alternatives. The ultimate goal is to select the most suitable binders and fabrication methods for different applications, enabling the production of materials that align with specific requirements.

Furthermore, to achieve the development of an eco-friendly material, a life cycle analysis will be conducted. In this context, topics such as the material's carbon properties, durability, and recyclability will be reviewed in the state of the art.

2.1 History of hemp and its applications

Hemp is a botanical crop belonging to the *Cannabis sativa L.* family, primarily cultivated for industrial or food purposes. It encompasses various varieties, each cultivated according to its primary application.

Hemp has been cultivated since ancient times and is considered one of the oldest crops, with evidence of cultivation dating back 10,000 years [12]. It is a multifunctional crop with applications in various fields. While textile production has historically been the most significant application, the industrial era has seen an expansion of marketable parts and products derived from this crop [13]. Notable applications include the use of hemp fibers in the textile industry, hemp seeds and oils in the food industry, biomass fuel, construction materials, insulation materials for automotive textiles, paper manufacturing from cellulose, medicinal applications, and more.

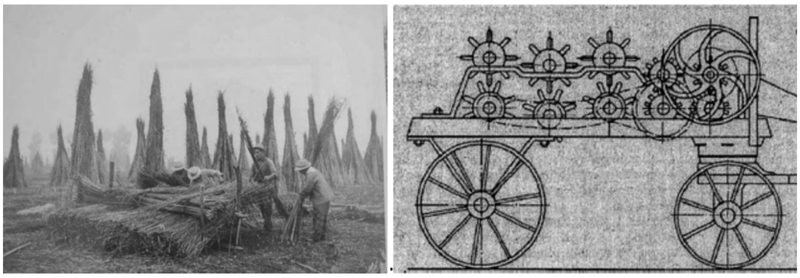


Figure 2.1: Hemp cultivation in the early decades of the 20th century (left), hemp fiber processor in 1861 (right) [13]

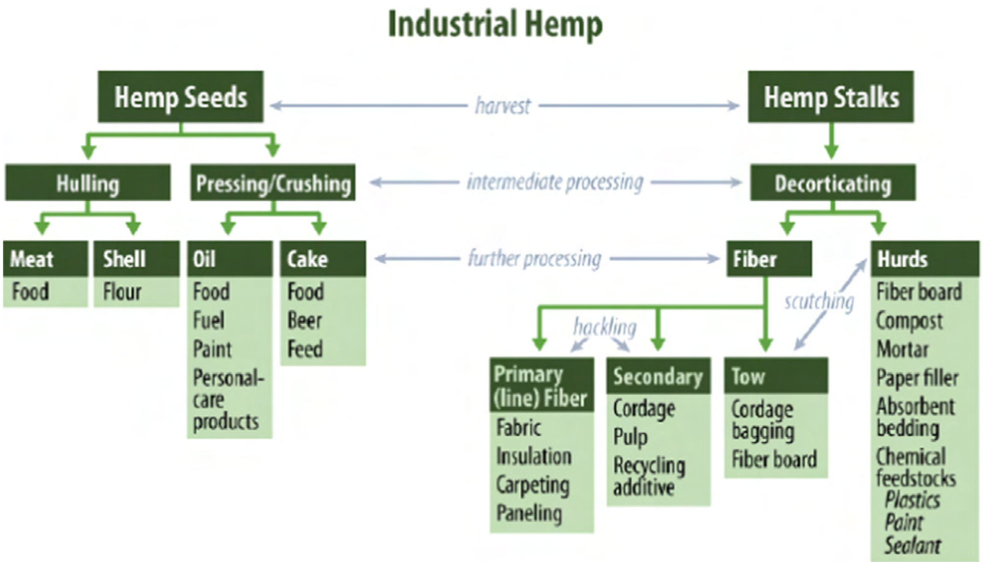


Figure 2.2: Flowchart of the use of hemp [14]

All these applications (Figure 2.2) have led to numerous studies on hemp aimed at increasing crop productivity and enhancing plant properties. Despite its long-standing cultivation tradition (Figure 2.1), ongoing research is crucial to optimize hemp cultivation due to the plant's numerous varieties [12]. Hemp is a fast-growing plant capable of producing up to four harvests per year. However, towards the end of the 20th century, hemp started to be replaced by synthetic fibers due to their superior properties and lower cost, resulting in a global decline in hemp cultivation [12].

In recent years, the remarkable versatility of hemp, improved material productivity, emerging applications resulting from research studies, and the need for more environmentally friendly materials have contributed to an increase in hemp cultivation. In Spain, for instance, data from the Ministry of Agriculture indicates that hemp cultivation expanded from 61 hectares in 2016 to 510 hectares in 2020 [15].

In addition to innovations in hemp production, there are other advancements related to less widespread applications. For example, hemp fibers are being utilized not only in textile manufacturing but also in construction due to their mechanical properties when used in the production of hemp fiber-based composite materials as substitutes for synthetic fibers [16, 17]. Hemp also shows significant potential in its use in building construction as a more sustainable and environmentally friendly insulating material that acts as a CO_2 storage, along with its extended life cycle resulting from recyclability, highlights its primary potentials [14]. In the field of bioenergy hemp can be used as a substitute for coal [18, 19] or to produce activated carbon derived from hemp crops that can be utilized as an electrode material in a hybrid supercapacitor in order to produce more environmentally friendly form of energy storage [20]. The reduction of CO_2 emissions can also involve the use of hemp, it can be utilized for its negative carbon footprint. Moreover, hemp biomass can also be processed into biochar, which has the ability to absorb various types of organic and inorganic pollutants in an environmentally friendly manner [21]. Currently, different parts of the plant have industrial uses (Figure 2.2):

- Vegetal fibers: textile industry
- Flowers: medicinal industry
- Seeds: food industry
- Leaves: medicinal industry
- Roots: medicinal industry

Nevertheless, it is important to note that the usage and production of natural fibers are influenced by the customs and traditions of specific countries and regions. For example, China and Bangladesh, both located in Asia, have a rich history of the use of natural fibers, particularly jute, sisal, and coconut. Conversely, France and Belgium are renowned for being major producers of flax, whereas North America, despite its significant economic impact, does not possess the same level of prominence in relation to natural fibers. West Africa, Latin American countries and India are the major oil palm cultivating countries [22, 23, 24, 25]. In the case of the hemp production, the hectares of farm are increasing every year. This trend is being reflected in Canada, USA, China and France being the countries with the highest hectares of hemp farms [26, 27, 28]. The industrial hemp production grew over 70% in Canada between 2011 and 2016 [18]. USA has increased its production from 9,000 hectares in 2016 to 93,000 in 2019 [29, 30]. Furthermore, the estimation of the industrial hemp market present gratifying results, the market size was estimated at $4.13b in 2021 and is expected to grow at CAGR, compound annual growth rate, of 16.8% from 2022 to 2030 [31].

The cultivation of hemp has played a crucial role throughout human history, providing numerous applications for each part of the plant. However, currently, the woody part of the stem called hemp stalk is a crop waste that is discarded or burned without adding any value, Figure 2.3. Despite hemp stalk is used in building applications or to bred animals, those applications do not cover all the offers of the material [19]. The hemp stalk which is localized in the inner part of the steam and it represents more than 50% by weight of the entire plant [32]. Despite not having any specific application that satisfies all the offer, it has interesting properties as insulating materials (0.049–0.082 W/m K thermal conductivity & 0.88–0.95 dB/dB acoustic absorption) [14, 33, 34].

In addition, it is a biomaterial, a materials that are produced and used in a way that minimizes harm to the environment, that contains cellulose and has woody fibers. Hemp fiber is one of the vegetable fibers that contain the highest percentage of cellulose (70-74%) [16, 35]. Although the hemp stalk has a lower amount of cellulose than the fibers, the percentage is still considerable, Table 2.1. These characteristics present an opportunity to explore new applications for hemp stalk, enhancing its value as a material and contributing to the cultivation of hemp for the production of more eco-friendly materials.

Figure 2.3: Sectioned stem of hemp consisting of plant fibers and hemp stalk [36] & hemp stalk

Table 2.1: Percentage of cellulose contained in hemp stalk

Cellulose (%)	**Hemicellulose (%)**	**Lignin (%)**	**Reference**
44	18	28	[37]
50-60	15-20	20-30	[38]
34-44	31-37	19-28	[39]
49	25	25	[40]

The objective is to revalue hemp stalk as the main material to develop a new biomaterial to use in green building, due to its insulating properties [14, 34, 41], negative carbon footprint, and its capability to produce a circular economy as a green material [42, 43, 44]. All these characteristics could be used to provide the stalk with new applications that add value to the material and, moreover, it will increase the value of hemp farming [45]. So, in this way, more ecological materials will be produced.

The main characteristics of hemp stalk that could make its use viable are its insulation properties and low density, which can be utilized in non-structural construction applications and urban furniture.

In order to obtain stable materials from hemp stalk particles, it is necessary to add a second material that acts as a binder for the different particles. There are numerous plant-based materials that can fulfill this function; however, in general, they have certain disadvantages compared to synthetic materials, such as higher raw material costs and lower mechanical properties.

Despite these drawbacks, different materials can be selected depending on the application, where high mechanical properties are not required, thus compensating for the extra cost of using more environmentally friendly materials, such as plant-based resins and binders, or other waste or recycled materials.

Based on these characteristics, the following applications are proposed:

- Acoustic insulating material
- Thermal insulating material
- Coffered ceiling
- Chipboard
- Parquet flooring

The following sections will involve an examination of diverse applications to determine their feasibility, exploration of potential plant-based binders, assessment of the current state of commercial materials, and the execution of supplementary evaluations. This methodology will aid in identifying the application that displays the greatest potential from among the options being considered.

2.2 Applications for low density solutions

In this section, thermal and acoustic insulation properties will be reviewed, with applications grouped based on similar properties and fabrication methods that result in low-density green composite materials.

2.2.1 Commercial material

Thermal/acoustic insulating materials encompass substances that inhibit the transfer of heat or sound. Within the context of construction, these insulating materials enhance energy efficiency and mitigate urban noise, thus creating a more comfortable environment.

Among the several types of insulation materials used in construction, the most commonly utilized ones are:

- Rockwool: A mineral wool with high thermal and acoustic insulation properties, renowned for its fire resistance, Figure 2.4.
- Expanded Polystyrene (EPS): A synthetic material known for its excellent thermal insulation properties, Figure 2.4.

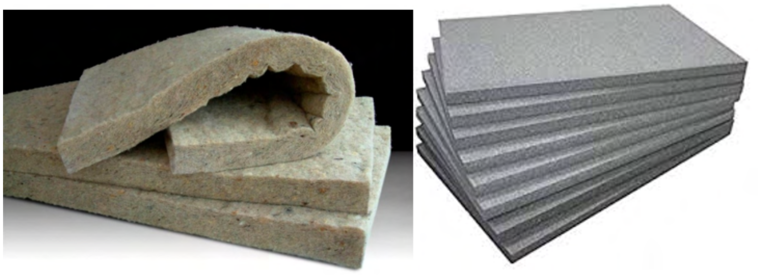


Figure 2.4: Rockwool (left) [46] & EPS (right) [47]

Both of these materials exhibit lightweight characteristics along with a notable level of porosity.

As part of the characterization process for the new green material, a comparative analysis will be conducted to assess its properties in relation to the aforementioned materials. The objective is to ensure that the new material is competitive and meets the requirements outlined by current regulations.

The different regulatory aspects to be considered for the characterization of the material are as follows:

- Thermal resistance: UNE EN-12664 [48]
- Acoustic resistance: ISO 10534 [49]
- Compression resistance: UNE EN-826 [50]
- Shear resistance: ISO 12236:2006 [51]
- Bending strength: UNE EN 12089 [52]
- Determination of short and long-term water absorption: ISO 29767:2019 [53]
- Fire resistance: UNE EN 13501 [54]

In addition to comparing the results with reference commercial materials, other regulatory aspects must be taken into account. In Europe in order to consider a material as thermal insulation, the thermal conductivity coefficient must be lower than 0.1 W/m K [55].

2.2.2 Insulating properties of hemp stalk

One of the most attractive properties of hemp are its insulating properties [56]. Moreover, in a comparison with conventional materials, natural fibers have similar hygrothermal properties and the process of retrofitting historical building envelopes is made more harmonious by their involvement [57].

Table 2.2: Properties of hemp stalk

Thermal conductivity $(W/m\ K)$	**Acoustic absorption** (α)	**Density** $\rho_{shives}(kg/m^3)$	**Porosity** $\phi_{inter}(\%)$	**Reference**
0.049-0.082	0.88-0.95	110-125	40	[33]
-	0.7-0.95	80-160	65-85	[58, 59]
0.064-0.115	0.88-0.99	97-120	-	[60]
0.051	-	72	70-80	[61]

According to the hemp stalk insulating properties, Table 2.2. Hemp stalk is a green material that can be use in insulating applications, even though its properties are less competitive than commercial materials. The thermal conductivity of rockwool is 0.036-0.037 (W/m k) (28%, better performance than hemp stalk) [62, 63] and EPS have 0.038 [63] (28%, better performance than hemp stalk). The acoustic absorption (α) of rockwool is 0.98-0.99 dB/dB [64, 65] (0.1 dB/dB, better performance than hemp stalk) and EPS 0.8-0.9 dB/dB [66] (same performance of hemp stalk).

The properties shown by the stalk are suitable for use as both thermal and acoustic insulating material, however it is a particle material, so it is necessary to use a green binder to manufacture a lightweight green composite material. In this insulating application, the mechanical resistance of the material is not a critical value, it is only necessary that it satisfy the minimum requirements and to be a stable material.

Hemp possesses exceptional insulating qualities, which are retained in the stalk. However, it is crucial to ensure its long-term stability as well as its chemical compatibility with the binders [67]. Since hemp is a particulate material, the sample's morphology and the internal porosity of the material is the critical factor that determines its performance [68].

2.3 Applications for medium density solutions

This section will proceed to conduct a comparable analysis for chipboard, coffer ceiling and parquet. The three applications will be categorized together based on the similarities in their manufacturing processes that result in medium-density green composite materials.

2.3.1 Commercial material

Particleboard

The particleboard, as shown in Figure 2.5, is a type of board manufactured using small wood chips. These chips are dried, mixed with resin glues, and then subjected to high-pressure pressing.

Figure 2.5: Particleboard [69]

There are various types of commercial particleboards available, each with modifications in particle size, particle orientation, and the type of synthetic resins used as binders.

The most frequently utilized resins in the industry are:

- Melamine resins
- Phenol-Formaldehyde (PF)
- Urea Formaldehyde-Melamine (MUF)
- Polymeric Diphenylmethane Diisocyanate (PMDI)
- Binary mixture of the above

Using binder proportions between 5% and 10% by weight, the manufacturing process involves applying a pressure of 8-13 MPa and curing the resin at the specified temperature for no more than 5 minutes [70].

For this case, a commercial particleboard will be used as a reference for comparing its properties with those obtained using green-based resins. Additionally, test specimens with non-green-based resins will also be fabricated to compare the manufacturing process conducted in the laboratory.

The characteristic properties taken as reference correspond to a P2-type particleboard (for indoor applications, including urban furniture, in dry environments) with the same thickness as the test specimens. In the existing literature, no research has been discovered that achieves a P2 performance using 100% green materials.

The different normative aspects that will be considered to characterize the P2-type particleboard are as follows:

- Tensile strength, UNE EN 319 [71] (0.4 N/mm^2 for commercial particleboard [72])
- Bending strength, UNE EN 310 [73] (11 N/mm^2 for commercial particleboard [72])
- Bending young modulus, UNE EN 310 [73] (1800 N/mm^2 for commercial particleboard [72])
- Industrial requirements for particleboards, EN 622 [74]

Coffered ceiling

The coffered ceiling, shown in Figure 2.6, serves as elements used in formwork to reduce the weight of constructions.

Figure 2.6: Coffered ceiling [75]

The purpose of this application is to replace non-reusable expanded polystyrene (EPS) coffers, which are commonly used to reduce the weight of constructions at a lower cost, but these coffers remain embedded in the structure and cannot be reused.

In this context, a primary limitation of green materials, as indicated by the literature, is the challenge of maintaining the stability of the hemp stalk and plant-based resin mixture under alkaline and highly humid conditions that occur during the concrete curing process. Furthermore, the material must exhibit adequate mechanical strength to endure the weight of the concrete during this curing phase.

To meet the construction standards for coffers and vaults, the material must exhibit a bending strength greater than 150 kPa and achieve a fire reaction rating of at least class E, which indicates the ability to withstand the attack of a small flame for a brief period without significant propagation of the flame [76]. Moreover, as a novel green material, it is crucial to ensure its stability under the specified environmental conditions.

The following properties will be tested for this application:

- Bending strength, UNE EN 12089 [52]
- Fire reaction, UNE EN 13501 [54]
- Static puncture test (CBR test), UNE EN 53974 [77]

In this application, the focus will be on utilizing plant-based resins, and their performance in the alkaline environment will be thoroughly evaluated. To ensure the material's durability and long-term functionality, a coating will be considered, if needed, to shield the composite from the impact of environmental conditions.

Parquet flooring

The parquet flooring, shown in Figure 2.7, consists of thin wooden pieces mechanically joined together to create a decorative pattern. To enhance its durability and scratch resistance, a protective surface layer is applied. The primary material used can be either natural wood or synthetic materials such as vinyl (PVC).

Figure 2.7: Parquet flooring [78]

This type of material involves using hemp stalk and plant-based resin for the interior part, providing the required mechanical strength, and combining it with a PVC coating to offer surface qualities, a visual pattern for the flooring, and interlocking between different strips.

In this approach, the compression and indentation resistance of the hemp-based material will be taken into account.

The different normative aspects that will be considered to characterize the material are as follows:

- Industrial requirements, EN 13489 [79]
- Hardness requirements, EN 1534 [80]

The patent by FLOORING TECHNOLOGIES LTD [81] showcases their manufacturing process, which includes applying pressure between 3 and 6 MPa and a temperature of 165°C for a brief duration, similar to the process used for particleboards.

According to this material specification, the resin and binder mixture must meet the hardness requirements, assessing the material's deformation when subjected to a constant load for a specific time frame. Furthermore, the final material must comply with all regulatory standards, ensuring there will be no slippage between the two layers of the material.

2.3.2 Mechanical properties of hemp stalk

The elastic modulus of the hemp stalk (10-16 GPa) depends on its position in the stem of the plant according to the height at which it is located [82, 83]. However, the area where the elastic modulus is greater is different among the species of hemp. The differences between the elastic modulus correspond to an increase or decrease in the size of the cell wall along the stem [82].

In these particular applications, the mechanical strength offered by plant-based resins might not meet the current regulations. Previous studies on materials incorporating hemp stalk with plant-based binders have demonstrated a maximum bending strength of 7 MPa using starch as a binder [84].

Consequently, the option of utilizing non-plant-based materials (up to 10%) is put forth, should the need arise. Furthermore, investigating structural strategies to augment the material's characteristics, like the creation of a sandwich-type composite, could be contemplated as an alternate avenue. Additionally, in the context of these three applications, the material must endure direct exposure to water, fire, or alkaline environments. Therefore, the application of an inorganic protective coating might be essential when employing hemp stalk for the proposed uses.

2.4 Green binder materials

The literature study in this section aims to explore various green materials that can serve as binders for the development of a green composite material based on hemp stalk. Recent publications have suggested that lignin-based resin, bio-epoxy resin [9], and recyclable cardboard fiber [2] hold promise as potential binding materials for creating a novel bio-composite material using hemp stalk.

This study focuses on green materials, however there are not many studies with a 100% green material, so some results of non-green binder will be presented to study the behavior of the material and find out which green materials could obtain the best performance.

Different applications are being studied to use hemp stalk, taking advantage of its low price, such as using it as insulation in buildings, adding lime to form non-structural blocks [85, 86]. A sustainable substitute for traditional walls, carried out a study of the acoustic absorption properties of lime and hemp stalk walls, obtaining an average of between 40-50% of acoustic absorption, obtaining better results with the less hydric binders, it can be manufactured on-site or placed prefabricated [87]. The main advantages of hempcrete is the insulating properties provide by the hemp stalk, and the lime binder provides a protection against moisture, fungi and fire [88]. In the same approach, adding hemp particles to the mortar as aggregates to reduce its density, increase the insulating properties and also the material will increase the capability of CO_2 storage, nevertheless, the mechanical properties decrease (maximum stress is reduced up to 30% when adding 8% hemp) [89, 90, 91]. Although cementitious matrices offer the benefits of affordability and adaptability, their use can result in chemical damage to hemp stalk [92]. In hot-dry regions where naturally ventilated buildings are preferred, the thermal design of walls based on compressed earth blocks (CEB) could be a viable and more ecological option [93]. Moreover, the thermal behavior of CEB was improved by 10% by incorporating a 0.5% of date palm waste [94]. While compacted earth is a compelling environmentally-friendly material, it is crucial to thoroughly examine its dependability and longevity in the absence of long fibers [95].

Hemp stalk particles, its also use as a raw material for materials that are made up of wood particles. In this way, a manufacture process is to mix it with a binder to fabricate a material similar to a chipboard. The manufacture of this type of material consists of mixing the stalk with the binder material and applying pressure and temperature in a mold, the adhesive cures and the material obtains the shape of the mold. The most commonly used binders are currently based on formaldehyde because of its mechanical properties, dynamic properties, abrasion resistance and affordability [96]. Nevertheless, due to the fact that it is a toxic material in large quantities, its use has been decreasing in order to reduce the formaldehyde. An intermediate solution is the use of 2 formaldehyde-based adhesives by partially substituting them for lignocellulose-based materials (wheat straw and pine and poplar particles) obtaining better results with PDMI (Polymeric Diphenylmethane Diisocyanate), by increasing the percentage of binding material [97, 98]. PDMI shows better binding properties than UF (Urea formaldehyde), curing at a temperature of 180°C and applying pressure for 3 minutes [99], these are the usual values in the industry. The process is also the same with vegetable agglomerate taking into account the curing temperature for each vegetable binder [100, 101]. In this type of manufacturing, the structure and size of the particles is also an important factor in the final properties of the material. If the particle size is very large, air gaps will be produced in the material as all the chips cannot be compacted together because the manufacture process do not use vacuum to prevent the air gaps. However, it can be solved by including saws and wood dust that occupy these holes together with the resin, thus increasing the mechanical properties of the material [102].

Another alternative is to completely eliminate formaldehyde-based resins by using natural ones. Although studies are being carried out to obtain vegetable resins that can achieve the regulatory requirements of the different applications, such as lignin-based wood adhesives [103, 104], obtaining a materials with great thermal properties, or vegetable proteins such as camellia protein, which is also a residue in the biodiesel production [105]. However, the main problem is the resistance to fire, that problem can be solved adding a fire resistance coating. There is also studies to develop a sustainable, high-performance, and flame-retardant wood coating based in a curing agent of ammonium hydrogen phytate (AHP) [106, 107]. Starch is also a good biobased binder for wood particles, for example, cassava starch binder can be use to elaborate a low density particleboard wit and excellent performance [108]. The fungi resistance is low, however it can be added citric acid to improve the fungal degradation in a 10% [109].

Different innovative renewable binders materials for thermal insulating application are studied. However, the technology still needs more research to have a competitive price and solve different technical difficulties to use materials like vacuum insulating panels, aerogels or nanocellulose. The researcher also proposes the use of recycled paper fiber as insulating material based on cellulose [110]. To bind the different hemp particles in a material that can be lightweight, a manufacture method is needed that does not require applying high pressure to provide the maximum porosity in the material, paper pulp fiber is proposed, witch also have a great thermal properties, cellulose paper waste have a thermal conductivity value of 0.046–0.054 W/m K [111]. Among the different paper fiber, cardboard is a great option due to the facility for recycling and the mechanical/binding properties. Cardboard fibers have a mechanical resistance 4 times greater than eucalyptus fibers (one of the most commonly used paper pulp fibers), measured from the binding [112, 113]. Moreover, the length of the fiber is longer (cardboard 2.7 mm in average length and eucalyptus 0.76 mm [114]). Several layers of corrugated cardboard were texted obtaining thermal conductivity values of 0.053 W/m K and a reduction of up to 80 dB in acoustic waves using several layers of corrugated cardboard [9]. In that case, the main problems are the durability and moisture/fungi/fire resistance.

Table 2.3: Advantages and disadvantages of bio-binder for stalk [9, 84, 103, 104, 105, 106, 108, 109, 110, 111, 112, 113, 114, 115, 116, 117]

Type of Bio-binder	Advantages	Disadvantages
Lignin based	Recycle the secondary products produced in paper pulping industries	Need to add a catalyst material
	Improve the modulus of elasticity	Increase the viscosity of adhesive
	Improve the thermal properties	Low fire resistance
	Improve the water resistance	Reduce the curing rate
	Good bonding strength	Low porous structure
		Low level of substitution
Starch based		Low stability upon time
		Low fungal resistance
	Good film formation property	Low fire resistance
	High level of substitution	Slow drying process
	Good bonding strength	Poor water resistance
		Need a surface treatment to increase the water resistance
Plant protein based	Improve thermal stability	Poor water resistance
	Good adhesion strength	Need a surface treatment to increase the water resistance
	High level of substitution	Low porous structure
		Low fire resistance
Paper pulp based	Recyclable	Poor fire resistance
	Good bonding strength	Slow drying process
	Improve thermal/acoustic properties	Need a surface treatment to increase the water resistance
	High porous structure	Low stability upon time
	High level of substitution	Low fungal resistance

Table 2.3 summarizes the comparison of performance, advantages, and disadvantages of bio-based wood adhesives for stalk. Regardless of the type of bio-based adhesive used, it is crucial to assess their potential for wood composite application and comprehend their interaction with wood. This evaluation can provide valuable scientific insights to guide the development of adhesives in terms of mechanical strength, water and moisture resistance, and thermal and acoustic properties.

2.4.1 Green binder for low density material

In this section, an analysis of the insulation parameters of composite materials will be conducted to enhance understanding of crucial characteristics for the mixture of hemp stalk and the green binder. Furthermore, the insulation properties of green materials with different binders will be presented and compared to assess their compatibility with binder characteristics. This evaluation aims to identify the most suitable binder for the applications studied in the thesis, with a specific focus on hemp stalk.

Acoustic insulation properties

When a surface is contacted by sound waves, the energy is distributed into three categories: incident, reflected and absorbed energy. In architectural acoustics design, it's helpful to utilize an average absorption coefficient that is assumed to rely solely on the physical attributes of the material. The sound absorption coefficient of any material is determined by the angle at which the sound wave hits the material and the frequency of the sound [118].

In the case of acoustic properties, the parameters of the hemp particles that influence the acoustic properties are the density of the particles, the bulk density, the thickness of the particles and the shape factor [58]; this relationship is also shown in others biomaterials. On the other hand, the results also show that for low-frequency waves, acoustic insulation is not very effective due to the porosity of the material.

Reducing the particle size and density of the stalk leads to enhanced acoustic properties, with optimal conditions achieved at a particle length of 6 mm and a density of 0.3 g/cm^3. This configuration significantly improves the acoustic absorption capabilities of the stalk material, Figure 2.8. Furthermore, the internal porosity can be predicted from the density of the particles [58, 59]. At a microscopic level, hemp has a structure that bring on the absorption of acoustic waves due to the voids provided by the porosity of the material [41]. Hemp has a porosity of 78% with pores between 0.9-3 μm [33, 119]. No studies have been found that prove whether the differences in cell size along the stem affect the insulating properties, detecting a research gap that could be interesting future research to increase hemp performance as raw material for insulating applications.

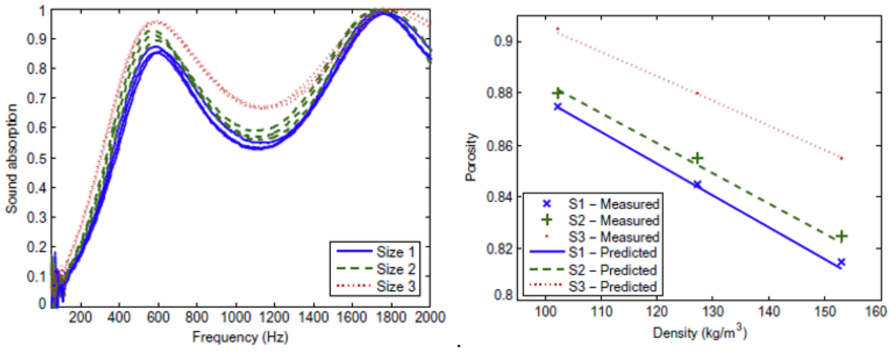


Figure 2.8: Acoustic absorption as a function of particle size, $size1 > size2 > size3$ (left) [58] and the relationship between density and porosity of hemp stalk (right) [59]

In the case of a composite material based in hemp stalk. The mechanism by which the materials absorb sound energy mainly involves 3 physical processes. Sound-absorbing composite materials have small holes that allow sound waves to access to their interior, causing gas flow and friction. It triggers the conversion of a portion of the sound energy into heat energy, which leads to sound absorption. In the case of hemp stalks, when sound waves hit them, the viscous effects between air cavities attenuate some of the sound energy, converting it into heat. Moreover, the sound-absorbing composite materials have the ability to absorb certain sound waves through their own vibrations. Due to the force between chain segments, unique hollow structure, and large specific surface of hemp stalks, sound energy is attenuated and converted into heat and mechanical energy during propagation, resulting in an effective sound-absorbing effect [41, 120, 121]. The internal porosity of the composite is a crucial factor to consider in the sound absorption mechanism of a low-density insulation panel. The presence of voids, inner and outer spaces, as well as the density and thickness of the composites directly influence sound absorption [122, 123]. Low-density materials with more open structures exhibit lower absorption at low frequencies, while denser structures show better performance at higher frequencies (above 2000 Hz) [118]. Several studies investigating sound absorption in porous materials have found a direct relationship between thickness and low frequency sound absorption. Increasing the thickness of the material leads to an increase in sound absorption at low frequencies. However, at higher frequencies, thickness has an insignificant effect on sound absorption [124]. Nevertheless, a sandwich-type material can increase acoustic absorption in low frequencies. The low porosity and high density of the material's surface cause sound waves to be reflected [68].

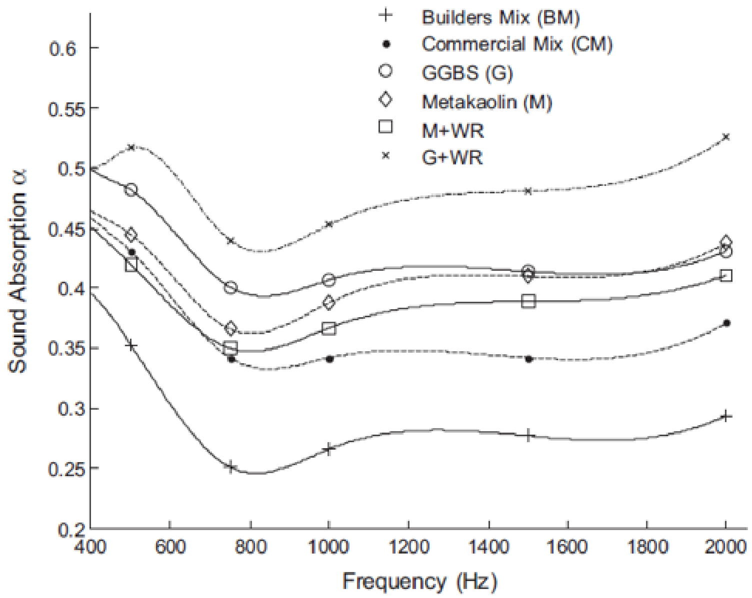


Figure 2.9: Sound absorption of hemp-lime samples with different binder [87]

The acoustic absorption values of composite materials based in hemp stalk, Figure 2.9, are lower than those presented by the hemp particles, in the Table 2.2, with means that the composition of the binder and the new internal microstructure is a more significant parameter than the internal porosity or the particle size of the hemp stalk.

In Table 2.4, is shown the acoustic insulation results obtained in different studies:

Table 2.4: Results of acoustic absorption of vegetal particles with different binders

Fiber	**Matrix**	**Eco-friendly material**	**Acoustic absorption (α)**	**Pore structure**	**Reference**
Hemp stalk	Polycaprolactone	NO	0.6-0.9	Hollow microstructure	[41]
Hemp stalk	Lime	NO	0.6-0.9	Porous material (70-75%)	[87, 59, 125]
Hemp stalk	Portland & MgO-cement	NO	0.1-0.25	Low porosity	[126]
Hemp stalk	C2-H	NO	0.6-0.8	Porous material	[127]
Hemp stalk	Wheat starch	YES	0.7	Porous material (88-90%)	[84, 128]
Sunflower stalk	Chitosan	YES	0.2	Low porosity	[129]
Sheep wool	Polypropylene	NO	0.3-0.6	Low porosity	[123]

Based on the results presented in Table 2.4, it can be observed that a highly porous material provides good acoustic insulation, as it contributes to the dissipation of sound waves, which is a more significant characteristic than sound wave reflection. Hemp stalk has been studied as an insulating material, but there are few studies on hemp due to the materials primarily used with inorganic binders that require the application of pressure and temperature to improve mechanical resistance, resulting in a denser material with low porosity that impairs its properties [130]. However, although a porous material performs better, a non-porous surface coating can be added to the sample to improve sound wave reflection. The use of certain binders can result in an elastic behavior of the composite material, allowing acoustical vibrations to be transmitted to the solid matrix. These elastic effects can have a significant impact on acoustical performance, causing both global effects on transmission and local effects on absorption [127].

Thermal insulation properties

The main objective of thermal insulation is to enhance energy efficiency by limiting the transfer of heat through the building envelope. Insulation materials are designed to conduct heat poorly in order to minimize heat loss [55]. Heat conduction occurs due to the interaction between particles in a substance (solid, liquid or gas) that results from particle movement. Hence, heat moves from more energetic particles to less energetic ones. Convection, on the other hand, refers to heat transfer between a solid surface and a fluid in motion, whereby heat is transferred through a combination of conduction from the solid to the fluid and bulk movement of fluid particles. Thermal insulation offers high thermal resistance, thereby retarding heat flow, primarily due to gases entrapped within the porous material structure [131]. Thermal insulation materials typically have low densities, which translates to high porosity. The insulation effect is largely attributed to the low thermal conductivity of still gases trapped within the voids of porous material [131]. The principal factors that affect thermal conductivity include temperature, porosity, moisture content, and density. Other factors are airflow velocity and thickness [55]. For cellulose-based insulating materials, factors such as temperature, moisture content, and mass density are critical in determining the thermal conductivity value [57, 110]. So in this case, the environmental conditions (temperature, humidity) affect its insulation capacity.

In addition to acoustic insulation, the hemp stalk also has high thermal insulation properties, obtaining a coefficient of thermal conductivity of 0.049-0.115 W/m K, Table 2.2. In Europe, materials with a λ value lower 0.1 W/m K may be classed as thermal insulating . Additionally, materials with thermal conductivity values lower 0.03 W/m K are deemed highly effective as thermal insulators [55].

There are studies on that topic to develop new biomaterials using starch as a binder [132]. Straw and a geopolymer can be used to form an insulating material, obtaining thermal conductivity values of 0.101 W/m K [133, 134], the biggest drawback being resistance to water and fire when using green materials. Other studies show materials made with corn particles and epoxy resins obtaining sufficient thermal conductivity values to consider it an insulating material [68].

The thermal insulating performance of materials is improved by higher porosity and bulk density, witch means a material with a large number of small pores. Unlike acoustic properties, the thermal properties of the matrix material also play a crucial role in determining the final properties of the composite material. Greater air bubbles promote more effective heat transfer within the material. As density rises, the air bubbles shrink, and the structural framework becomes more complex. Smaller bubbles lead to reduced heat transfer, and the intricate solid matrix system exhibits higher thermal resistance. Increased density results in a higher solid content, making the thermal conductivity of solid components more significant [55].

At a mean temperature of 24°C, the apparent thermal conductivity of hemp stalk was tested for various densities and found to increase with rising temperature. While wood-based fiberboards are utilized as thermal insulation materials due to their low density and high thermal resistance, their porous internal structures make them sensitive to environmental changes [135]. Consequently, their thermal conductivity increases by roughly 50% as the temperature increases from 10 to 60°C [136].

In Table 2.5, is shown the results of thermal insulation obtained in different studies:

Table 2.5: Results of thermal absorption of vegetal particles with different binders

Fiber	**Matrix**	**Eco-friendly material**	**Thermal conductivity (W/m K)**	**Reference**
Hemp stalk	Lime	NO	0.08-0.13	[87, 137, 138]
Hemp stalk	Portland & MgO-cement	NO	0.08-0.115	[126, 139, 125]
Hemp stalk	Wheat starch	YES	0.06-0.07	[84, 128]
Hemp stalk	Cassava starch	YES	0.026	[140]
Hemp stalk	Reactive vegetable protein	YES	0.078	[141]
Sunflower stalk	Chitosan	YES	0.056-0.058	[129]
Corn stalk	Rice huck ashes	YES	0.06-0.08	[132]
Bamboo powder	Bio-glues	YES	0.10-0.20	[142]
Corn stalk	Epoxy	NO	0.10	[68]
Sheep wool	Polypropylene	NO	0.06-0.10	[123]
Flax stalk	Lignin & and biobased epoxy	NO	0.074	[104]

Elevated relative moisture in materials can result in reduced thermal insulation properties and an increased susceptibility to mold formation. Studies have shown that as material moisture increases from 0% to 10%, thermal conductivity also increases [143]. In the case of composites made from jute, flax, hemp stalks, and fibers, a relative air humidity of 70% leads to a material humidity level of 5-10%. Therefore, it is essential to protect the material from ambient conditions and stabilize it to achieve an optimal thermal conductivity.

In addition to using vegetable resins, it is also proposed to replace the particles of pine and other common trees with crop waste such as hemp stalk. The influence of the starch-stalk ratio on the properties of the material is studied, it shows that by increasing the hemp particle ratio, the mechanical properties are reduced due to the increase in porosity, which decreases the load transfer capacity. Nevertheless, it improve the thermal [128].

Selected binder for low density solution

To create a lightweight material by binding various hemp particles, a bonding method is sought that preserves maximum porosity, which mean that a manufacture method that not requires high-pressure conditions. As a result, the use of different vegetable resins are excluded.

Numerous previous works have demonstrated the viability of insulating biomaterials employing the mentioned components. Moreover, several manufacturing methods and materials have been proposed, advocating the use of recycled paper and cardboard, primarily based on cellulose, as insulating materials [110]. This approach yields impressive thermal conductivity values of 0.053 W/m K and reduces acoustic waves by up to 80 dB using multiple layers of corrugated cardboard [9] (see Figure 2.10).

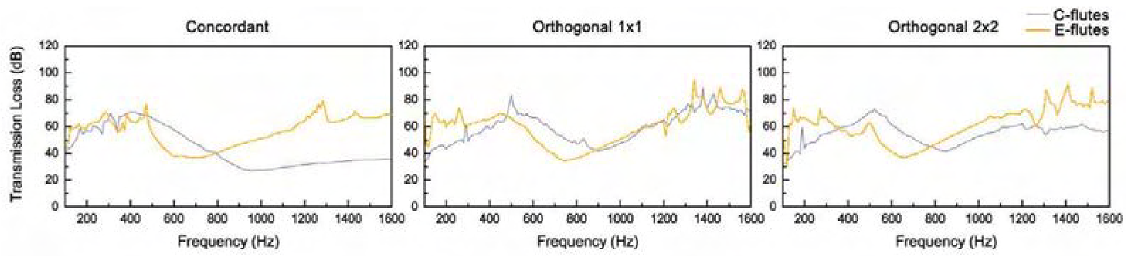


Figure 2.10: Sound transmission loss through corrugated cardboard panels [9]

The primary concern when using cardboard as a binder is its limited water stability. To address this, surface treatments utilizing vegetal materials are proposed [144]. In this research, bamboo particles undergo a surface coating with colophony (pine resin), resulting in a transformation from hydrophilic to hydrophobic behavior after treatment, along with increased environmental stability. Moreover, arabic gum exhibits promising outcomes as a coating for humid conditions [145]. However, its direct contact with water is not suitable due to its solubility. This implies that the protective layer would dissolve, leaving the material without adequate coating.

The bonding process between hemp and cardboard fibers would involve cross-linking the cellulose molecules present in both materials by mixing them with water. Notably, hemp contains a high cellulose content, as depicted in Table 2.6.

Table 2.6: Chemical composition of different plant fibers [16]

Fiber	Origin	Cellulose (%)	Lignin (%)	Hemicellulose (%)	Pectin (%)	Wax (%)	Ash (%)
Hemp	Bast	70-74	3.5-5.7	15-20	0.8	1.2-6.2	0.8
Jute	Bast	61-72	12-13	18-22	0.2	0.5	0.5-2
Sisal	Leaf	78	8	10	-	2	1
Flax	Bast	64-72	2-2.2	18-20	1.8-2.3	-	-
Ramie	Bast	69-91	0.4-0.7	5-15	1.9	-	-
Harakeke	Leaf	56-64	7.8	23-31	-	-	-
Coconut coir	Fruit	36-43	0.15-0.25	41-45	3-4	-	-
Kenaf	Bast	45-57	22	8-13	0.6	0.8	2-5

2.4.2 Green binder for medium density material

Given the characteristics of these applications, an appropriate manufacturing method resembling particleboard production seems suitable, given its resemblance to the constituent wood particles.

In Table 2.7 is shown the compresion young modulus of vegetal particles with differents binders:

Table 2.7: Compression young modulus compared with other stalk based materials

Fiber	Matrix	Eco-friendly material	Compression Young Modulus (MPa)	Reference
Hemp stalk	Wheat starch	YES	0.1-0.15	[84, 128]
Hemp stalk	Reactive vegetable protein	YES	1.1-3.0	[141]
Corn stalk	Epoxy	NO	0.11-0.29	[68, 100]
Hemp stalk	Porlant cement & MgO-cement	NO	0.4-2.1	[89]
Hemp stalk	Lime	NO	0.3-0.5	[85]

In the event that outcomes using plant-based binders fail to satisfy regulatory standards [115], an alternative approach is suggested: a sandwich-type solution featuring a core composed entirely of plant-based material, coupled with an external layer crafted from synthetic resin or PVC. This external layer will not only enhance the material's structural integrity but also shield the plant-based core from environmental factors like water or fire. If deemed necessary, the proposed approach entails optimizing the organic core of the material and adapting the outer layer to ensure compliance with the regulatory requisites of the intended applications.

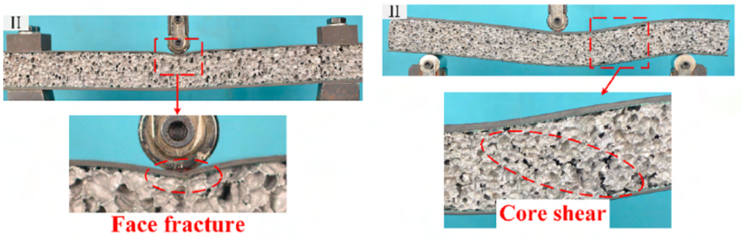


Figure 2.11: Change of failure mode in 2 sandwich-type specimens with different boundary conditions [146]

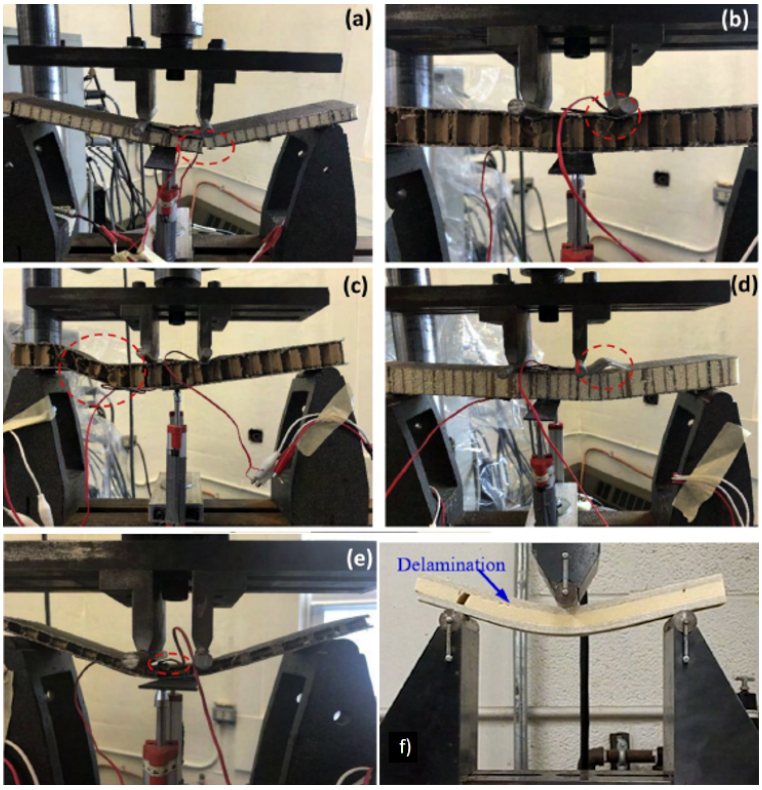


Figure 2.12: Diverse failure modes resulting from cross-section modification: (a) tensile rupture (b) indentation (c) core shear (d) wrinkling (e) dimpling [147] (f) delamination [148]

Nonetheless, in the context of the three proposed applications, it is essential to consider how the outer layer impacts the material's failure mode. This is because if the observed failure mode differs from the expected one, the mechanical properties could be compromised. Therefore, in addition to assessing regulatory requirements, an examination of the material's failure mode would be indispensable to ensure that the sandwich-type meets the applications requirements. The Figures 2.11 & 2.12, illustrates the different failure modes depending in boundary conditions, initial imperfection of the material and the crosssection material relation.

2.5 Life cycle

The purpose of conducting a life cycle analysis is to substantiate the eco-friendly attributes proposed in the thesis across all phases of the material's lifespan, confirming a reduced carbon footprint and more efficient utilization of resources through recycling.

The stages considered in this study encompass:

- Material and component acquisition
- Factory production
- Distribution and sale
- Usage
- Final disposal/Recycling

The subsequent guidelines prescribe how to conduct a life cycle analysis of materials:

- Environmental management - Life cycle assessment - Principles and framework: ISO 14040:2006 [149]
- Environmental management - Life cycle assessment - Requirements and guidelines Amendment 2: ISO 14044:2006 [150]

For the proposed material, the emphasis will lie on utilizing secondary materials with a minimal carbon footprint. The aim is to guarantee that the material's carbon footprint contributes to a reduction in emissions generated during all the life phase. Furthermore, it is essential that the material is recyclable and does not generate harmful chemical byproducts that could adversely affect the environment.

To conduct a life cycle analysis, properties such as carbon storage and the durability of green materials will be reviewed.

2.5.1 Carbon storage properties of hemp stalk

Developing a biomaterial based on hemp stalk can increase the value of the hemp crop, produce more environmentally friendly materials, and also result in materials that act as CO_2 accumulators [6].

The hemp absorbs CO_2 meanwhile it is growing by the photosynthesis process. Moreover, due to very rapid growing, the biomass accumulated by hemp crop has significant effect in absorbing atmospheric CO_2, however some of the carbon stored in this biomass is turned back to atmosphere as CO_2 due to biodegradation of leaves and roots. Assuming a concentration of 0.5 kg of carbon per kg of dry matter, it can be calculated that 1.84 kg of CO_2 are sequestered per kg of dry hemp through photosynthesis during the plant's growth.

This means that a ton of dry hemp can store 325 kg of CO_2, taking into account the amount of emissions during the farming as the diesel consumption, transportation of the seeds, etc [151].

An example of a CO_2 accumulator is hempcrete, a sustainable building material made from hemp stalks, lime, and water. Lime in the material also contributes to CO_2 sequestration due to the carbonation process, but the overall process has a positive carbon footprint due to the CO_2 released during the lime manufacturing process. During the carbonation process, the lime reacts with the CO_2 present in the air, resulting in the conversion of calcium hydroxide ($Ca(OH)_2$) into calcium carbonate ($CaCO_3$) [152]. The carbonation increases the mechanical resistance of the hempcrete and, additionally, the absorption of CO_2 during this process can have significant implications for the environmental impact of this product [153]. Hempcrete can store 300 kg of CO_2 per m^3 [154, 155, 156]. With these two properties hempcrete reduce the energy consumption on the building due to the thermal insulation capacities and also store CO_2, obtaining a material that would passively help to obtain buildings with zero emissions [157]. However, the carbonation process is notably slow, and the amount of CO_2 absorbed is lower than what is generated during the material acquisition phase. As a result, the overall carbon footprint is not deemed sufficient.

Inferences can be drawn from the provided examples that underscore the considerable influence of the binder choice on the composite material's carbon footprint. The suggestion involves the utilization of recyclable materials with minimal carbon footprint impact for the binder material.

2.5.2 Durability of the selected green materials

In order to develop a new biocomposite insulating materials it is necessary to ensure the durability conditions to ensure the commercial product can be reached. However, there are only a few papers that study the durability for long term test in those materials. The longevity of bio-composites can be impacted by various forms of biological degradation, such as mold growth, as well as environmental factors like fluctuations in temperature and humidity [131].

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 insulating, mechanical properties and dimensional stability [143, 158].

Hemp fibers also present a low stability against the contact with water, which see their Young's modulus reduced by 50% after immersion in water [159]. Moreover, using a 100% vegetable material increase the risk to the mold growing in high humidity conditions [142]. Mold growth can be relieve by increasing the pH of the material, as is the case with hempcrete, which has an antifungal properties [88].

In the case of VFRCM (vegetal fabric reinforced cementitious matrix), the issue arises from the alkaline hydrolysis resulting from the production of $Ca(OH)_2$ during cement hydration. The calcium adsorption is pH dependant, the pectin contained in fibres can react with calcium ions in an alkaline environment [160]. Moreover, a reduction in compressive strength has been observed in hempcrete when using $Ca(OH)_2$ treated hemp stalks compared to untreated ones [161, 162]. To incorporate a material that is rich in Al_2O_3 and SiO_2 can mitigate the degradation of the hemp fibers. This material should have the ability to consume $Ca(OH)_2$ during mortar hydration [163]. Alternatively, the fiber can undergo physical or chemical treatment. To extend these findings to vegetable binders, it is crucial to ensure compatibility between the hemp stalk and the vegetal binder to achieve optimal results.

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 [164, 165, 166, 167].

A treatment of colophony is apply to bamboo particle in order to obtain a hydrophobic material. After the coating, bamboo particles become a stable material against the environment [144]. The diterpenoid structure equips rosin with excellent hydrophobicity. Over the recent years, rosin and its derivatives have been used to modify wood materials, wood-based panels, packaging, and nanocomposites increasin the water resistance and the mechanical strenght [168, 169]. Nevertheless, the colophony worsen the fire resistance properties.

Arabic gum 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 arabic gum is soluble in water, so when the layer is in direct contact protective coating is removed until the material is uncoated [145]. 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, as seen in the previous sections.

2.6 Conclusions

According on the results presented in the different studies of the last decades, hemp is positioned as a green material with great insulating properties that could replace some inorganic commercial materials, to improve the insulating performance of building and produce non-structural materials to build green building. These data can be corroborate by the increase in interest in the scientific community, with an increase in research each year, and also with the increase in the hemp industry in the business sector.

- Varieties and positions on the stem can cause differences in the elastic modulus of the hemp stalk of up to 60%. However, no studies have been found to confirm whether these differences in cell size impact the insulating properties of hemp.

  This research gap presents an interesting opportunity for future studies to explore ways to enhance the performance of hemp as a raw material for insulating applications. With the farming of the different varieties of hemp, it is necessary to study which variety produces the hemp stalk with the best insulating properties and in which part of the stem is localized.

- It is suggested to use a binder that aids in creating a low-density and porous material as the microstructure of the composite material plays a significant role in determining its insulating properties.

  It is still necessary to delve into some issues in order to develop new green insulating materials, such as the study of more green binders that do not need to apply pressure or temperature during manufacturing process. In this way, the manufacturing cost is reduced, and lightweight materials with high porosity would be produced that would stimulate the insulating properties of the composite material. In this way, paper pulp based binder shows great opportunities for the development of future research. Interestingly, although cardboard fiber exhibits favorable insulation characteristics, no studies investigating its combination with hemp shiv have been found.

- Despite all the studies, further research is still needed on the use of a 100% plant-based composite material, including the binder. However, among those presented, starch stands out.

  By using starch as a binder, an acoustic absorption (α) of 0.7 and a thermal conductivity of 0.03 W/m K have been achieved, which are respectively 0.2db/db lower and 15% higher than conventional materials.

- The studies reviewed have indicated that the mechanical properties of a fully plant-based material might fall short of meeting the criteria for utilization in the suggested non-structural applications.

  To enhance the material's mechanical performance, a proposal is put forth to incorporate an inorganic skin that would additionally serve to safeguard against degradation due to environmental conditions.

  Another approach involves utilizing a resin containing a proportion of green materials to enhance mechanical performance, offering a cleaner alternative than commercial materials, even though it may not constitute a completely 100% environmentally friendly solution.

- It has been observed that only a few studies have investigated the acoustic properties, fire resistance, fungi growing and long-term durability of 100% green composite materials.
- A ton of dry hemp can store 325 kg of CO_2.

  Hemp is exhibit as a material capable of storing CO_2, and to produce a renewable material with circular economy. The binder also affect to the carbon footprint of the composite material. To prioritize the carbon footprint, a recycled biomaterial such as cardboard fiber can be chosen as a binder.
- It is necessary to protect the biomaterial against the ambient condition.

  The main problem for a biocomposite material is the degradation over time. So in order to produce a commercial product it will be necessary to ensure the stability of the material in ambient conditions. Nevertheless, no research has been found that investigates the influence of using an eco-friendly coating on the insulation properties of a composite material

  To protect the biocomposite material against ambient degradation some vegetable coating such as colophony shows a great performance. Arabic gum is proposed as an effective solution for fire protection, but it only provides protection against moisture and not direct contact with water.

In this thesis study, a large amount of research related to the development of new hemp stalk-based composite materials has been compiled, a lot of research has been done and it is arousing increasing interest in the scientific community for its performance and sustainability reasons. Therefore, it is recommended that these issues continue to be investigated in order to develop a new competitive material capable of replacing current inorganic materials.

It is proposed the use of hemp stalk as a primary material for a sustainable insulation product using a 100% green material to develop it. While previous studies have explored insulating materials based on hemp stalk and vegetal binders, there is still a need for further research to enhance their insulating properties and evaluate their resistance to moisture and fire.

Interestingly, although cardboard fiber exhibits favorable insulation characteristics, no studies investigating its combination with hempstalk have been found. It is possible that the compatibility between these two materials may not be optimal, despite their similar compositions. Hence, this study proposes the investigation of a potential cross-linking agent, such as citric acid, which has not been previously explored in relation to its effect on hemp stalk.

While this new bio-composite material based on hemp stalk and eco-friendly binding materials shows promise as a replacement for traditional inorganic insulating materials in building construction, further research is needed to improve its long-term stability. It is important to ensure that the material maintains its structural integrity and insulating properties over time to ensure its effectiveness and durability in real-world applications. Continued investigation and development of this material will be crucial to its success as a sustainable building material.

The main characteristics of hemp stalk that make it suitable for various applications are its insulating properties and low density, which can be advantageous in non-structural construction applications and urban furniture. To obtain stable materials from hemp stalk particles, it is necessary to add a second material that acts as a binder for the particles. There are numerous plant-based materials that can fulfill this function. However, they generally have certain disadvantages compared to synthetic materials, such as higher raw material costs and lower mechanical properties.

Despite these drawbacks, different materials can be selected depending on the application, where high mechanical properties are not required, thus compensating for the extra cost by using more environmentally friendly materials. Examples include plant-based resins and binders, as well as recycled waste materials such as recycled paper fibers.

Chapter 3

Exploring applications: material composition and manufacturing process

In this chapter, the preliminary studies conducted are presented. These studies encompass the exploration of compatibility between different plant-based binders and hemp stalk, the formulation of material mixing rules, and the thorough characterization of their properties. This constitutes a research study on the material and evaluates the viability of various applications. Based on these studies, the most promising application is chosen as the primary focus of the thesis. Considering that this is a characterization phase, it will not be necessary to obtain all properties of the material, but rather the most characteristic ones, in order to observe the most promising manufacturing methods and applications.

Compatibility tests between hemp stalk and different selected binders will be carried out, along with initial characterization tests to choose the best binders and their proportions. The manufacturing process will also be adapted for each selected application. Based on the literature, five applications have been selected: thermal insulation material, acoustic insulation material, coffered ceiling, particleboard, and parquet flooring. The study will be divided into 2 blocks due to the similarities in specimen fabrication, potential binder materials, and necessary tests. The two blocks are as follows:

- Low density solution: Thermal Insulation Material & Acoustic Insulation Material
- Medium density solution: Coffered ceiling, Particleboard & Parquet Flooring

3.1 Characterization of low density solution

In the application of the insulating material, following the recommendations from the literature, a binder with high insulating properties and a manufacturing method that promotes a highly porous microstructure will be used. This approach will enhance both acoustic and thermal insulation properties. However, these a low density with high porosity material also result in lower mechanical properties of the material.

Given these considerations, the selected binder material is paper fiber, and the manufacturing method involves mixing the components in water, using the interlacing of fibers to achieve cohesion during the material's drying process.

3.1.1 Materials

Hemp stalk

The hemp stalk used in this research was provided by Planteles Lloveras, a harvesting company. The moisture content of the hemp stalk used in ambient conditions is 9-13% with a particle leng among 5-20 mm, Figure 3.1. For additional details regarding the properties of hemp stalk, refer to section 2.2.2 & 2.3.2.

Eucalyptus pulp

The paper pulp used is a ECF (elementary chlorine free) bleached eucalyptus kraft pulp, which was supplied by the company ENCE, Figure 3.4. Since hemp stalk and eucaliptus pulp are of plant origin and possess similar compositions, the cellulose bonds are more likely to form, thereby facilitating the connection between the two components and resulting in the creation of the new biomaterial.

Initially, the use of commercial paper fiber was chosen to confirm the effectiveness of the bond formed by the paper fibers and assess their properties. This approach involved using a controlled commercial material before proceeding with the experimentation using recycled fiber (newspaper, cardboard, etc), which can introduce greater variability and dispersion in the results obtained.

3.1.2 Characterization test

In this section, the tests that will be used to characterize the material and verify its feasibility for use in the application as an insulating material will be defined.

The tests conducted encompassed an examination of the material's compatibility, acoustic and thermal insulation properties, as well as its mechanical response under compression, shear and tension, Table 3.1, where %wt means the composition of every material in weight . The raw result of the different test carried out in the thesis are provided in the Appendix A section.

Table 3.1: Characterization test for low density solution

Test	Variable	Number of samples
Compatibility with paper fiber	%wt. of materials: 9 Moisture of hemp stalk: 2	18
Thermal resistance - ISO 12664	%wt. of materials: 2	8
Acoustic resistance - ISO 10534	%wt. of materials: 2	8
Compression resistance - EN 826	%wt. of materials: 2	6
Shear resistance - EN 12236	%wt. of materials: 2	6
Tensile resistance - ISO 1607	%wt. of materials: 1 Fabrication pressure: 1	6

3.1.3 Compatibility of the materials

Methodology

The initial step involves assessing the compatibility and feasibility of manufacturing using the proposed materials. To achieve this, small samples with varying composition percentages will be fabricated, and their stability will be carefully observed. The outcomes will be visually assessed using the fabricated specimens by manipulating them to confirm the material's stability and absence of drooping. The objective is to identify any differences among the materials' compositions. In Appendix A.1, the conducted tests are described in detail.

Results

To study the compatibility of hemp stalk (Figure 3.1) with fibers, different specimens were prepared to assess the material's stability. Subsequently, mechanical strength, thermal insulation, and acoustic isolation tests will be conducted.

The bonding method employs water to facilitate polymer bonding, specifically through the crosslinking of cellulose, between the two materials. In this process, both materials are mixed in water and then dried to achieve the union.

Before initiating the manufacture of the test samples, an initial evaluation of the raw hemp stalk material to be employed was carried out. Initially, an inspection unveiled that the dried straw comprised a blend of diverse particle and fiber dimensions, Figure 3.2. Consequently, it was determined to submerge the material in water for several days to observe its interaction with water, considering its intended mixing in water during the manufacture process.

Figure 3.1: Hemp stalk size

Figure 3.2 illustrates the makeup of the material, with two distinct samples being gathered. Sample 1 consisted of larger straw pieces akin to wood shavings, while sample 2 consisted of shorter, more delicate fibers. Finally, sample 3 encompassed the complete mixture of straw. Furthermore, the two samples exhibit distinct behaviors in water due to their differing densities. As depicted in Figure 3.2, it can be observed that sample 1 floats, whereas sample 2 sinks.

Hence, a proposition was made to investigate the impact of using dry or wet hemp stalk in the mixing process. This suggestion stems from the possibility of generating a less uniform material if the hemp stalk were to float in the mix, Figure 3.3.

Certain considerations must be taken into account before proceeding. Initially, commercial paper paste is used, utilizing bleached eucalyptus Kraft paste (ECF) to ensure consistent composition in each sample and eliminate possible variations between paper specimens. Once the potential use of hemp stalk as an insulator is confirmed, the commercial paste will be replaced with recycled fibers.

Figure 3.2: Hemp stalk sample classification

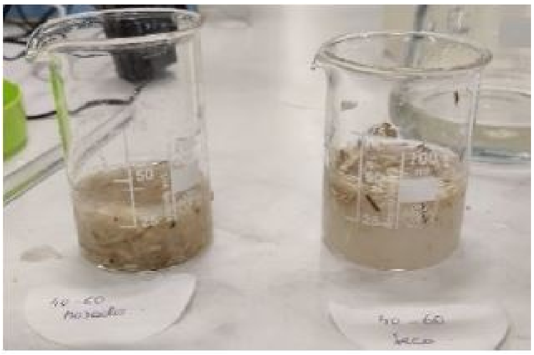


Figure 3.3: Mixing process with wet (left) and dry (right) hemp stalk

The Kraft paste needs to be disintegrated so that the fibers can be individually bonded to the hemp stalk. To accomplish this, a disintegrator is used, creating a centrifugal process in water to separate the paper fibers, Figure 3.4.

To calculate the material mixing ratio, the dry weight of both materials is considered, excluding the water used during disintegration or absorbed by hemp stalk and kraft fiber due to ambient humidity. This ensures an accurate proportion of each material. Figure 3.5 shows the use of a moisture analyzer to determine the dry weights of each material after determining their respective moisture percentages.

Figure 3.4: Disintegrator (left) and disintegrated Kraft paste (right)

In the case of hemp stalk, a study was conducted to determine whether there are differences when using it in a dry or wet state during the mixing process. Hemp stalk in ambient conditions has a moisture content of 9-13%. For the wet hemp stalk, a sample was submerged in water for 24 hours, and afterward, it was separated from the water by filtering the sample using a vacuum pump, Figure 3.6. It was found that the sample increased in weight by 300% due to the water it absorbed. This weight increase is independently calculated each time to ensure that the dry weight remains consistent and the mixing ratio is accurate for each specimen.

Figure 3.5: Sartorius moisture analyzer with a sample of hemp stalk

Figure 3.6: Hemp stalk in water (left) and hemp stalk filtered by vacuum (right)

Once both materials are prepared, the mixture is produced with manual agitation. The mixture will be poured slowly onto a filter paper attached to the bottom of a Buchner funnel, and the excess water is removed by applying vacuum, as shown in Figure 3.7. After vacuum application, the sample is left to air dry for 24 hours.

Figure 3.7: Sample water filtration

The following samples were fabricated using both wet and dry hemp stalk:

- 100% eucalyptus - 0% hemp stalk
- 80% eucalyptus - 20% hemp stalk
- 70% eucalyptus - 30% hemp stalk
- 60% eucalyptus - 40% hemp stalk
- 50% eucalyptus - 50% hemp stalk
- 40% eucalyptus - 60% hemp stalk

- 30% eucalyptus - 70% hemp stalk
- 20% eucalyptus - 80% hemp stalk
- 0% eucalyptus - 100% hemp stalk

Where the percentage indicates the dry weight of each material. It was considered that since the insulating properties of hemp are superior to those of the paper pulp, and considering that hemp is the desired material to be utilized, the goal is to achieve the highest possible percentage of hemp in the material.

The maximum was reached at 70% hemp content with wet hemp stalk, as shown in Figure 3.8. However, at 80% hemp content, the material became unstable with detachment occurring due to insufficient paper fiber to bind all the hemp particles, as shown in Figure 3.9.

On the other hand, the mixture performs better with wet hemp stalk. Dry hemp stalk possesses a density lower than the water used in the mixing process, causing all the hemp components to float to the sample's surface. This leads to the creation of a non-homogeneous material, making the portion with fewer paper fibers unstable. As a result, at a 60% hemp content, detachment of the material occurs.

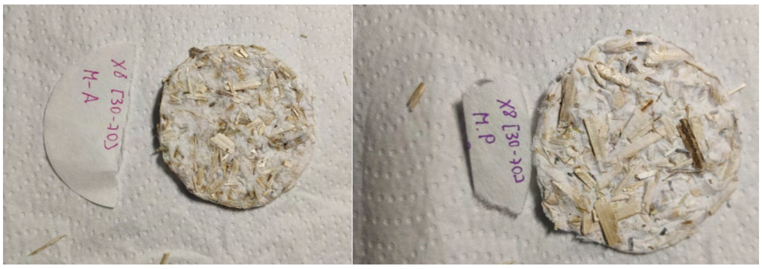


Figure 3.8: Sample 70% wet hemp stalk

Figure 3.9: Sample 80% wet hemp stalk - detachment of the material

With this initial study, a compatibility between hemp stalk and paper fiber is confirmed by manufacturing stable material. Based on this study, the weight ratios for the following samples are determined, with 70% and 60% of dry weight being hemp stalk, and the mixture will be prepared using wet hemp stalk. Once the compatibility is confirmed, different weight sample were produced in order to test the influence in the thickness, Table 3.2.

Table 3.2: Sample thickness with different weight

Sample	Dry mass (g)	Thickness (cm)
30E	5	1.68 ± 0.08
30E	6	1.75 ± 0.05
30E	7	2.28 ± 0.2
40E	6	1.68 ± 0.1
40E	7	2.08 ± 0.05
40E	8	2.30 ± 0.1

3.1.4 Fabrication method

The next step in the compatibility study was to verify if it was possible to achieve a thickness of 5 cm and the required grammage. Circular test specimens with a diameter of 20 cm were prepared, the dry weight of the mixture was increased, and the final thickness of the sample was measured. The result was a grammage of 6 kg/m^2 to achieve a thickness of 5 cm, corresponding to a density of 120 kg/m^3 (for comparison, the density of expanded polystyrene, EPS, ranges from 20 to 50 kg/m^3).

The manufacturing process remained the same, except for slight variations in the water extraction method due to the dimensions. The samples were fabricated using a paper sheet former called Frank, following the Rapid-Köthen method, Figure 3.10. The extraction principle was the same, applying vacuum from the bottom while preventing the sample from being removed by using a mesh, although there were minor material losses. For the 5 cm thick specimens, a drying oven was necessary, set at 40°C for 48 hours, to reduce drying time and prevent the occurrence of fungi.

Figure 3.10: Frank sheet former (left) and drying oven (right)

With this method, the specimens are fabricated following the selected configuration from the previous process:

- 70% wet hemp stalk - 30% eucalyptus fibber: 30E
- 60% wet hemp stalk - 40% eucalyptus fibber: 40E

All the fabricated specimens resulted in stable and homogeneous material, as shown in Figure 3.11, which underwent characterization tests to verify their insulating and mechanical properties.

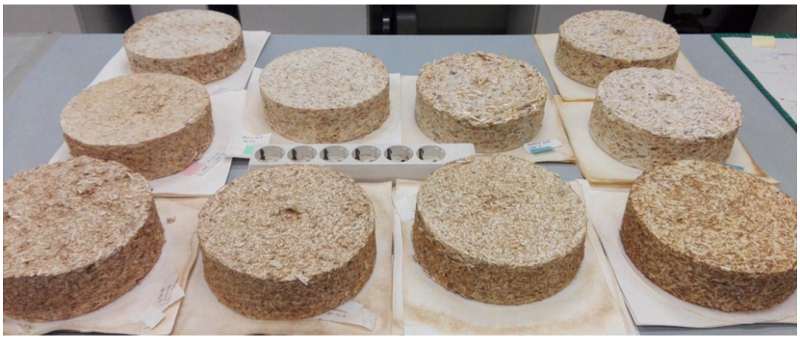


Figure 3.11: Hemp stalk-eucalyptus sample

The tensile test was conducted on compressed samples. The goal of the compressed sample is to examine its potential for incorporation into a sandwich-type material using the same constituents. This sandwich structure entails an uncompressed material serving as a low-density core and the compressed material as the outer layer. The bonding of these materials will be achieved using an environmentally-friendly epoxy resin, as depicted in Figure 3.13.

In the case of the tensile test, a difference was introduced in the manufacturing process explained earlier. To increase the tensile strength of the material, a pressing step was proposed to be performed while the material is still damp, immediately after the vacuum drying process. The pressing was carried out using a pressure of 0.6 MPa in a paper press, as shown in Figure 3.12. Applying this pressure reduced the thickness from 50 to 6 mm, measured with a micrometer, Figure 3.12. To maintain a more uniform thickness and improve material cohesion for this test, a different composition was chosen, consisting of 50% hemp stalk and 50% paper fiber.

Figure 3.12: Compressed hemp stalk-eucalyptus sample (left), manual paper press (center) and micrometer (right)

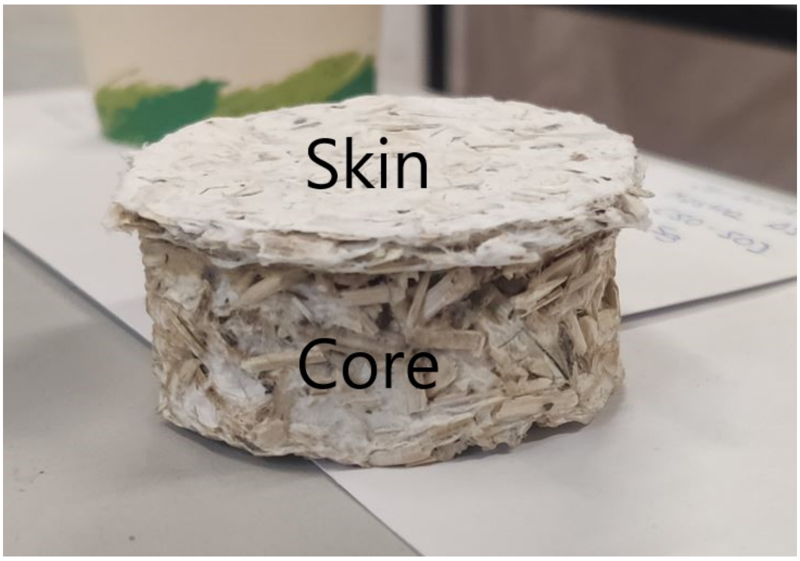


Figure 3.13: Sandwich type sample, core formed with 30E and skin with 50E compressed

In the characterization tests, three distinct sample types were analyzed, both employing the identical binder, eucalyptus fiber, albeit in varying weight proportions. One sample contained 30% eucalyptus fiber by weight, designated as 30E, while the other contained 40%, labeled as 40E. Additionally, a compressed sample with 50% eucalyptus fiber by weight was labeled as 50E.

3.1.5 Acoustic insulation properties

Methodology

The acoustic properties were assessed using a two-microphone impedance tube, specifically the Brüel & Kjaer type 4206, Figure 3.14, in accordance with EN 10534 [49]. 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, Figure 3.15. 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. In Appendix A.2, the conducted tests and the raw data obtained are described in detail.

The test sample is mounted at one end of a straight, rigid, smooth and airtight impedance tube. Plane waves are generated in the tube by a sound source (random, pseudo-random sequence, or chirp), and the sound pressures are measured at two locations near to the sample. The complex acoustic transfer function of the two microphone signals is determined and used to compute the normal-incidence complex reflection factor, the normal-incidence absorption coefficient, and the impedance ratio of the test material.

From these data, a result is obtained for normal incidence on the sample, from which diffuse incidence coefficients can be estimated. This represents a rapid and valid prototype-level study, eliminating the need for tests in large acoustic chambers.

In this case, 2 microphones are used, so it is necessary to calibrate the equipment before conducting the test to ensure that the two microphones are in phase and amplitude.

Figure 3.14: Kundt's Tube

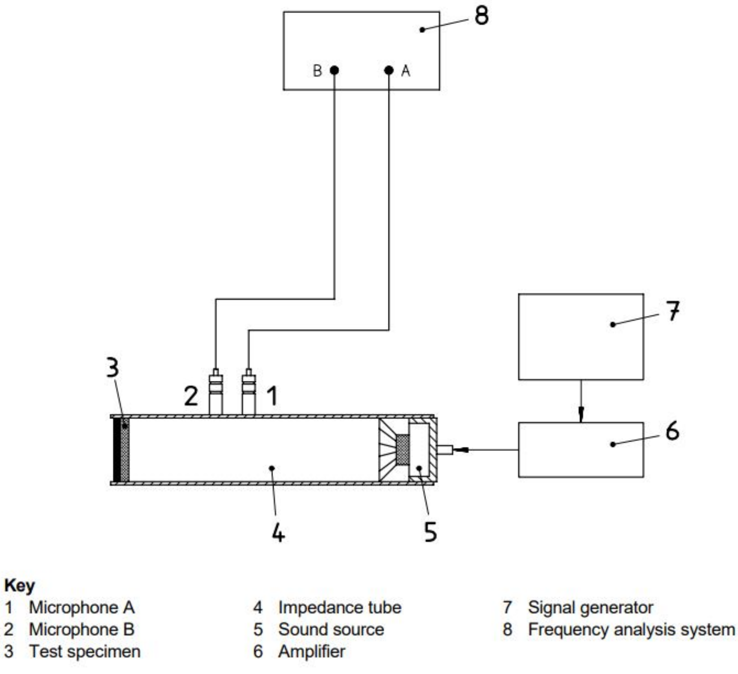


Figure 3.15: Layout for acoustic test equipment [49]

Equipment:

- Impedance tube: tube with a test sample holder at one end, a sound source at the other and two microphone ports.
- Microphones: two identical microphones to produce the sound waves with the same amplitude and phase.
- Signal generator: a signal generator capable to o generate a stationary signal with a flat spectral density within the frequency range of interest. It may generate one or more of the following: random, pseudo-random, periodic pseudo-random or chirp excitation. Brüel & Kjaer type 4206 produces random signal.
- Amplifier: sound signal amplifier
- Frequency analysis system: a two-channel Fast Fourier Transform (FFT) analysing system. The system is required to measure the sound pressure at two microphone locations and to calculate the transfer function H12 between them.
- Computer with the Brüel & Kjaer signal analysis software

Results

During the acoustic test, it's crucial to consider that the test requires completely flat sample surfaces for accurate results. In the case of the material developed in this thesis, the surface displays some roughness due to the presence of hemp stalk. This roughness appeared to affect the acoustic absorption within a specific frequency range during the test. However, it's important to note that this reduction is merely an artifact caused by the sample's geometry and does not reflect accurate values. To address this, the same sample was tested with a rotation around its central axis, altering the wave's path and eliminating the artifact. It's essential to highlight that this rotation only impacts the frequency range where accurate results weren't initially obtained, while in the remaining range, the margin of error is less than 1%, thus validating the final sample results.

Additionally, it is important to note that the thickness of the test specimen is 2 cm, while the manufactured sample has a thickness of 5 cm, and there is a difference in porosity along the thickness. For this reason, three specimens were created based on the selected position:

- 1: Bottom part
- 2: Middle part
- 3: Top part

Figure 3.16: Distribution of paper fiber along the sample: top part in the image was in contact with the vacuum

Due to the vacuum process during manufacturing, it is observed in the samples that the lower part contains a higher amount of paper fibers, Figure 3.16. This is because they are capable of penetrating the gaps formed by the hemp stalk, resulting in a non-completely homogeneous distribution. This effect is less noticeable with a lower percentage of hemp stalk. In Figure 3.17 the impact of this process on performance can be observed, where the sample of the top part show a better acoustic properties due to have a higher porosity. Furthermore, to achieve a more homogeneous material for future studies, it is advisable to reduce the thickness and avoid using the vacuum process, even though it may require more time for the samples to dry.

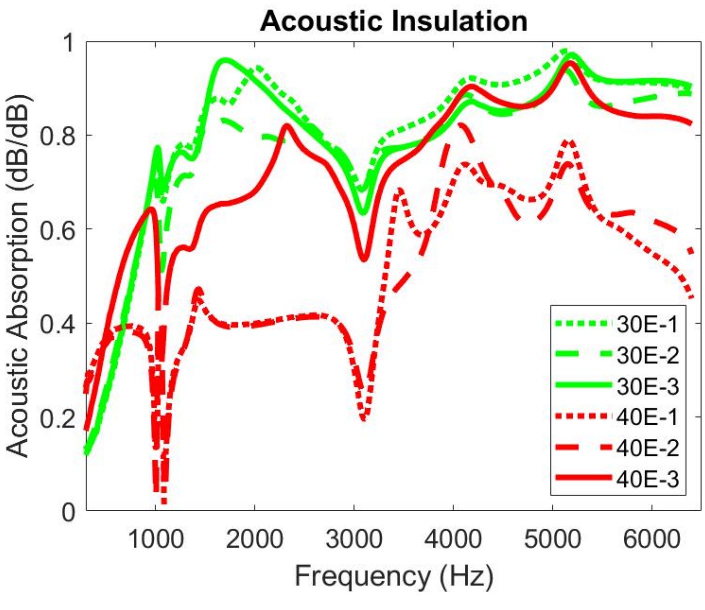


Figure 3.17: Acoustic absorption of the hemp stalk-eucalyptus specimens

In Figure 3.18 the results of the acoustic absorption (α) of hemp stalk-Kraft paste specimens with 40% and 30% weight of the top part in the sample are compared with commercial synthetic materials such as rock wool and EPS.

It can be observe that the absorption is reduced in the lower frequency range (below 1000 Hz) but increases until reaching a maximum and then decreases, following the trend reported in the literature [59]. The lower absorption at low-frequency ranges is attributed to the material's high porosity, while the increased porosity favors higher absorption at higher frequencies.

From 1000 Hz onwards, the absorption values remain above 0.7, making it an effective insulating material, although the results are slightly inferior to those of rock wool.

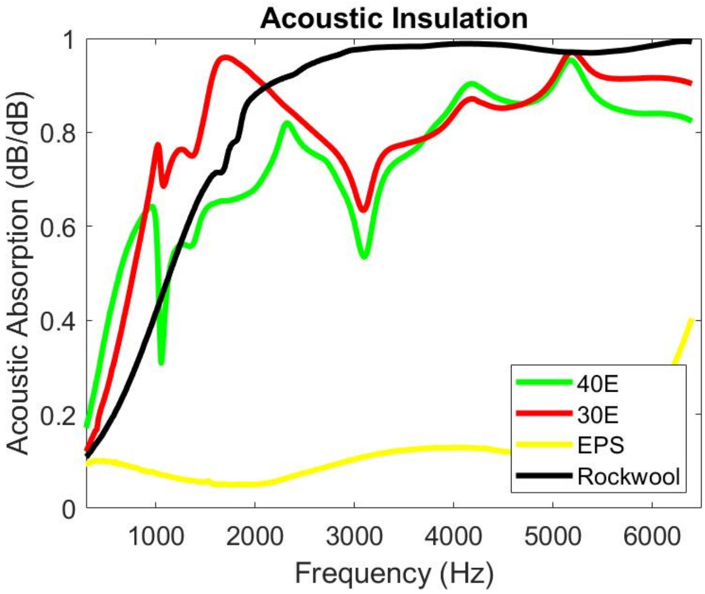


Figure 3.18: Acoustic absorption of the hemp stalk-eucalyptus specimens compared to commercial materials (EPS and rockwoll)

3.1.6 Thermal insulation properties

Methodology

For assessing thermal insulation properties, a square test specimen measuring 10 cm in width and 5 cm in thickness is positioned within a thermal chamber, Figure 3.19 & Figure 3.20 . 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 [48] 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. To ensure the thermal stability of the material the gradient of temperature is calculated after 1 hour of test, when the quasi-stationary phase is archived. In Appendix A.3, the conducted tests and the raw data obtained are described in detail.

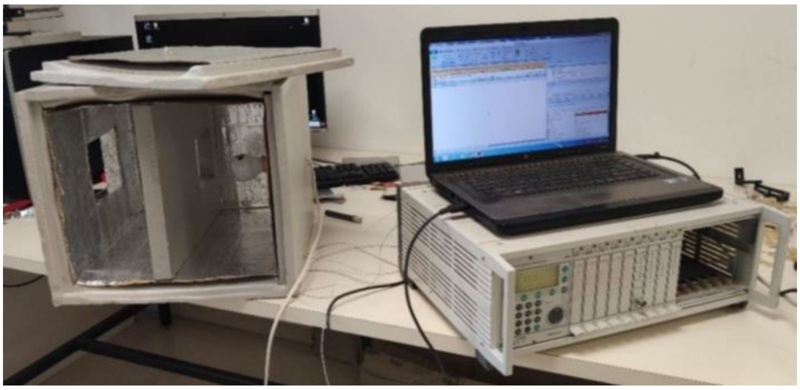


Figure 3.19: Thermal resistance test

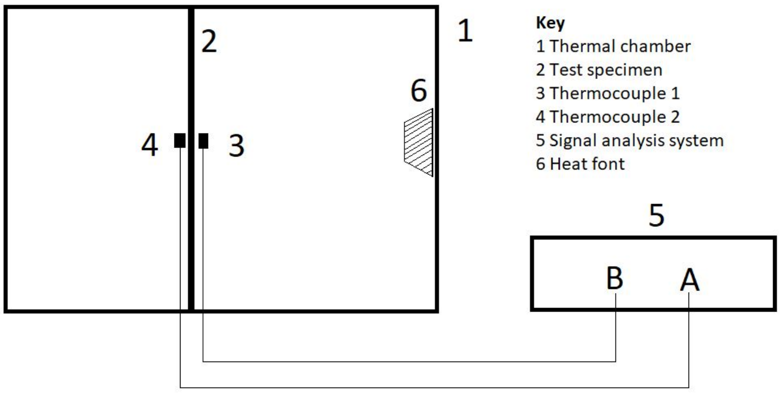


Figure 3.20: Layout for thermal test equipment

Under the assumption of a steady-state condition, a unidirectional heat flow within the thermal chamber, and exclusive heat transfer through conduction along the specimen, the thermal conductivity coefficient of the material (λ) is determined by utilizing the two acquired temperature values and the geometric properties of the test specimen.

$$Q_x = -\lambda \ A \ \frac{\partial T}{\partial x} \tag{3.1}$$

$$\frac{\partial T}{\partial x} \approx \frac{\Delta T}{\Delta x} = \frac{T_2 - T_1}{e} \tag{3.2}$$

$$\lambda = \frac{Q_x \ e}{A \ (T_1 - T_2)} \tag{3.3}$$

- Q_x: Applied heat (W)
- λ: Thermal conductivity coefficient ($\frac{W}{m \ K}$)
- A: Crosssection of the sample (m^2)

- e: Thickness of the sample (m)
- $\frac{\partial T}{\partial x}$: Temperature gradient in the x-direction
- T_1: Temperature measured with the thermocouple in the position near to the heat font (K)
- T_2: Temperature measured with the thermocouple in the outer part of the sample (K)

For calibrating the Q_x value, a commercially available material with tabulated thermal conductivity (λ) is introduced into the thermal chamber. As a result, Q_x is determined using the temperature values recorded by the thermocouples.

Equipment:

- Thermal chamber: Insulating chamber capable to reduce the heat transfer to the exterior in order to obtain a high data accuracy.
- 60 W heat font.
- Thermocouples: 2 identical thermocouples to measured the temperature of the sample.
- MGCPlus: HBM signal acquisition.
- Computer with the CATMANEasy software.

Results

The outcomes of the various conducted specimens are presented in Figure 3.21. Notably, in this instance, the thermal conductivity (λ) is observed to be 10-15% higher than that of commercial materials, indicating that the developed material holds promise as an exceptional thermal insulator.

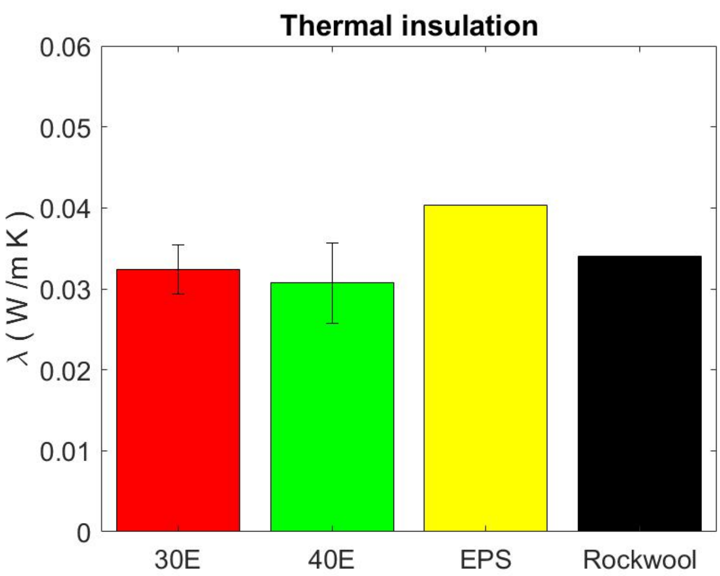


Figure 3.21: Thermal resistance of the hemp stalk-Kraft specimens compared to commercial materials

3.1.7 Mechanical resistance: compression, shear & tension

In addition to the previous tests, mechanical property tests were conducted to assess the strength of the material. These tests aimed to compare the mechanical properties of the material with those of commercially available materials used in similar applications. The objective was to ensure that the material would withstand its intended lifespan without experiencing any significant breakage or failure by comparing its mechanical performance with other commercial insulating panels (EPS).

Methodology - compression

For the compression test, prismatic specimens with dimensions of 100x100x50 mm were prepared. These specimens were subjected to compression testing at a constant test speed of 5 mm/min with a MTS insight 10 kN. The purpose of the test was to evaluate the material's resistance to compression and determine its ability to withstand 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 [50]. A preload of 10 N is applied to the samples. In Appendix A.4, the conducted tests and the raw data obtained are described in detail.

When the value of the maximum stress corresponds to a strain of less than 10%, it is designated as compressive strength and the corresponding strain is reported. If no failure is observed before the 10% strain has been reached, the compressive stress at 10% strain is calculated and its value reported as compressive stress at 10% strain, Figure 3.22.

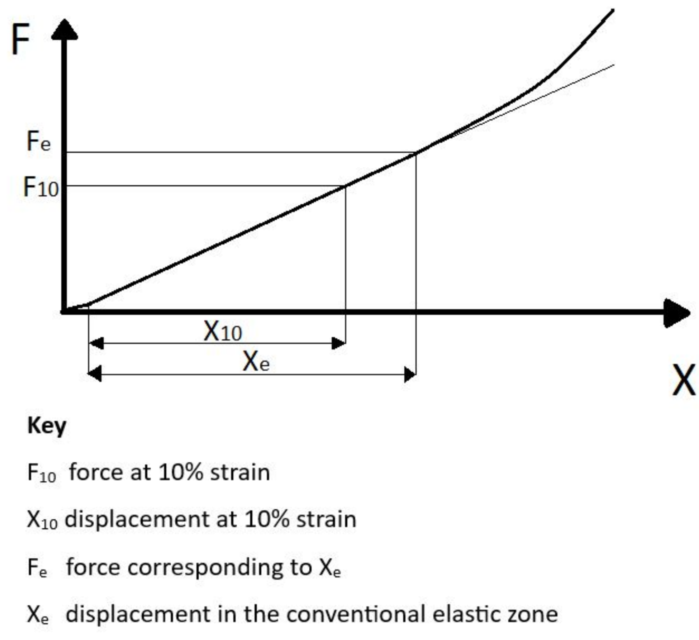


Figure 3.22: Example of compression strain-stress curves [50]

$$\sigma = \frac{F}{A_0} \tag{3.4}$$

$$\varepsilon = \frac{x}{e}\ 100 \tag{3.5}$$

$$E = \sigma_{10}\ \frac{e}{x_{10}} \tag{3.6}$$

- σ: Compressive stress (MPa)
- ε: Relative deformation (mm/mm)
- F: Applied force (N)
- x: Displacement (mm)
- A_0: Initial surface of the sample (mm^2)
- e: Initial thickness of the sample (mm)
- E: Compression modulus of elasticity (MPa)

Equipment:

- MTS insight 10 kN: Universal testing machine, designed to suit the range of force and displacement involved and having two very rigid, polished, square or circular plane parallel platens with a minimum side length (or diameter) equal to the side length (or diagonal) of the test specimen. One of the plates shall be fixed and the other movable.
- Computer with the MTS software.

Methodology - shear

The shear test was performed using a static puncture test (CBR test). Cylindrical specimens with a radius of 10 cm and thickness of 5 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 [51]. The shear test was conducted using an MTS Insight 300 kN machine, with a constant test speed of 5 mm/min. A preload of 20 N is applied. In Appendix A.5, the conducted tests and the raw data obtained are described in detail, Figure 3.23.

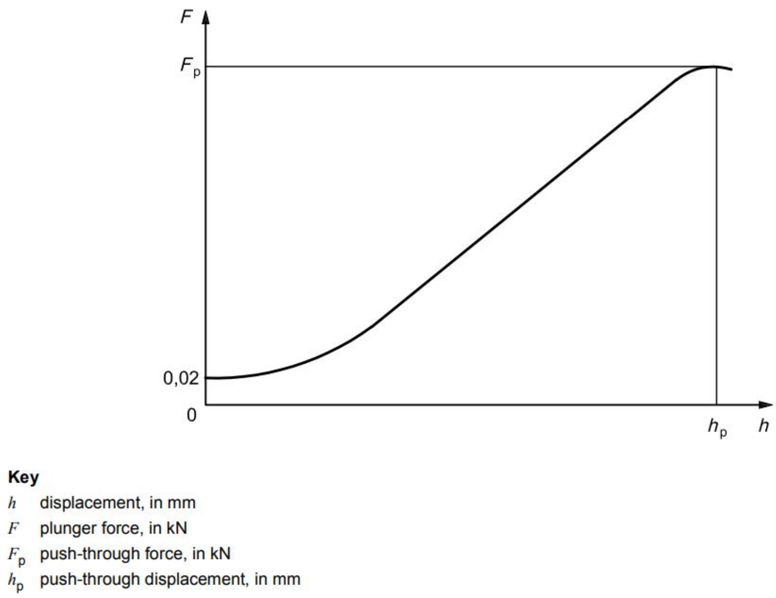


Figure 3.23: Example of static puncture test curves [51]

$$\tau = \frac{F}{A_0} \tag{3.7}$$

$$\gamma = \frac{h}{e} \tag{3.8}$$

- τ: Shear stress (MPa)
- γ: Relative deformation (mm/mm)
- F: Applied force (N)
- h: Displacement (mm)
- A_0: Initial shear surface of the sample (mm^2), which correspond to the lateral surface that the plunger will pass through ($2\ \pi\ r_{plunger}\ e$).
- e: Initial thickness of the sample (mm)

Equipment:

- MTS insight 300 kN: Universal testing machine, designed to suit the range of force and displacement involved and having two very rigid, polished, square or circular plane parallel platens with a minimum side length (or diameter) equal to the side length (or diagonal) of the test specimen. One of the plates shall be fixed and the other movable.
- Plunger: Steel plunger with a diameter of 60 mm.
- Clamping system: PVC clamping system that prevent slippage or cutting of the specimens with a

internal diameter of 120 mm.

- Computer with the MTS software.

Methodology - tensile

Tensile test was performed by placing the material between two clamps, one of which was movable to stretch the material. The test procedure followed the regulations outlined in UNE 1607:2013 [170]. Prismatic specimens with dimensions of 100x50x6 mm were tested. Tensile test was conducted using an MTS Insight 10 kN, with a constant test speed of 5 mm/min. In Appendix A.7, the conducted tests and the raw data obtained are described in detail, Figure 3.24.

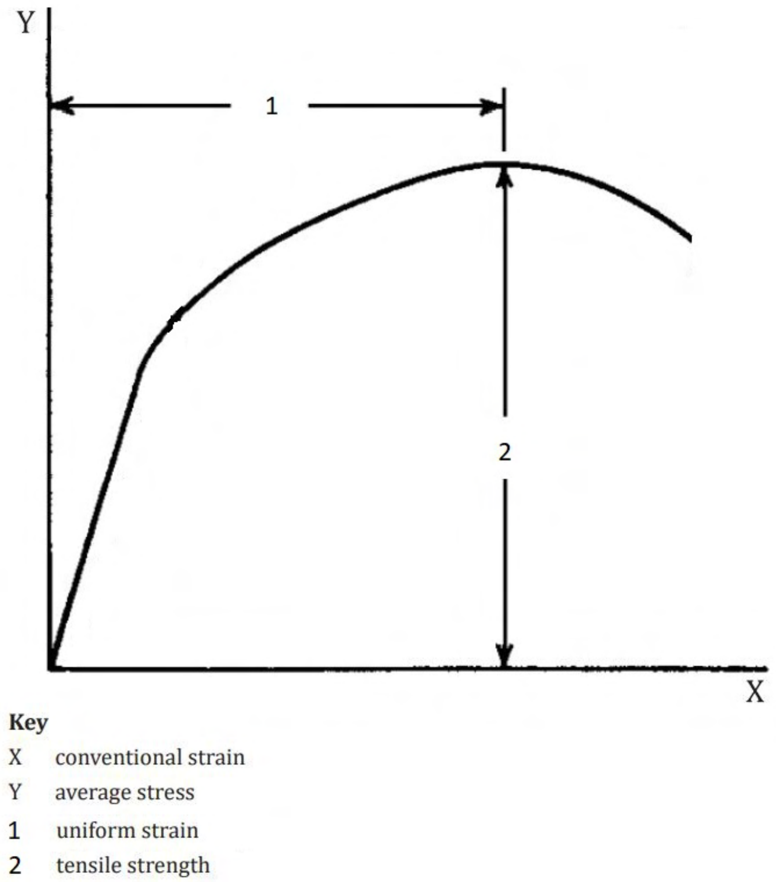


Figure 3.24: Example of tensile test curves [170]

$$\sigma = \frac{F}{A_0} \tag{3.9}$$

$$\varepsilon = \frac{d}{l} \tag{3.10}$$

- σ: Tensile stress (MPa)
- ε: Relative deformation (mm/mm)
- F: Applied force (N)
- d: Displacement (mm)
- A_0: Initial crossection area of the sample (mm^2).
- l: Initial length of the sample (mm)

Equipment:

- MTS insight 10 kN, for more details see 3.1.7.
- Clamp: Clamps capable to hold the test specimen without allowing it to slip and designed so that they do not cut or otherwise weaken the test specimen. The jaws should be installed in the plates of the testing machine.
- Computer with the MTS software.
- Extensometer: Extensometer of 50 mm to measure the displacement with a precision of 0.005 mm.

Results

Once the insulating properties have been verified, mechanical strength tests are conducted to validate the mechanical properties of the developed material.

To validate the results of the mechanical tests, they will be compared with the mechanical properties of EPS panels. This comparison will help determine whether the developed panels are strong enough to withstand storage, transportation, and installation without being damaged. Figure 3.25 show the compression test carried out. Figure 3.26 show the configuration of the static puncture test.

Figure 3.25: a) Compression test b) 40E before test c) 40E after test

Figure 3.26: Static puncture test - shear test (left) and sample 40E after test (right)

For the tensile test, the samples were reinforced with fiberglass impregnated with epoxy resin (MasterBrace P 3500) at both ends to facilitate gripping with the clamps during the test, Figure 3.27.

Figure 3.27: Tensile samples with glass fibber to improve the grip

The purpose of conducting a tensile test, Figure 3.28, on the pressed material is to verify its mechanical strength to study the possibility of using the pressed material as a skin for a composite material, if needed.

Figure 3.28: a) Tensile test b) ending of the tensile test

Table 3.3: Compression, shear and bending test results

Sample	**Compression**	**Shear**	**Tensile**
	Young Modulus (MPa) C.V. (%)	**Shear stress (kPa) C.V. (%)**	**Tensile strenght (kPa) C.V. (%)**
30E	0.810 (15)	15.1 (27)	-
40E	0.664 (39)	17.0 (33)	-
50E	-	-	259.0 (21)
EPS	0.5	30.0	850.0

In Table 3.3, the compression behavior resembles that of EPS. The green composite material does not break under compression stress, as its porosity allows it to be compressed and reduce its thickness significantly. This results in an exponential curve for force-displacement, but for data analysis, only displacements up to 10% are considered within the elastic behavior range. The compression Young's modulus is 50% higher than that of EPS in the case of 30E.

Cardboard fibers exhibit a mechanical strength four times greater than eucalyptus fibers, as measured by their bonding capacity [112, 113]. This bonding capacity provides a more representative value than the resistance of an individual fiber due to their geometry (cardboard fibers have an average length of 2.7 mm, while eucalyptus fibers have 0.76 mm [114]). In Table 3.3, the shear stress values of the specimens made with Kraft paper are 50% lower than EPS. To enhance the shear performance of the material, it is necessary to use a fiber with higher mechanical strength. Instead of using newspaper paper fiber, cardboard provides superior mechanical strength, longer fibers, and is obtained through a recycling process, making the final material more sustainable than using the initial Kraft paper.

In the case of compressed samples, the tensile strength is too low to use it as a skin in a composite material. However, it can be reinforced with continuous hemp fibers and impregnated with resin to improve its mechanical performance.

3.1.8 Fungal growth

Some of the samples were not completely dry when they were taken out of the oven, creating a moist interior that favored fungal growth, as illustrated in Figure 3.29. After being stored in a controlled environment at 23°C and 50% humidity for several days before testing, fungal growth started. As a result, these samples were considered unsuitable for the characterization tests. This highlights the importance of fully drying the samples in the oven. Additionally, the inclusion of a protective layer is crucial to safeguard the material against fungal growth in highly humid environmental conditions.

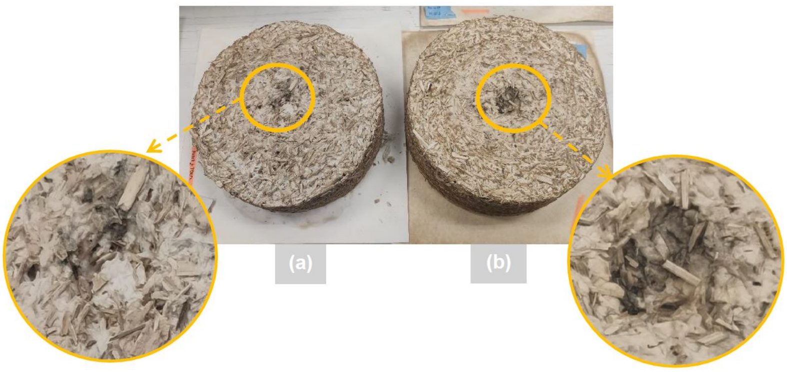


Figure 3.29: Samples with fungi growth; a) 30E; b) 40E

3.2 Characterization of medium density solution

In this section, three different applications (coffer ceiling, chipboard, parquet flooring) will be analyzed by using the same materials and subject them to mechanical tests. The main goal is to determine the most suitable application to study with the various materials currently under development.

For this purpose, the mechanical performance of the materials holds significant importance in the decision-making process. To achieve the highest mechanical performance, it is crucial to manufacture the materials in a manner similar to chipboard, which is known for its high-density resulting from the application of substantial pressure during the manufacturing process. However, it is essential to consider that the binding of the hemp stalk requires a binder with superior mechanical properties, although it should be noted that green resins generally exhibit lower performance compared to inorganic resins.

3.2.1 Materials

Hemp stalk

The hemp stalk provided by Planteles Lloveras, a harvesting company. For more information see section 3.1.1.

Colophony

Colophony, an abundant and cost-effective natural resin derived from pine trees, is a renewable and biodegradable resource with low molecular weight. Its primary constituent is abietic acid, a partially unsaturated compound comprising three fused six-membered rings and one carboxyl group [169]. It was provided by the company La Mezcla Perfecta, Figure 3.30.

Arabic gum

Gum Arabic, or acacia gum, is an exudate produced by acacia trees in sub-Saharan countries, which operates as a natural wound plaster, thus shielding trees against insects, molds and droughts. Its a highly water soluble material [145]. It was provided by the company La Mezcla Perfecta, Figure 3.30.

Corn starch

Starch, Figure 3.30, is a carbohydrate composed of multiple glucose units connected by glycoside bonds. When mixed with hot water, it creates a wheat-like dough, frequently used as a thickening agent, stiffener, or adhesive. In industrial settings, starch, particularly in non-food applications, plays a significant role as an adhesive in paper manufacturing.

Bioepoxy

Epoxy resin made in a 30% with green materials in order to corroborate the mechanical properties of an intermediate solution, Figure 3.30, provided by sportresins. The datasheet is provided in appendix B.1.

White glue

White glue HM-425 serves as a reference material since it ranks among the most frequently employed inorganic adhesives for wood-based products, Figure 3.30, provided by Obramat. The datasheet is provided in appendix B.2.

(a) Colophony

(b) Arabic gum

(c) Corn starch

(d) White glue

(e) Bioepoxy

Figure 3.30: Binder for medium density application

3.2.2 Characterization test

In this section, the characterization tests that are utilized to assess the material's suitability for different applications and validate its feasibility will be defined.

Table 3.4: Characterization test for medium density solution

Test	Variable	Number of samples
Tensile test - EN 319	Binder: 7 %wt. of materials: 3 Curing time: 3 Fabrication method: 3	78
Bending test - EN 310	Binder: 7 %wt. of materials: 3 Curing time: 3 Fabrication method: 3	78

The tests carried out encompass the examination of material compatibility, involving the characterization of its mechanical response in both tensile and bending scenarios, Table 3.4. Should a particular application be identified as the most promising, the corresponding test will be conducted accordingly.

3.2.3 Fabrication method

This section presents the study of the compatibility between hemp stalk and the resins to be used in the applications of particle boards, parquet, and coffer ceiling. To conduct the study, composite specimens were fabricated using different resin selections and three different compositions (10 g of hemp stalk with 2/4/6 g of binder) to verify the stability of the resulting material, Figure 3.33. Subsequently, mechanical characterization tests were performed to determine the best proportions and binders.

In the case of arabic gum and colophony, they are in a solid state initially. To create the binder, they need to be dissolved. Colophony is dissolved in acetone with a 2:1 ratio at 50°C, while arabic gum is dissolved in water at a 2:1 ratio at 90°C, as shown in Figure 3.31.

Figure 3.31: Colophony (left), arabic gum (center) and adhesive made with corn starch (right)

Corn starch was employed as a binding agent. For this purpose, food-grade starch was utilized following proportions outlined in the literature, with a ratio of 100 g of water for every 18 g of starch at 65°C, and the mixture was subjected to temperature and agitation [84], as illustrated in Figure 3.31.

For making the specimens, two steel molds were purchased, Figure 3.32, to contain the hemp stalk mixed with the previously prepared binder, and pressure was applied from below to obtain the correct dimensions. In industrial manufacturing, 5 MPa pressure is applied for 5 minutes at temperatures above 100°C, depending on the binder used. Synthetic binders employed in the industry can cure in 5 minutes under these conditions. They use synthetic binders that accelerate the curing process at high temperatures, such as PMDI or UF.

In the case of vegetable resins, temperature does not reduce the curing time. To make an initial approximation of the best selection, it was decided to apply 5 MPa pressure within the molds for 5 minutes instead of applying pressure during the entire curing time and completing the curing process outside the molds at ambient conditions. This process results in slightly lower properties, but it will facilitate the selection of resins with comparable results.

Once the resins are selected, the improvement of the manufacturing process will be considered to meet the necessary specifications for each proposed application.

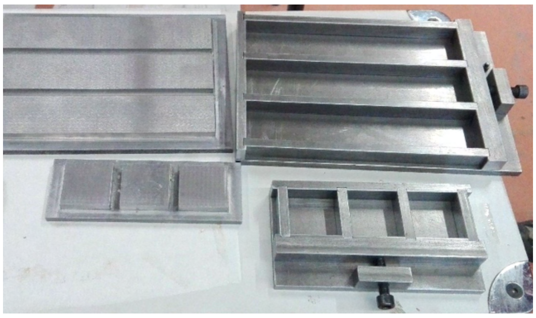


Figure 3.32: Molds for the fabrication of tensile and bending specimens

Table 3.5: Nomenclature structure of the samples

Composition - % wt. relation binder-hemp stalk	Binder
	Colophony
2-10	Arabic Gum
4-10	Corn starch
6-10	Bioepoxy
	White glue

Figure 3.33: Specimens made with arabic gum

Favorable results were obtained with the five resin tested, allowing subsequent mechanical tests to be carried out. However, it was observed that curing outside the mold at ambient temperature caused the specimens to increase in size during curing. Consequently, the properties obtained will be inferior to those achieved when pressure is applied throughout the entire curing process. In adition, a highly viscous binder, such as white glue, presented challenges during the mixing process of the two materials, especially when used in small quantities, leading to a non-homogeneous mixture.

Notably, the specimens made with arabic gum, which utilizes water, exhibited the most significant changes in dimensions. Specimens with higher proportions of binder tended to break due to the significant increase in dimensions. This phenomenon occurs because the hemp absorbs water from the resin, leading to volume expansion.

Due to the similar behavior observed in the samples, an optimization study of the fabrication method was conducted using the most promising resin. Subsequently, this optimization process will be extended to the other resins that were utilized. The primary emphasis of the optimization for vegetable-based resins was placed on refining the manufacturing process and managing humidity levels during the curing stage, as these were the primary challenges encountered during the initial fabrication.

3.2.4 Mechanical test: Tensile & bending

Methodology - tensile

The test procedure followed the regulations outlined in EN 319 [71]. Prismatic specimens with dimensions of 50x50x5 mm were used. Tensile test was conducted using an MTS Insight machine 10 kN, with a constant test speed of 5 mm/min. In Appendix A.10, the conducted tests and the raw data obtained are described in detail.

The test setup resembles the tensile test described in the preceding section, but with a variation in the mechanism. In this case, a self-aligning ball and socket joint component is positioned within the testing machine and connected to a wooden block to exert force on the sample, as shown in Figure 3.34. The sample is affixed to the wooden block using white glue to ensure that the failure occurs within the sample itself and not at the bonding interface.

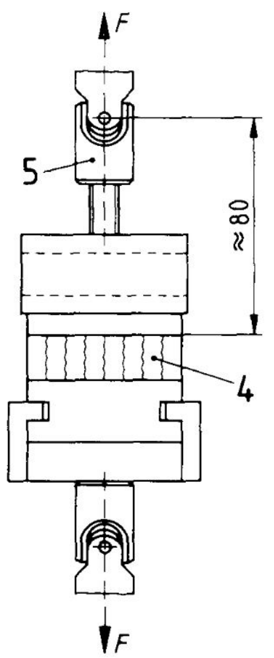


Figure 3.34: Self-aligning ball and socket joint configuration [71]

$$\sigma = \frac{F}{A_0} \quad (3.11)$$

$$\varepsilon = \frac{d}{l} \quad (3.12)$$

- σ: Tensile stress (MPa)
- ε: Relative deformation (mm/mm)
- F: Applied force (N)
- d: Displacement (mm)
- A_0: Initial crossection area of the sample (mm^2).
- l: Initial length of the sample (mm)

Equipment:

- MTS insight 10 kN: Universal testing machine, for more details see 3.1.7.
- Self-aligning ball and socket joint: This component is responsible for securing the sample and aligning the test configuration to generate tensile stress. The wooden piece and adhesive utilized to secure the sample must offer sufficient strength without interfering with the test, thus preserving the properties of the sample.
- Computer with the MTS software.

In this situation, it was not possible to install the extensometer during the tensile test due to limitations in the sample's geometry. Nevertheless, the applied load was small enough to disregard any displacement of the connecting tools. Consequently, it is assumed that the displacement of the machine is the same that the displacement of the test sample.

Methodology - bending

A 3-point bending test configuration was employed, Figure 3.35, with a distance of 200 mm between the supports. The test procedure followed the regulations outlined in EN 310 [73]. Prismatic specimens with dimensions of 250x50x5 mm wer tested. Tensile test was conducted using an MTS Insight machine 10 kN, with a constant test speed of 5 mm/min. In Appendix A.11, the conducted tests and the raw data obtained are described in detail.

The formulas described below correspond to the standard used, where a linear stress distribution is assumed. It's important to note that this assumption doesn't hold for the proposed material. However, the formulation will be retained to facilitate comparisons between the test specimens and commercially available results.

Developing a formulation capable of addressing the nonlinearity of this material would increase the complexity of the results analysis, which is not the primary scope of this thesis.

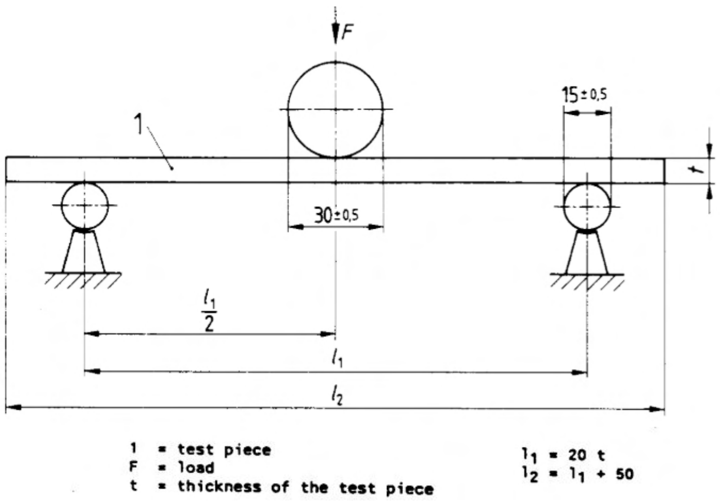


Figure 3.35: Bending test configuration [73]

$$\sigma = \frac{3\ F\ l_1}{2\ b\ t^2} \tag{3.13}$$

$$E = \frac{l_1^3\ (F_2 - F_1)}{4\ b\ t^3\ (a_2 - a_1)} \tag{3.14}$$

- σ: Bending stress (MPa)
- E: Bending young modulus.
- F: Applied force (N)
- d: Displacement (mm)
- l_1: Distance between the centres of the supports (mm)
- b: Width of the sample (mm)
- t: Thickness of the sample (mm)
- $F_2 - F_1$: Increment of load on the straight line portion of the load-deflection curve. F_1 and F_2 shall be approximately 10% and 40% of the maximum load (N).
- $a_2 - a_1$: Increment of deflection at the mid-lenght of the sample corresponding to F_1 and F_2 (mm).

Equipment:

- MTS insight 10 kN: Universal testing machine, for more details see 3.1.7.
- Cylindrical load head.

- Two parallel supports.
- Computer with the MTS software.

Figure 3.36 show the configuration of the 2 mechanical test presented.

Figure 3.36: Tensile test (left) and bending test (right)

Results

The outcomes of the preliminary specimens are presented in Table 3.6. The table presents the results obtained for each case. However, it should be noted that certain samples became unstable and broke on their own before testing, leading to values in the table without a coefficient of variation. Of particular significance is the case of starch resin, where all the samples prepared proved to be unstable due to the high moisture content of the resin, causing the samples to swell during the curing process, so it was removed from the table.

The primary objective of this initial investigation is to establish the appropriate blending protocol and validate specimen fabrication. For non-vegetable resins, an increase in resin quantity corresponds to improved tensile and bending properties. For the white glue, the 2-10 composition exhibited low binder content to produce a good mix.

However, this pattern does not hold true for vegetable resins. In the case of 6-10 composition with arabic gum and colophony, the tensile sample were unstable. This discrepancy is attributed to residual moisture in these resins, which leads to an enlargement of hemp stalk and an expansion of specimen dimensions during air curing.

Both vegetable and synthetic resin results obtained fall significantly below the benchmarks set by commercial materials (tensile strenght: 0.4 MPa & bending strenght: 11 MPa [72]), underscoring the need for production enhancements. Subsequently, the focus shifts toward refining the conducted processes to minimize specimen numbers. These interim steps will exclusively involve vegetable resins. Once the final process is selected, specimens of all types would be produced to facilitate comparative analysis.

Table 3.6: Tensile and bending test results

Binder	Composition	Tensile strength (kPa), C.V. (%)	Bending young modulus (MPa), C.V. (%)	Bending strength (kPa), C.V. (%)
White glue	2-10	45.7 (15)	-	-
	4-10	109.5 (40)	21.27 (-)	150.6 (-)
	6-10	279.5 (-)	39.70 (-)	491.9 (1)
Bioepoxy	2-10	19.5 (35)	2.12 (29)	317 (45)
	4-10	139.7 (15)	59.16 (26)	208.9 (23)
	6-10	298.6 (26)	44.85 (1)	317 (36)
Arabic gum	2-10	174.1 (40)	32.74 (9)	150.7 (5)
	4-10	46.3 (5)	31.91 (3)	106.3 (6)
	6-10	-	45.97 (47)	202.6 (26)
Colophony	2-10	22.8 (23)	75.55 (28)	195.8 (34)
	4-10	33.1 (28)	136.48 (16)	486.2 (33)
	6-10	-	148 (12)	586.3 (4)

Based on the outcomes presented modifications in the manufacture process are proposed to improve the quality of the samples. In terms of composition, mechanical strength exhibits linear dependence on applied resin. As a result in the following characterization process, the compositions will be reduced to two, representing the highest and lowest resin content.

The first suggestion is to concentrate the resins for the purpose of minimizing moisture within the mold. The designation "C" denotes a reduced water-to-gum arabic ratio during formation. Initially, the ratio is 2g of water per 1g of arabic gum; however, in these instances, a 3:2 ratio is adopted. This proportion was applied to two different cases, 2-10 and 6-10. In both cases, the dimensions expansion of the specimens decreased during curing. This effect was more pronounced in the 6-10 case, where a greater reduction in water was achieved, resulting in improved stability. In the case of colophony, the concentration ratio was adjusted to 1:1 instead of the initial 2g of acetone for every 1g of colophony.

For specimens featuring higher resin content, superior results are attained by enhancing the maximum resistance strength, Table 3.7. These results follow the trend that higher proportions of binder yield better mechanical properties. Conversely, in cases of lower resin content, the outcome remains comparable due to the diminished water usage (and subsequently less reduction in overall water quantity) as well as the heightened challenge in blending the two components with a smaller amount.

Based on the garnered findings, it is recommended to utilize concentrated resin exclusively in the scenario of the 6-10 composition. This adaptation results in a reduction of 30 g of water within the bending specimens.

Table 3.7: Tensile and bending test results with concentrated arabic gum

Binder	**Composition**	**Tensile strength (kPa) (C.V.)**	**Bending young modulus (MPa) (C.V.)**	**Bending strength (kPa) (C.V.)**
Arabic gum	2-10	174.1 (40)	32.74 (9)	150.7 (5)
	2-10 C	109.2 (9)	34.01 (3)	219.5 (12)
	6-10	-	45.97 (47)	202.6 (26)
	6-10 C	397.3	23.01 (8)	339.9 (5)

A further recommendation is to prolong the duration of pressure application within the mold. The label "5h MTS" indicates that the sample remained in the steel mold under pressure for 5 hours at 5 MPa and was then left to air dry for 1 week. This approach aims to mitigate sample expansion by allowing more time for resin curing, resulting in higher strength upon specimen demolding.

"1h oven" signifies that following a 5-minute pressure application, the mold was introduced into a 120°C oven for 1 hour. Similar to the previous modification, this method aims to expedite sample drying, thereby reducing the curing time while the specimen remains in the mold.

Samples were also fabricated using smaller-sized hemp shivs, Figure 3.37. Using smaller hemp particles can enhance properties [171]; however, the hemp stalk used were already within the described range. To obtain the finer hemp stalk, the sample was filtered through a sieve, resulting in a median particle size of 5 mm compared to the initial 10 mm medium lenght. That modification is labeled as "Thin".

Figure 3.37: Hemp stalk thin size

Table 3.8 show the results of the different fabrication methods proposed.

Unfortunately, the achieved outcomes are not favorable, mainly because the 5-hour duration within the mold proves inadequate for the complete resin curing process. After removing the specimen from the mold, it was observed that the humidity inside the mold was substantial. Moreover, during the week of curing outside the mold at ambient conditions, the dimensions increased significantly. This phenomenon occurred because the binder began to cure inside the mold, causing the hemp to absorb the remaining water, as the mold did not allow moisture to escape.

Figure 3.38: Sample with pressure applied in the mold

Endeavors to expedite the curing process through elevated temperatures fail to enhance specimen outcomes, as this approach leads to the development of internal cracks caused by the swift removal of water, as illustrated in Figure 3.39.

Figure 3.39: Crack formed during drying in the oven at 120°C

The filtration of hemp stalk in order to use smaller particles does not contribute to improved results, Figure 3.40. Consequently, these attempted modifications are disregarded.

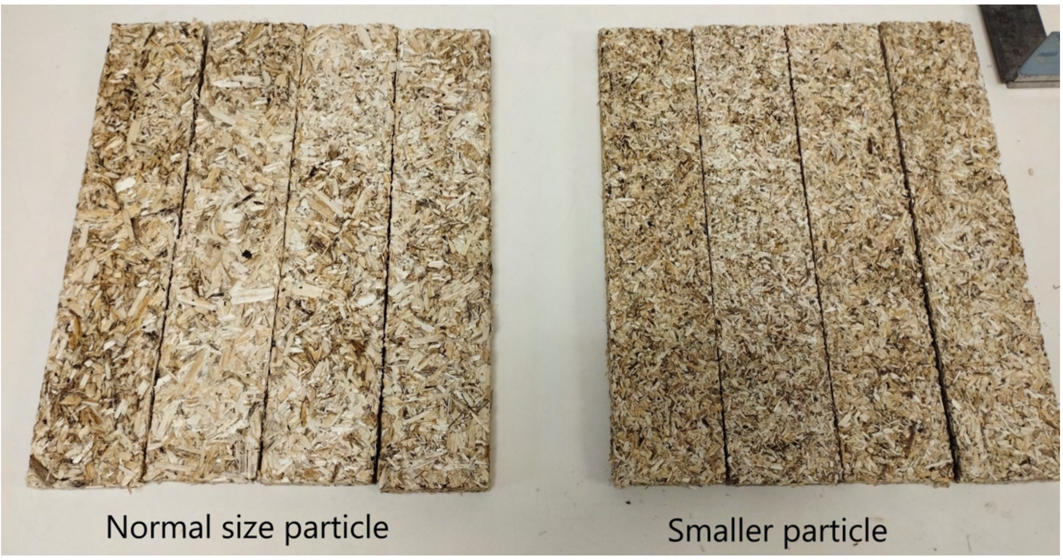


Figure 3.40: Comparison of sample with different size particle

The hemp stalk were soaked for 24 hours to prevent further moisture absorption from the binder and to prevent volume changes. Nevertheless, utilizing damp hemp stalks hinders the effective curing of the arabic gum, leading to the instability of the specimens.

Table 3.8: Results of tensile and bending tests varying the manufacturing methods

Binder	**Composition**	**Tensile strength (kPa), C.V. (%)**	**Bending young modulus (MPa), C.V. (%)**	**Bending strength (MPa), C.V. (%)**
Arabic gum	2-10	174.1 (40)	32.74 (9)	0.15 (5)
	2-10 5h MTS	10.9 (-)	12.55 (-)	0.11 (-)
	6-10 C	1802.3 (45)	222.79 (1)	0.74 (1)
	6-10 C 1h oven	913 (15)	398.46 (5)	4.09 (18)
	6-10 thin	327.4 (28)	133.15 (28)	30.58 (26)

Based on these results, it was proposed to keep a sample in the mold for 1 week without applying pressure, using bleeding paper and absorbent paper similar to those used in composite material fabrication with infusion methods, Figure 3.41.

The wood mold and absorbent paper reduces the moisture absorbed by the hemp, and since the specimen remains in the mold during curing, it maintains its dimensions. With these two processes, the dimensions improved from 15 mm to 10-11 mm, increasing the compactness of the samples, Figure 3.42, resulting in improved properties, as shown in Table 3.9. These process were extrapolated to the other initially considered resins.

Figure 3.41: Wooden mold with bleeding and absorbent paper (left) and obtained specimens (right)

Figure 3.42: Decrease of thickness expansion

Furthermore, for the arabic gum and colophony composites, a two-step curing process is proposed using the wooden mold. This is because colophony, which utilized acetone, does not exhibit as many moisture-related issues. However, it's important to note that the optimal curing time varies for each resin due to their distinct curing mechanisms, as indicated in Table 3.9. For arabic gum, water absorption by the wood and paper components creates a high-moisture environment over an extended period, which can decrease the strength of arabic gum, and the hemp stalk may also absorb moisture, reducing its mechanical properties. In contrast, colophony undergoes elimination upon contact with air. In the mold, the evaporation process is slower, resulting in a more resilient sample with one week of curing time.

In this scenario, the most favorable outcomes were achieved, with values approaching those of commercial materials (tensile strenght: 0.4 MPa & bending strenght: 11 MPa [72]). Consequently, this modification will be implemented for the other binders as well.

In the initial stages, corn starch was utilized as a binding agent. However, the material exhibited instability due to the significant amount of water required for the binder. Through refining the manufacturing process, which involved employing a wooden mold with a drainage system and allowing for one week of drying time with absorbent paper, the mixture became sufficiently stable for mechanical testing. The results obtained from using food-grade starch, coupled with this optimized manufacturing approach, are detailed in Table 3.9. Nevertheless, these outcomes fell short of those achieved with the two vegetable resins applied. In the case of 1-10, the samples remained unstable due to the high water content in the mixture, even with the improved fabrication method.

Table 3.9: Results of tensile and bending tests varying the curing times in the mold with absorbent paper

Binder	**Composition**	**Tensile strength (MPa), C.V. (%)**	**Bending young modulus (MPa), C.V. (%)**	**Bending strength (MPa), C.V. (%)**
Arabic gum	6-10 C 1 week	1.80 (45)	222.79 (1)	0.74 (1)
	6-10 C 1 day	2.16 (18)	747.19 (29)	5.25 (24)
Colophony	6-10 C 1 week	0.61 (13)	594.54 (40)	2.77 (34)
	6-10 C 1 day	0.26 (65)	558.28 (8)	1.33 (15)
Corn starch	0.5-10 1 day	0.31 (56)	172.70 (24)	1.05 (19)
	1-10 1 day	0.03 (-)	21.31 (-)	0.12 (-)

In Table 3.10, a comparison is presented between all the resins employed in the study and the top-performing scenarios. The fabrication process involved applying pressure for 5 minutes, followed by placing the sample in a wooden mold with absorbent paper for curing. For white glue, bioepoxy, and colophony, the curing period was 1 week, while for arabic gum and corn starch, it was 1 day. For the case of colophony and arabic gum the concentrated resin was used.

Analyzing these findings reveals that, when compared to commercial benchmarks, the attained tensile strength improve the best instances by a factor of up to 5. Nonetheless, the bending strength remains within the range of 50% of that seen in commercial boards (tensile strenght: 0.4 MPa & bending strenght: 11 MPa [72]) for the most successful cases, arabic gum 6-10.

Table 3.10: Results of tensile and bending tests for the best cases

Binder	**Composition**	**Tensile strength (MPa), C.V. (%)**	**Bending young modulus (MPa), C.V. (%)**	**Bending strength (MPa), C.V. (%)**
White glue	6-10	1.06 (49)	194.20 (22)	2.22 (23)
Bioepoxy	6-10	1.73 (42)	338.73 (2)	5.15 (24)
Arabic gum	2-10	0.37 (32)	54.70 (3)	0.25 (35)
	6-10	2.16 (18)	747.19 (29)	5.25 (24)
Colophony	2-10	0.12 (63)	67.72 (9)	0.35 (30)
	6-10	0.61 (13)	594.54 (40)	2.77 (34)
Corn starch	0.5-10	0.31 (56)	172.70 (24)	1.05 (19)

Regarding the attained outcomes, the current material's suitability as a particle board is limited due to its inadequate bending mechanical characteristics. Considering the best case scenario with arabic gum, the bending Young's modulus is 50% lower than commercial materials (1800 MPa [72]). Additionally, the amount of binder used is higher, with a 5-10% ratio in commercial chipboards [72].

This deficiency would necessitate structural reinforcement, despite its superior tensile properties in comparison to commercial counterparts. The same limitation is relevant for parquet applications.

Within the coffered ceiling, the attained mechanical strength values meet regulatory standards, and its bending strength is two times greater than EPS (150 Kpa) in the case of 2-10 with colophony (350 kPa). This suggests the potential utility of this material as a disposable formwork block in construction endeavors. Colophony is chosen for its hydrophobic properties, aiming to shield the hemp stalk from the alkaline environment created by concrete. It is necessary to protect the material during the initial week to prevent degradation of its mechanical properties. In the case of arabic gum, since it is soluble in water, the material may not be resistant enough to withstand the weight of the concrete until it is fully cured.

After the concrete is poured and undergoes curing, these blocks become an integral component of the structure. It's crucial to verify material compatibility and ensure that the green material remains stable for at least 7 days, allowing the concrete curing process to take place without degradation of the mechanical properties of the bio composite or the concrete.

3.3 Selection of the most suitable application

In this section, the application to be undertaken during the thesis development will be determined. This decision will be based on the assessment of different characteristics, with assigned weights reflecting their significance. Each application will be evaluated against these attributes, and the ultimate choice will be the one that garners the highest aggregate score.

3.3.1 Methodology

Below, the primary decision factors and their respective selection ratios are outlined:

- Key characteristic 45%: This factor pertains to either insulation or mechanical properties, depending on their relevance to the application

  A score of 10 is awarded if the material's properties match or exceed those of commercial counterparts. The score decreases incrementally based on the percentage of performance deviation from commercial standards. This aspect holds significant importance since, despite the material's primary benefit being its eco-friendly nature, its potential for commercialization remains substantial even without necessitating a direct enhancement of existing materials.

- Green Factor 40%: The subsequent key consideration is the material's environmental friendliness. To evaluate this, scoring will be segmented into several subcategories:
  - Eco-Friendly Materials 10%: This factor assesses the overall environmental friendliness of the proposed composite solution. The score will be based on the materials employed. Each material will be assigned a score based on its eco-friendliness, which will then be weighted by the proportion of the proposed solution's weight that each material constitutes
  - Recyclability 10%: This category considers whether the raw materials are recyclable or if the final material could potentially be recycled
  - Circular economy 10%: This factor evaluates the final material's potential to contribute to a circular economy.
  - Environmental Impact 10%: This segment evaluates the environmental impact of the materials used, including factors such as CO_2 emissions during production, the use of environmentally harmful materials, and more.
- Potential Limitations 15%: Assigning a score of 10 when no drawbacks are evident, this category addresses gaps within the current literature that might impede the development of the final material. It also encompasses aspects that may have been initially overlooked but are essential for the material's effective utilization

3.3.2 Results and selected application

Subsequently, the scoring for each application will be elaborated upon, culminating in a comprehensive summary table, Table 3.11, showcasing the scores attributed to each application:

- Thermal insulation panel
  - Key characteristic: 10

    The thermal conductivity is lower than that of the proposed commercial materials, resulting in improved thermal insulation.
  - Eco-friendly material: 10

    A score of 9 is assigned due to 70% of the material being composed of hemp, which receives a score of 10, and 30% recycled cardboard, also receiving a score of 10.
  - Recyclability: 10

    Similarly to the previous point, the two materials composing the insulator are recyclable and could be reused.
  - Circular economy: 5

The primary material consists of waste material combined with recycled cardboard. However, once integrated into the building, its expected lifecycle aligns with that of the structure, indicating that due to its extended use, the product isn't anticipated to significantly promote a circular economy.

- Environmental impact: 10

  The main material is a blend of waste material and recycled cardboard, resulting in the use of waste resources for a more sustainable impact.

- Potential limitations: 6

  The literature lacks studies on the compatibility of cardboard with hemp. Additionally, considering that vegetal-based coatings may not adhere to all construction regulations, such as fire resistance, there's a possibility of needing to introduce a non-green coating. Moreover, adding a coating might require the use of environmentally unfriendly materials, potentially resulting in negative environmental implications or non-recyclable materials. However, it's important to note that the negative impact occurs prior to the addition of a coating, not during the feasibility assessment, hence a score of 6 is assigned.

- Acoustic insulation panel
  - Key characteristic: 8

    The material exhibits acoustic absorption that is 0.20-0.25 dB/dB lower than that of rock wool.

  - Eco-friendly material: 10

    The green factor section is scored similarly to the thermal case because the material and its usage over its lifecycle are comparable.

  - Recyclability: 10
  - Circular economy: 5
  - Environmental impact: 10
  - Potential limitations: 6
- Chipboard
  - Key characteristic: 4

    The primary property of the particleboard pertains to its bending strength. In the case of use arabic gum with a composition of 6-10 the beding young modulus is 747.10 MPa which in this instance is 40% lower compared to commercial materials (1800 MPa).

  - Eco-friendly material: 9

A score of 9 is assigned as the material is composed of 60% hemp and 40% arabic gum. However, adding a non-environmentally friendly reinforcement might be necessary to enhance the properties if this application is selected. A hemp mesh with epoxy resin could be used in that case.

- Recyclability: 5

  Similarly to the previous cases, a score of 5 is assigned due to the non-recycled nature of arabic gum, which comprises 40% of the material. An additional point is deducted for potential modifications that might be required.

- Circular economy: 8

  In this case, the lifespan is shorter, and the proposed material would be 100% recyclable except for the reinforcement part, thus receiving a score of 8.

- Environmental impact: 9

  The environmental impact for this type of material is minimal, thus receiving a score of 9, similar to the insulation panels, considering the potential impact of the reinforcement.

- Potential limitations: 5

  In this case, several limitations could affect the application, such as a high percentage of non-sustainable materials in the reinforcement or concerns about the water resistance of the assembly.

• Coffer ceiling

  - Key characteristic: 10

    The bending strength exhibited by the samples with colophony, even in the case of lower resin content (350 kPa), surpasses that of EPS (150 kPa).

  - Eco-friendly material: 9

    A score of 9 is assigned because 82% of the material is composed of hemp, which receives a score of 10, while 18% is the binder. In this case, the binder must be colophony due to its resistance to moisture ambient. The colophony is mixed with acetone in a 1:1 ratio, resulting in a score of 5.

  - Recyclability:8

    A score of 8 is assigned because 82% of the material is composed of hemp, which receives a score of 10, while 18% is the binder. In this case, the binder must be colophony due to its resistance to moisture ambient. Colophony could not be obtained from recyclable sources.

  - Circular economy: 0

    The material will have amalgamated with the concrete and undergone significant degradation over time. Consequently, it cannot be repurposed as raw material for other applications, it receives a score of 0.

- Environmental impact: 9

  Similarly to the previous application, the environmental impact of this material will be positive. However, it receives one point less due to the use of acetone.

- Potential limitations: 8

  There are potential limitations that might impede its application, primarily related to compatibility during concrete curing and fire safety regulations. Nonetheless, if these challenges arise, they can be addressed by adding a surface coating to safeguard the material. While this may reduce sustainability to some extent, it wouldn't be a definitive barrier. Hence, a score of 8 is chosen.

• Parquet

  - Key characteristic: 0

    The primary characteristics considered are hardness and incompressibility. However, these properties have not been tested. A rating of 0 is assigned because the test specimens used demonstrate deformation under low loads, suggesting that their use as ground material may not be viable.

  - Eco-friendly material: 9

    The green factor section is scored similarly to the chipboard case because the material and its usage over its lifecycle are comparable.

  - Recyclability: 5

  - Circular economy: 8

  - Environmental impact: 9

  - Potential limitations: 0

    It received a score of 0 due to its potential infeasibility for use in this application because of its lower mechanical properties.

In Table 3.11, the scores for each of the applications are presented. Based on the obtained data, thermal insulation is the selected application. However, both types of insulation will be retained in the research due to the similarity in the test specimens and procedures.

Table 3.11: Application score table

	Thermal	**Acoustic**	**Chipboard**	**Coffer ceiling**	**Parquet**
Key characteristic (45%)	**10**	8	4	**10**	0
Eco-friendly material (10%)	**10**	**10**	**9**	**9**	**9**
Recyclability (10%)	**10**	**10**	5	8	5
Circular economy (10%)	5	5	**8**	0	**8**
Envirommental impact (10%)	**10**	**10**	**9**	**9**	**9**
Potential limitations (15%)	6	6	5	**8**	0
Score	**8.9**	8	5.65	8.3	3.1

This newly developed bio-composite material, incorporating hemp stalk and eco-friendly binders, demonstrates potential as a sustainable alternative to conventional inorganic insulating materials in the construction industry. However, to ensure its practical viability and long-term stability, further research is required. It is crucial to verify that the material retains its structural integrity and insulating properties over extended periods, thus ensuring its efficacy and durability in real-world applications.

Continued investigation and development of this material will play a pivotal role in its success as a sustainable building material. The subsequent sections of this chapter will delve into the specific properties of hemp stalk, the evaluation of potential green binders, and the characterization of the resulting bio-composite material to shed light on its suitability for the selected applications. By comprehensively understanding and optimizing this material, we aim to contribute to the advancement of eco-friendly solutions in the construction industry.

This material could be more versatile, not only serving as an insulating panel but also as a suitable substitute for EPS in various applications. For instance, in concrete sandwich materials or even coffer ceiling. The addition of EPS in concrete sandwitch composite materials results in a lighter, impact-resistant, thermally efficient, and cost-effective material, with an extended lifespan [172, 173]. This opens up more potential applications for the proposed material. However, this research will primarily focus on its individual application as an insulating panel.

3.4 Conclusions

Insulation panel and the coffer ceiling application exhibit significant potential. The thesis will primarily investigate the insulation panel, while the coffer ceiling is suggested for future research.

- At least 30% by weight of short fiber material based on cellulose is needed to bind the stalk particles
- The proposed insulating material is a hemp-based material composed of the following materials: 70% by weight of stalk and 30% by weight of recycled paper fiber (cardboard or newspaper)

  Based on the results obtained with eucalyptus fiber, a proposal is to replace it with recycled cardboard fiber, which is more durable. Since reducing eucalyptus fiber improves insulation properties, and the aim is to maximize hemp stalk usage, the initial ratios remain unchanged. However, further investigation is required to validate this relationship. If the hemp stalk does not interact well with cardboard fiber, one potential solution could involve using recycled newspaper fiber or alternative eucalyptus fibers for the application.

- It necessary to use a coating to protect the material against moisture

  Additionally, there is a suggestion to explore the use of plant-based coatings on the material's surface for protection against environmental and external factors. Gum arabic provides protection against moisture. Colophony protects against direct contact with water for a long period of time.

- Once the insulating material is damaged, it can be disintegrated with water and reused as a raw material, obtaining great recycling and increasing the useful life of the materials and a circular economy
- The insulating properties obtained are comparable to the commercial material

  The final material obtained has a high coefficient of acoustic insulation 0.8, although it is 0.1-0.2 dB/dB lower than commercial materials. In the case of thermal insulation, the proposed material is between 5-10% higher than conventional commercial synthetic materials. Obtaining a material with thermal conductivity coefficient of 0.035 W/m K.

- The proposed insulating material meets the mechanical resistance requirements necessary to be used as an insulating panel in construction. Reaching compressive and shear strength values higher than EPS
- The application of coffer ceiling is feasible using colophony as binder.

  While the mechanical properties of green resins may not match those of commercial materials, they are adequate for cofer ceiling application. Hence, colophony is recommended due to its water and moisture resistance. For particleboard application, structural reinforcement is necessary to improve bending strength. However, using hemp stalk with the proposed solutions for parquet is not feasible.

- For the manufacture of these composite materials, it is advisable to use manufacturing processes and materials with low water content

The mechanical properties are greatly influenced by moisture during the curing process, as the hemp stalk absorb water, leading to swelling and a reduction in mechanical properties. Therefore, employing techniques to minimize moisture content in the material during curing is recommended.

Comparing the requirements for insulating panels and the properties offered by synthetic materials with the properties of hemp stalk, this study proposes a solution for creating a fully plant-based material using hemp stalk as the primary component. In the context of this specific application, mechanical strength is not of utmost importance; rather, the material needs to meet minimum criteria to ensure stability during transportation and installation, preventing breakage, material detachment, or fissures. Consequently, a 100% plant-based material is defined for this purpose.

Following the selection of the most promising application from the ones studied, the subsequent section will conduct a more in-depth analysis to confirm the properties using recycled cardboard, a more eco-friendly material. Furthermore, a thorough examination of the properties will be carried out, considering aspects such as material durability and compliance with regulatory requirements, including fire and moisture resistance.

Chapter 4

Insulating panels

In this chapter, the development process for the selected application, which involves creating insulating panels using hemp and recycled cardboard, will be outlined. The procedures encompass both mechanical and chemical processes aimed at improving the material composition. Subsequently, the material will undergo comprehensive testing for evaluation, including a durability test to assess its real-world feasibility.

This study presents a unique approach, focusing on the development of a sustainable and environmentally friendly material primarily using hemp stalk while incorporating recycled cardboard fiber as a binding agent. The following sections will explain the material improvement process, emphasizing enhancements in insulating properties, durability, and compliance with regulatory standards such as fire resistance.

To facilitate comparisons, samples with cardboard without any modifications will be manufactured and compared with the eucalyptus samples from the previous chapter. Then, various modifications will be introduced to assess their impact on material properties. The chapter will commence with an explanation of the theoretical basis for these modifications, followed by details on the materials used and the new fabrication method. Finally, characterization tests will be conducted, and once the optimal mix is selected, durability tests will be performed.

4.1 Improving the insulating panel

In this section, an explanation of the three proposed modifications to the material mix will be provided.

4.1.1 Material & fabrication method

The utilization of recycled paper and cardboard for cellulose-based insulation materials has been previously proposed in prior research [110]. Nevertheless, this thesis introduces an innovative approach by incorporating hemp stalk along with recycled cardboard fibers for the production of insulating panels [1]. Recycled cardboard possesses several key attributes that render it an excellent choice for these applications:

- Recycled cardboard can be easily disintegrated to achieve desired bonding and shape through cellulose crosslinking
- No chemical products are required to process the recycled fiber.
- Recycled cardboard exhibits remarkable insulating properties.
- It is a lightweight material with a hollow microstructure, depending on the manufacturing method employed.

Furthermore, in the previous chapter, non-homogeneity was observed due to the application of vacuum. Therefore, in this case, the manufacturing process will not necessitate the use of a vacuum. However, for the initial samples, vacuum will still be employed for comparative purposes. Nevertheless, considering that the cardboard fibers are longer than the eucalyptus fibers, the use of vacuum should have a lower impact on the cardboard samples.

4.1.2 Green coating

The primary challenges associated with this material involve its susceptibility to water, moisture, and fire. To address these issues, green surface coatings using colophony and arabic gum are proposed as protective measures.

By applying a pine resin coating to the fibers, a hydrophobic material is created, providing increased stability against environmental factors. The addition of colophony in bamboo raises the contact angle from 60º to 93º and reduce the porosity [144], and when the contact angle exceeds 90º, the material is considered hydrophobic. It is important to note that the addition of colophony may adversely affect the fire resistance properties of the material. On the other hand, arabic gum shows promising results as a coating in terms of humidity and fire resistance. Arabic gum can protect against moisture by creating a barrier that prevents water from penetrating the surface of the coated material. This is due to the film-forming properties of arabic gum, which enable it to create a continuous and uniform coating over the surface of the material. This coating can reduce the permeability of the material to water vapor and prevent the absorption of moisture, which can cause degradation, spoilage, or loss of functionality in the product. However, it cannot be used in direct contact with water, as it is soluble and may cause the protective coating to be removed when in contact with water [145]. The gum arabic membrane, as evidenced by a water contact angle of 43º, does not produce a hydrophobic coating [174]. However, it is worth noting that gum arabic contains active enzymes such as peroxidases, oxidases, and pectinases, which possess antimicrobial and antifungal properties [175]. Overall, the coating not only protect the material but also contributes to stabilizing its properties.

4.1.3 Crosslinking agent

Recycled cardboard has remarkable insulating properties and easy disintegration, which facilitates the desired bonding and shaping process. Cellulose bonds are employed as the bonding method. Hence, a comprehensive investigation has been conducted to enhance the bonding process between the two materials and explore various methods for improving their properties and overall durability. Several types of acids have been found to enhance the bonding between cellulose molecules, including:

- Citric acid (CA)
- Tannic acid (TA)
- Butyltetracarboxylic acid (BTCA)
- Ethylenediaminetetraacetic acid (EDTA)
- Polycarboxylic acid (PCA)
- Hyaluronate acid (HA)

Numerous studies in the literature have utilized these acids to enhance cellulose bonding in various materials. A biodegradable hydrogel can be developed from cellulose using EDTA to enhance water retention in soil and reduce the required fertilizer amount [176]. A biodegradable hydrogel can be manufacture using HA as a crosslinker and a carboamide solution as a reticulant, resulting in improved bonding between cellulose and enhanced mechanical properties of the hydrogel [177]. Adding BTCA in cotton fibers improve the efficiency of cellulose reticulation, which can be determined by chemical or mechanical processes [178]. In addition to using acids to enhance cellulose reticulation, catalysts can also be incorporated. For instance, in the case of tannic acid, the inclusion of cellulose nanocrystals improves the mechanical and physicochemical properties of chitosan films [179].

Among these, PCA and CA are noteworthy reticulants for paper fibers. Moreover, CA can be obtained from certain fruit wich could be suitable for a gree solution. CA have demonstrated their ability to enhance the mechanical properties of corrugated cardboard boxes and improve their resistance to moisture, thereby extending their lifespan [180]. Furthermore, citric acid augments the moisture barrier properties in other materials, such as starch. Acting as a crosslinker between starch and cellulose, it forms a viscoelastic foam, thereby enhancing thermal, mechanical, and water absorption properties [181].

Acid citric has been identified as potential additives to enhance specific properties of paper products. This improvement is attributed to the reduction of OH groups, which enhances the resistance to moisture of the cellulose bonding [182, 183]. In order for the acid to be effective, the curing temperature must exceed the acid's melting point, leading to thermolysis decomposition and the production of di- and tricarboxylic acids (UAs), among others. The percentage of bonding improves as the curing temperature increases. As shown in Figure 4.1, when the temperature reaches 170°C or higher, the percentage of unbound citric acid is less than

10%. Moreover, the curing temperature impacts the curing time of the sample, reducing it, and influences the final color. Higher temperatures result in a darker color due to increased CA decomposition. However, there are no significant differences in compression strength after moisture loss [180].

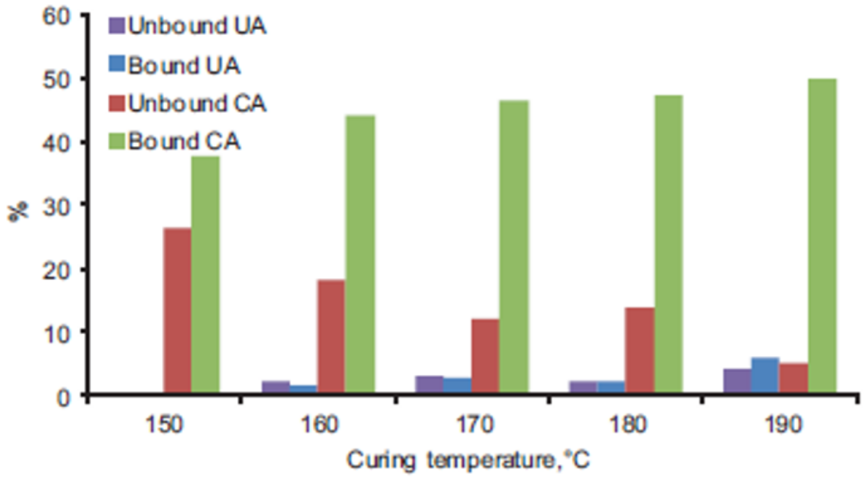


Figure 4.1: Effect of curing temperature on the percentage of citric acid (CA) crosslinks and their thermal decomposition [180]

Apart from using acid, it is advisable to consider the use of a catalyst or ensure that the mixture falls within an optimal pH range. In the case of PCA, NaH_2PO_2 can serve as a catalyst, or if not using a catalyst, the pH should be maintained between 2.0 and 3.5 [180].

Other studies have employed NaH_2PO_2 as a catalyst between citric acid and polyethylene glycol. However, better results are observed without the use of a catalyst as it leads to the formation of macromolecules that cannot be leached out during the filtration process through the filter paper [184].

Regarding citric acid (CA), there is no evidence indicating the necessity for a catalyst. As for pH, there seems to be no specific optimal range for cellulose bonding, suggesting that CA acts as a self-catalyst (see Figure 4.2). An additional advantage of CA over the other presented acids is its lower melting point, enabling curing at lower temperatures.

Figure 4.3 illustrates how the addition of CA increases the lifespan of cardboard boxes up to 4 times. However, when the catalyst is added, the lifespan decreases as the catalyst makes the box more susceptible to ambient humidity [180]. The lifespan of CA-treated cardboard diminishes over time during storage, as the percentage of cellulose-CA bonds reduces with time, as confirmed by the FTIR-ATR spectrum analysis [180].

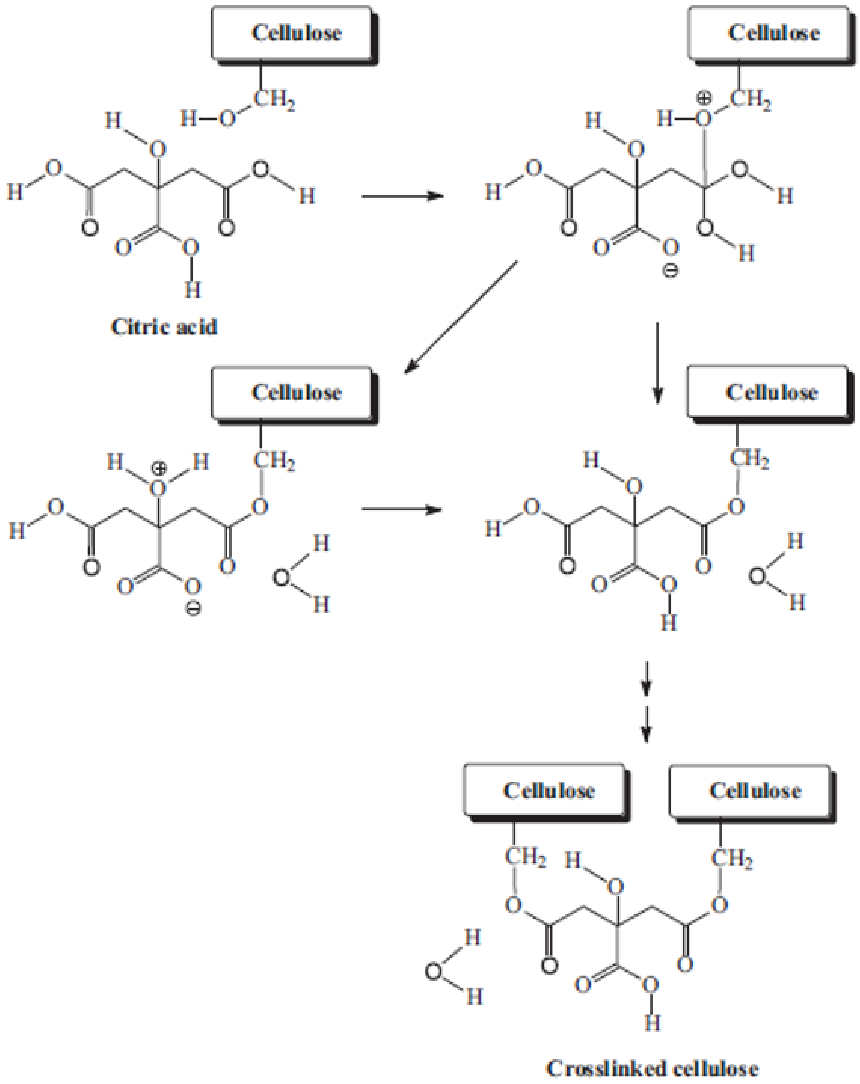


Figure 4.2: Crosslinking of cellulose molecule with citric acid [180]

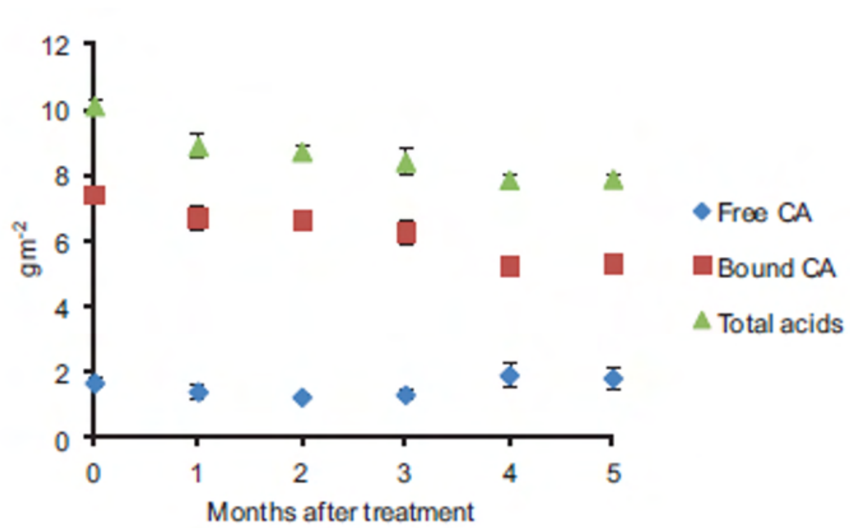


Figure 4.3: Crosslinking of cellulose molecule with citric acid [180]

In conclusion, citric acid is a material capable of enhancing cellulose bonds, leading to increased compressive strength, moisture resistance, and lifespan of cellulose-based materials, without the need for a catalyst and at lower curing temperatures compared to other acids. Moreover, CA could be obtained from a green sources. These properties make it an intriguing material for studying cellulose reticulation in proposed insulation panels.

This study presents a novel approach by developing a sustainable and environmentally friendly material primarily using hemp stalk, incorporating recycled cardboard fiber as a binding agent, applying vegetable resins as a surface coating, and utilizing citric acid to enhance the crosslinking of cellulose and improve the material's long-term durability.

4.2 Materials

The following raw materials have been used to develop the materials samples used in the different experimental test:

Hemp stalk

The hemp stalk provided by Planteles Lloveras, a harvesting company. For more information see section 3.1.1.

Eucalyptus pulp

The paper pulp used is a ECF (elementary chlorine free) bleached eucalyptus kraft pulp, which was supplied by the company ENCE. For more information see secction 3.1.1.

Cardboard fiber

Cardboard fiber is known for its higher resistance and longer fibers compared to ecucaliptus fiber [112, 113, 114]. Additionally, it is unbleached, which reduces the need for chemical products in the production of raw materials. Furthermore, the bleaching process in paper fiber can result in a degradation of its mechanical properties. For this study, cardboard obtained from packing boxes was used, and a disintegration process was carried out to obtain the required pulp, Figure 4.4. The disintegration process was carried out using water at a temperature of 70°C and a rotational speed of 1250 rpm for 60 minutes.

The use of recycled material approach aligns with the goal of maximizing resource efficiency and sustainability by repurposing and reusing materials that would otherwise be discarded.

Coating

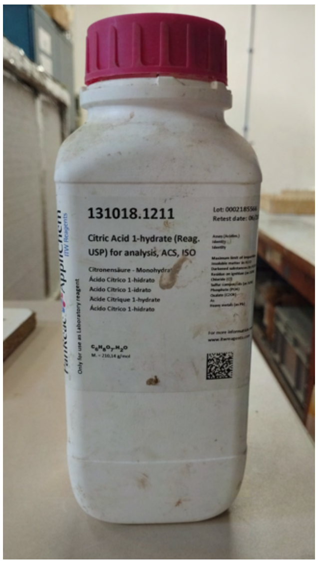
The addition of a superficial vegetal coating is proposed to provide protection against moisture for the interior of the material. To apply the resin as a coating, gum arabic is dissolved in water using a ratio of 2g of gum arabic for every 3g of distilled water at 90°C. On the other hand, colophony is dissolved in acetone with a 1:1 ratio at 50°C. The 2 materials were provided by the company Mezcla Perfecta. For more information see secction 3.2.1.

Citric Acid

Citric acid monohydrate (CA) $C_6H_8O_7 \cdot H_20$ with 99.9% purity and molecular weight of 210.14 g/mol, Figure 4.4, supplied by ALCO company, is employed to activate the hydroxyl groups (OH) present in the cellulose molecules. Moreover, CA can be obtained from certain fruits. The datasheet is provided in appendix B.3

(a) Pulper

(b) Recycled cardboard pulp



(c) Citric acid

(d) Citric acid

Figure 4.4: Materials

4.3 Characterization test of insulating panels

The tests conducted encompassed an examination of the material's acoustic and thermal insulation properties, as well as its mechanical response under compression, shear, and bending. Additionally, an evaluation of its fire resistance and water-related characteristics was performed. Furthermore, the samples underwent microscopy analysis to study their microstructural features of the different samples, and the chemical variations induced by the presence of citric acid were investigated using Fourier Transform Infrared Spectroscopy (FTIR). The conducted tests are detailed in Table 4.1. For the durability test, the insulation and mechanical properties will be reported under various humidity cycles, as indicated in Table 4.2.

4.4 Fabrication method

4.4.1 Fabrication method of samples

The manufacturing process used to produce the proposed material is explained below, taking into account the differences among the samples, Figure 4.5.

Figure 4.5: Hemp stalk-cardboard (left) and hemp stalk-eucalyptus (right)

In the manufacturing process, the initial step involves soaking the stalk for one day, to ensure proper moisture content. This is particularly important because of when it is used dry hemp stalk, as without moistening, it tends to float on the water's surface, impeding its effective integration with the paper fibers. By moistening the hemp stalk, its density increases, which helps in better mixing with the binder and ultimately results in a homogeneous material. The selected fiber, either recycled cardboard or kraft pulp, is disintegrated in distilled water. Then mixed with the previously moistened hemp stalk, maintaining a specific proportion of each sample based on the dry weights of each material. Thorough investigation has been carried out to ascertain the maximum amount of hemp stalk that can be effectively bonded with the fiber, resulting in a composition of 70% of the total mixture. It is important to note that exceeding an 70% hemp stalk content leads to an unstable sample [2].

Table 4.1: Characterization test of insulating materials

Test	Variable	Number of samples
Thermal resistance - ISO 12664	Paper fiber: 2 %wt. of materials: 4 Coating: 3 Crosslinking agent: 2 Curing temperature: 2	56
Acoustic resistance - ISO 10534	Paper fiber: 2 %wt. of materials: 4 Coating: 3 Crosslinking agent: 2 Curing temperature: 2	20
Compression resistance - EN 826	Paper fiber: 2 %wt. of materials: 2 Coating: 3 Crosslinking agent: 2 Curing temperature: 2	30
Shear resistance - EN 12236	Paper fiber: 2 %wt. of materials: 2 Coating: 3 Crosslinking agent: 2 Curing temperature: 2	30
Bending resistance - EN 12236	Paper fiber: 2 %wt. of materials: 2 Coating: 3 Crosslinking agent: 2 Curing temperature: 2	30
Microscopy	%wt. of materials: 1 Coating: 3 Crosslinking agent: 2	6
Moisture Buffer Value - NORDEST project	%wt. of materials: 1 %wt. of materials:1 Coating: 3 Crosslinking agent: 2	18
Water absortion - ISO 29767	%wt. of materials: 1 Coating: 3 Time: 2	6
FTIR	%wt. of materials: 1 Crosslinking agent: 2	3
Fire resistance - EN 13501	%wt. of materials: 1 Coating: 3 Crosslinking agent: 2	5

Table 4.2: Durability test of insulating materials

Test	Variable	Number of samples
Thermal resistance - ISO 12664	Paper fiber: 1 %wt. of materials: 1 Coating: 3 Crosslinking agent: 2 Durability Cycle: 4 Curing temperature: 1	48
Acoustic resistance - ISO 10534	Paper fiber: 1 %wt. of materials: 1 Coating: 3 Crosslinking agent: 2 Durability Cycle: 4 Curing temperature: 1	16
Compression resistance - EN 826	Paper fiber: 1 %wt. of materials: 1 Coating: 3 Crosslinking agent: 2 Durability Cycle: 4 Curing temperature: 1	48
Shear resistance - EN 12236	Paper fiber: 1 %wt. of materials: 1 Coating: 3 Crosslinking agent: 2 Durability Cycle: 4 Curing temperature: 1	48
Bending resistance - EN 12236	Paper fiber: 1 %wt. of materials: 1 Coating: 3 Crosslinking agent: 2 Durability Cycle: 4 Curing temperature: 1	48

To ensure a uniform mixture of the two materials, a substantial amount of decalcified water is necessary. It is recommended to maintain a 5% consistency based on the dry material, which translates to using 1 liter of water for every 50 g of dry 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 [180]. The water used in this process can be reused for subsequent samples, promoting water conservation and minimizing resource consumption. However, a careful control of the amounts of recovered components is necessary to maintain the desired proportions of the sample. Overall, this method supports sustainable manufacturing practices while ensuring the integrity and consistency of the final product.

When the removal of excess water from the mixture is accomplished using two different methods, depending on the specific sample being processed. The first method involves the use of a laboratory sheet former following the Rapid-Köthen technique. The mixture is introduced into the former, and vacuum pressure is applied to the bottom area for 10 minutes. Cylindrical specimens with a diameter of 20 cm are obtained, and to achieve a 5 cm thickness, 180 g of dry material is required. The second method involves removing the excess water by allowing it to drip through a mesh due to gravity. Subsequently, the sample is left exposed to the air for a period of 2 hours, in this case, a test sample measuring 35x25x3 cm is produced using 300 g of dry material. Obtaining a theoretical dry density of 115 kg/m^3 in both cases.

After forming the sample, it is placed in an oven to ensure thorough drying. Once the sample is completely dry, a coating, if necessary, is applied to the surface using a brush. The coating is applied at a rate of 0.3 kg/m^2, ensuring thorough coverage. The sample is then left to cure for 48 hours in the oven conditions.

The samples were stored in a specially controlled room with a temperature of 23°C and a humidity level of 50%. This climate-controlled environment is carefully maintained to ensure the stability of moisture within the samples until the corresponding tests are conducted. By controlling the temperature and humidity conditions, any potential fluctuations that could impact the samples' moisture content and overall performance are minimized, thereby maintaining the integrity and consistency of the samples for accurate testing and analysis. Despite maintaining controlled environmental conditions in the room, the application of the coating introduces excess moisture to the samples and impedes the complete transmission of moisture from the ambient environment. As a consequence, a noticeable discrepancy in interior humidity arises. The variation in moisture content can have an impact on the material properties. The test samples were prepared by cutting them with a saw, considering the geometry specified in the standard.

Table 4.3: Nomenclature structure of the samples

Composition (%wt. of binder fiber)	**Fiber**	**Curing temperature**	**Coating**	**Crosslinking**
30				
40			∅	
50	E (Eucalyptus)	L (60 ºC)	C (Colophony)	∅
60	R (Recycled Cardboard)	H (170 ºC)	GA (Gum Arabic)	CA (Citric Acid)
70				

The manufacturing process for the samples involved some variations depending on the specific materials used and the inclusion of citric acid, CA in the nomenclature Table 4.3.

The samples made with eucalyptus fiber and the first cardboard fiber (XXE/R-L-E/C-0), where citric acid was not used, the vacuum water extraction method was employed to remove excess water. Subsequently, the samples were manually mixed and then dried in an oven at a temperature of 60°C for 48 hours.

The samples made with the cardboard fiber that incorporated citric acid (XXR-L-C-0/AC-0/C/GA), a different approach was taken. The drip water extraction method was used to remove excess water from the samples. The drying process involved placing the samples in an oven at a higher temperature of 170°C to activate the citric acid in the mixture. This activation was carried out over a duration of 24h. Additionally, the mixing of the materials was conducted using an electric mixer at a speed of 900 rpm for 3 minutes, ensuring thorough blending and distribution of the components. This adjustment was implemented to standardize the samples, irrespective of their manufacturing date. The cardboard was fully disintegrated on the same day and subsequently stored to maintain consistent properties across all samples. Over time, during storage, the cardboard fibers tend to bind together, making it challenging to blend with the hemp stalk. To address this issue, the introduction of an electric agitator proved effective, as it helped to separate the cardboard fibers and enhance the mixture of the two materials.

4.4.2 Fabrication method of insulating panels

Following the completion of the respective tests, the subsequent phase entailed the fabrication of demonstrator panels to assess the potential for producing larger sizes and to conduct bending tests. The manufacturing process involved introducing a blend of hemp and paper fiber into a mold equipped with a drainage mesh, relying on gravity for water removal, Figure 4.6.

Figure 4.6: Manufacture process of insulating panel

The mold is placed within a drying oven at 60°C for a period of 48 hours. Subsequently, the surface coating is applied, and the resin is cured within the oven. The initial test specimens that were produced include:

- 70% hemp stalk - 30% cardboard fiber with colophony

- 70% hemp stalk - 30% cardboard fiber with arabic gum
- 60% hemp stalk - 40% cardboard fiber with colophony
- 60% hemp stalk - 40% cardboard fiber with arabic gum

These panels are employed to confirm that the results obtained from the small samples are equivalent to the performance of a full-sized panel.

4.5 Microstructure

4.5.1 Methodology

The microstructure analysis of the samples was conducted using an Olympus BH-2 optical microscope, which was equipped with a x4 objective lens, Figure 4.7. This setup enabled to examine and observe the surface coating as well as the internal structure of the samples.

To ensure that the captured images accurately represent each of the samples, the surfaces of various test specimens were examined at both central and lateral positions.

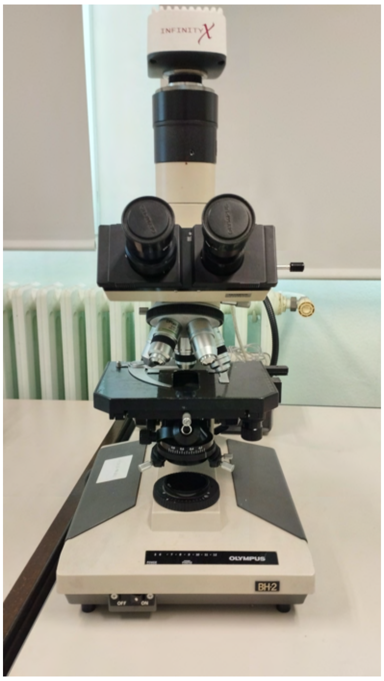


Figure 4.7: Olympus BH-2 optical microscope

4.5.2 Results

Microstructure plays a crucial role in determining the insulating properties of green materials. As such, it is important to understand how the application of coatings on the surface of these materials can impact their microstructure and ultimately affect their insulating properties. This information is of great significance in the development of effective and sustainable insulation materials. Additional figures of the samples are presented in Appendix A.8.

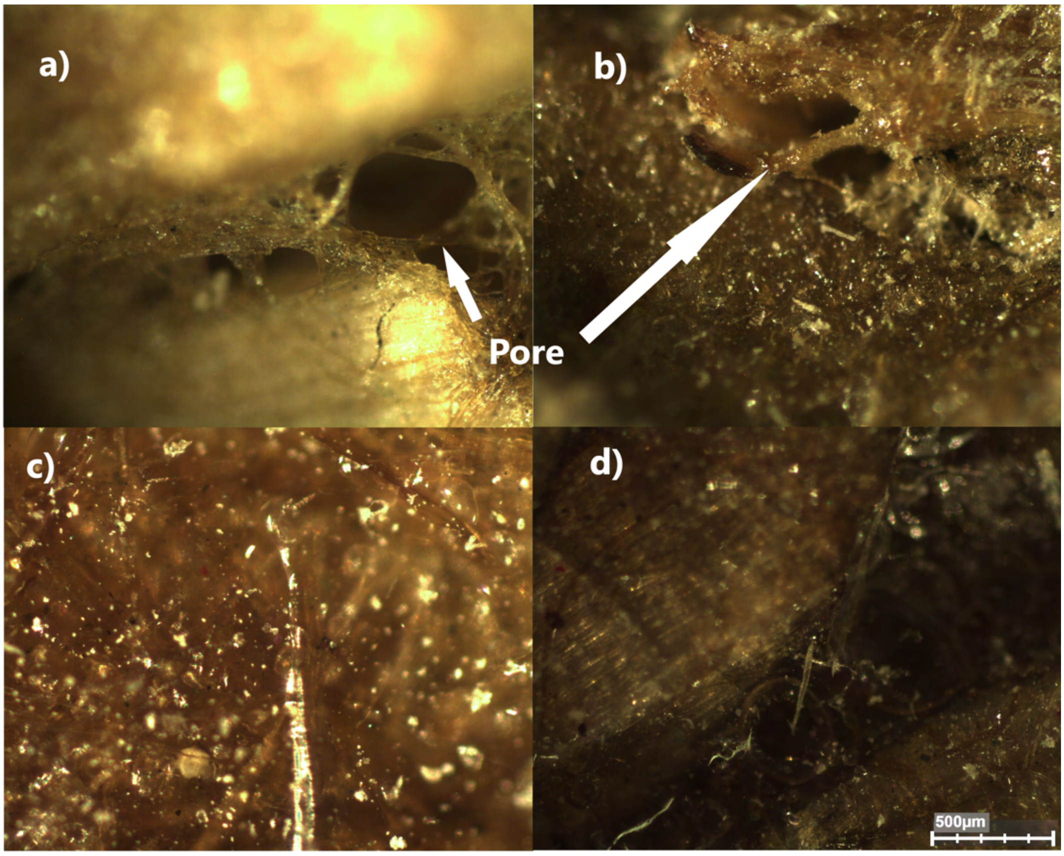


Figure 4.8: Surface of different samples: a) Colophony with acid citric, b) colofony, c) arabic gum with acid citric, d) arabic gum

Both coatings exhibit full surface adherence to the hemp-based material, Figure 4.8. However, there is a noticeable distinction between them. The colophony coating effectively seals the smaller radius pores while leaving the larger ones intact, whereas the gum arabic coating forms a superficial membrane that effectively covers the pores of the material. The size of the membrane pores falls within the range of 38-60 nm [174]. This difference in behavior is crucial to consider when choosing a coating material, as it can significantly impact the material's microstructure and overall insulation properties. In the case of bamboo, the introduction of colophony into the internal pores resulted in a reduction of porosity and total pore volume by 14.42% and 23.81%, respectively [144], the colophony fill the pores creating a barrier that inhibited water diffusion and transmission [185].

The internal microstructure of the material was examined in Figure 4.9, and no discernible differences were detected at the microscopic level between the samples treated with citric acid and those that were not treated. The internal microstructure of the samples exhibits horizontal layers with fibers arranged in a random fashion.

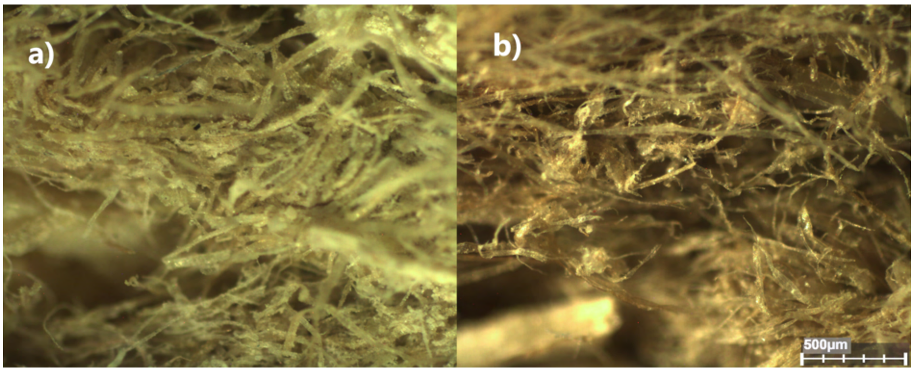


Figure 4.9: Internal microestructure of sample with acid citric (a), and without it (b)

4.6 Moisture Buffer Value

4.6.1 Methodology

The moisture buffer value (MBV) is a measure of a material's ability to regulate moisture levels. It is determined using the method specified in the NORDTEST project [186], which provides a standardized approach for assessing the practical moisture buffer value of materials under dynamic conditions. The MBV quantifies the amount of moisture that a material can absorb and release per unit of open surface area, taking into account daily cyclic variations in relative humidity. By measuring and understanding the moisture buffer value, we can evaluate a material's ability to regulate moisture and its potential impact on indoor environments and occupant comfort.

The method involves subjecting the samples to humidity cycles of 75-33% for 8-16 hours each at a constant temperature of 23°C. The Ineltec climatic chamber was used to conduct the test. During the test, the weight of the samples was monitored until mass stability was achieved. Mass stability was defined as a mass change of less than 5% over three consecutive cycles. For the test, 100x100x30 mm samples were employed, and to ensure their sealing, aluminum coating was applied to five out of the six faces of the sample, Figure 4.10.

Figure 4.10: Ineltec climatic chamber

4.6.2 Results

Based on the categorization from the NORDTEST Project, these materials demonstrate exceptional hygroscopic regulation properties (MBV > 2 g/(m^2 %RH)) [186]. When the moisture buffering capacity of a material results in a comparable average moisture flow rate to that caused by the minimum necessary air exchange, the buffering effect can be regarded as highly impactful for the moisture dynamics within a room. The ideal moisture buffering response should aim for maximum effectiveness and rapidity. In Appendix A.9 the data of the tested samples are presented.

Table 4.4: Moisture buffer results

Sample	**MBV** ($g/m^2\%RH$)	**C.V.** (%)
30R-H-0-0	4.49	7.4
30R-H-0-CA	4.20	3.2
30R-H-C-0	3.78	7.9
30R-H-C-CA	3.95	5.4
30R-H-GA-0	6.01	26.5
30R-H-GA-CA	5.37	6.5

All the samples presented exhibit similar values within a consistent range, indicating a rapid response to fluctuations in ambient humidity, Table 4.4. The addition of citric acid to the samples leads to a reduction in the measured values. However, this effect is minimal and falls within the expected range of variability among the samples.

The presence of a colophony coating on the sample (30R-H-C-0/AC) acts as a protective surface barrier, resulting in a modest 11% decrease in MBV compared to the uncoated samples (30R-H-0-0/AC). In the case of bamboo, previous studies have shown a more significant reduction in water uptake of 24.7% due to the treatment's ability to decrease porosity [144]. The applied coating on the proposed material primarily impacted the surface layer, resulting in a limited reduction in internal porosity. Consequently, the decrease in water uptake was smaller in comparison to previous studies and does not significantly differ from that of the uncoated samples.

In contrast, the incorporation of arabic gum (30R-H-GA-0/AC) amplifies the moisture transfer process by 30%. Although the arabic gum membrane is able to fill all the superficial pores of the material [174], its water-soluble nature results in a higher tendency for moisture absorption on the surface of the coated sample. Consequently, the moisture buffer value is increased, indicating an enhanced capability for regulating moisture within the material.

Regarding the presented material, the findings demonstrate its notable capacity to absorb substantial quantities of ambient humidity, which improves the comfort and quality of life for the residents. However, it is important to note that excessive levels of humidity can have adverse effects on the material. High humidity content weakens the cellulose bonds, leading to a reduction in insulation properties and material strength [187].

4.7 Water absorption by partial immersion

4.7.1 Methodology

In accordance with ISO 29767:2019 [53], a partial immersion experiment was conducted to assess the material's behavior during short periods of direct contact with water. The samples were partially immersed in water for a duration of 10 minutes. This test was conducted to evaluate the material's response to water exposure and assess its performance in terms of water resistance and durability.

4.7.2 Results

To complement the findings, a water immersion test was conducted using three test samples: one without any coating, one coated with colophony, and one coated with gum arabic, Figure 4.11. The results revealed that the uncoated material could withstand immersion for approximately 3 minutes before disintegration, while the gum arabic-coated sample exhibited extended resilience, lasting for approximately 15 minutes. The water contact angle of hemp fiber has been reported to be 77-87º [188], whereas the arabic gum membrane exhibits a contact angle of 43º. Despite the reduction in contact angle observed with the arabic gum coating, it serves as a protective layer that shields the interior of the material until it dissolves in water. This characteristic leads to an increased duration of contact between the material and water, thereby extending the material's resistance to water exposure. Importantly, if the material was removed from water before complete disintegration, it showed the ability to regain its structural integrity upon complete drying. Regarding the colophony coating, after 15 minutes, the sample experienced a 5% increase in weight, and the inner part remained dry, indicating effective moisture absorption by the surface layer and a successful coating. The colophony-coated sample demonstrated exceptional durability due to the hidrophobic properties (raising the water contact angle [189]). The material remained intact when immersed in water for a duration of 90 days without any visible damage; however, some discoloration of the coating was observed, Figure 4.12. This observation may indicate a degradation process happened, highlighting the need for further investigation. Additionally, literature reports indicate that the water uptake of samples treated with colophony increased after 30 days of water immersion [142].

Based on these findings, it can be confirmed that the application of colophony creates a hydrophobic characteristic in the material, as evidenced by its performance in the water immersion test. However, it is important to note that the moisture buffer value (MBV) test showed that the material still allows for moisture transfer with the surrounding environment.

(a) Colophony

(b) Arabic gum

(c) Whitout coating

Figure 4.11: Water absorption test

Figure 4.12: Decoloration of colophony after 90 days

Nevertheless, the disintegration of the material upon contact with water has an advantage. Once the material has deteriorated, broken, or reached the end of its lifespan, it can be easily disintegrated using water to obtain the raw material and be manufactured again. In the case of colophony, a mechanical process can be applied to remove the superficial part, allowing the interior zone to be reused as raw material for manufacturing. On the other hand, gum arabic can be completely reused by separating the coating using water. Although gum arabic is not suitable as a coating in direct contact with water, it still offers protection against moisture, making it suitable for specific applications.

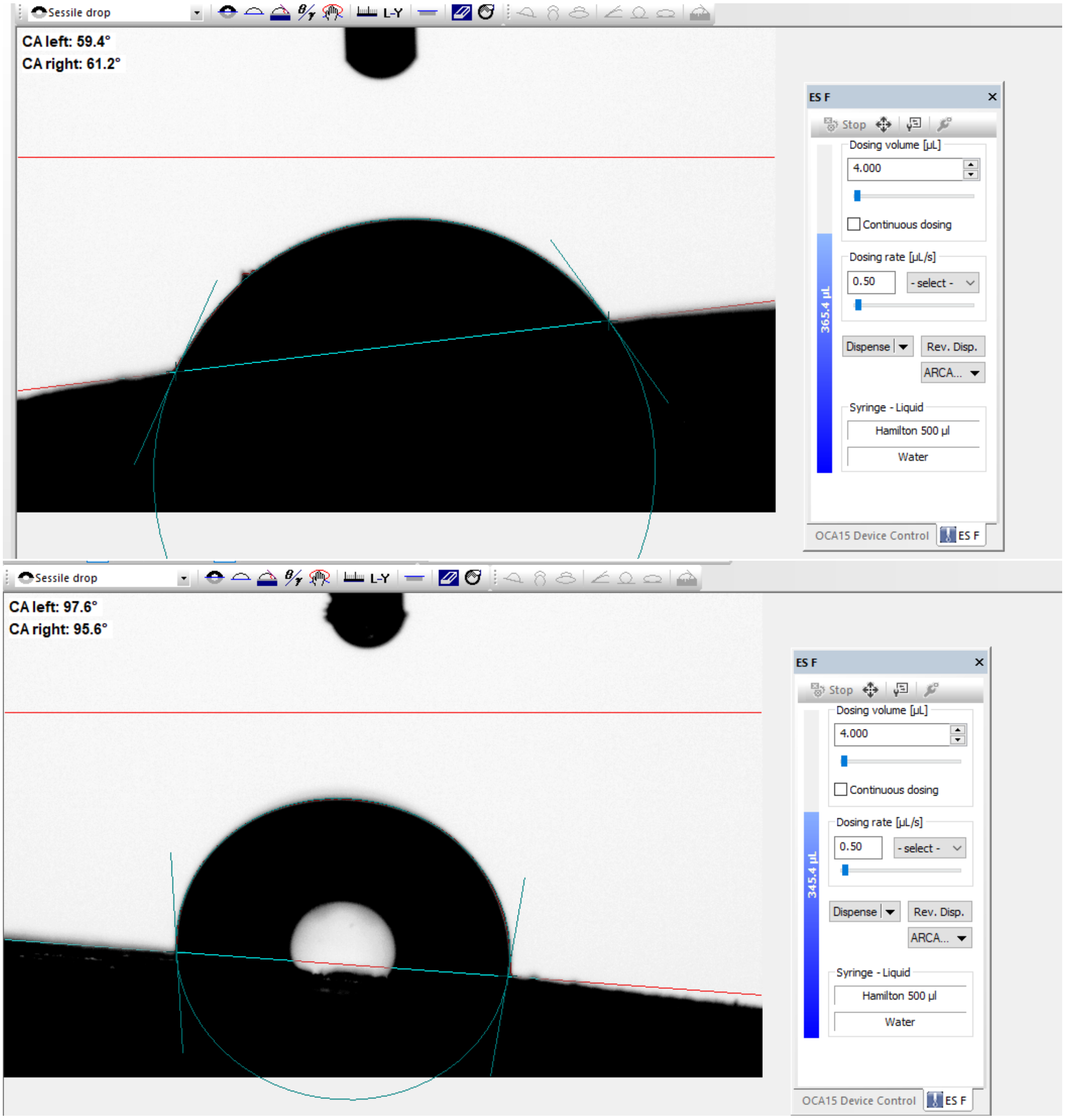


Figure 4.13: Water contact angle: arabic gum (up) and colophony (down)

To confirm the similarity of the water contact angles reported in colophony and arabic gum to the samples used, a water contact angle test was conducted on the raw solid resin. To ensure an appropriate surface for the test, the resin blocks were filed. The test was carried out using an optical contact angle apparatus with SCA 20-U software, employing the sessile method with a 4 μL drop of distilled water dispensed via a Hamilton 500 μL needle, Figure 4.13. A total of 5 repetitions were performed on different surfaces. The results obtained are presented in Table 4.5:

Table 4.5: Water contact angle results

Sample	**Water contact angle (º)**	**C.V. (%)**
C	95	3
GA	72	7

4.8 FTIR-ATR

4.8.1 Methodology

Fourier Transform Infrared–Attenuated Total Reflectance (FTIR-ATR) spectra of a sample treated with acid citric and untreated were collected with a Thermo Scientific Nicolet 6700 FT-IR spectrometer, Figure 4.14. For each sample, absorbance spectra of the scan range 4000-400 cm^{-1} were recorded. To identify the chemical groups present in the samples, spectra of the most significant groups in similar materials were examined based on existing literature. Additionally, to assess the impact of citric acid, the spectra of all three samples were compared, with consideration given to the area of each group. Any alterations in the areas of these groups indicated changes in chemical composition.

Figure 4.14: Sample 80E

4.8.2 Results

According to the literature, specific bands should be considered for analyzing the effect of the acid on the sample. The decrease in intensity of the hydroxyl group (OH) stretching vibrations, indicated by a broad peak at 3336 cm^{-1}, along with the appearance of a shifted peak at 1726 cm^{-1} corresponding to the formation of ester's carbonyl group (C=O), can be regarded as characteristic peaks for crosslinking reaction according to several studies [180, 190, 191], however in Figure 4.15 a reduction in the intensity of these peaks is not observed in the sample treated with CA. The 2900 cm^{-1} band represent the CH stretching [192]. Futhermore, the weakening of the hydrogen bond likely alters the vibrational energy of the entire cellulose chain, thereby influencing the characteristic band at 1159 cm^{-1} [182], as can be observed in Figure 4.16.

Additionally, with the addition of CA, characteristic bands of C–O stretching, which are characteristic of CA, can be observed at 1704 cm^{-1} in the spectra, Figure 4.16. This observation confirms the continued presence of the acid in the material's composition [193]. The appearance of the strip at 1725 cm^{-1} in the 30R-H-0-AC sample provides evidence of the chemical process involving the crosslinking of cardboard with citric acid [192]. However, this peak is relatively inconspicuous due to the limited quantity of cardboard fiber in comparison to the hemp stalk. The presence of a peak at 1373 cm^{-1} indicates CH bending in cellulose [180]. Peaks at 1730 cm^{-1} and 1720 cm^{-1} correspond to esterified carboxyl groups of both CA and UAs, while the absorbance at 1030 cm^{-1} can be attributed to the vibration associated with C–O–H deformation [192].

In summary, the spectra indicate a minimal presence of acid in the sample and a reduction in hydrogen bonds, possibly resulting from a brief reaction between cellulose present in the cardboard and citric acid, followed by acid decomposition producing the increase of esterified carboxyl groups. However, the spectra also reveal that the crosslinking of cellulose has not significantly improved. This may be due to the absence of cellulose reaction in the hemp stalk, suggesting the potential need for a catalytic material to initiate the reaction. Therefore, The spectra suggest that the addition of CA to the samples does not enhance their mechanical properties, indicating that CA does not improve the crosslinking between the cardboard and the hemp stalk. However, it could potentially improve the durability of the samples by reducing the hydrogen bonding within the cellulose chain. In this manner, the durability test will aid in confirming the hypothesis.

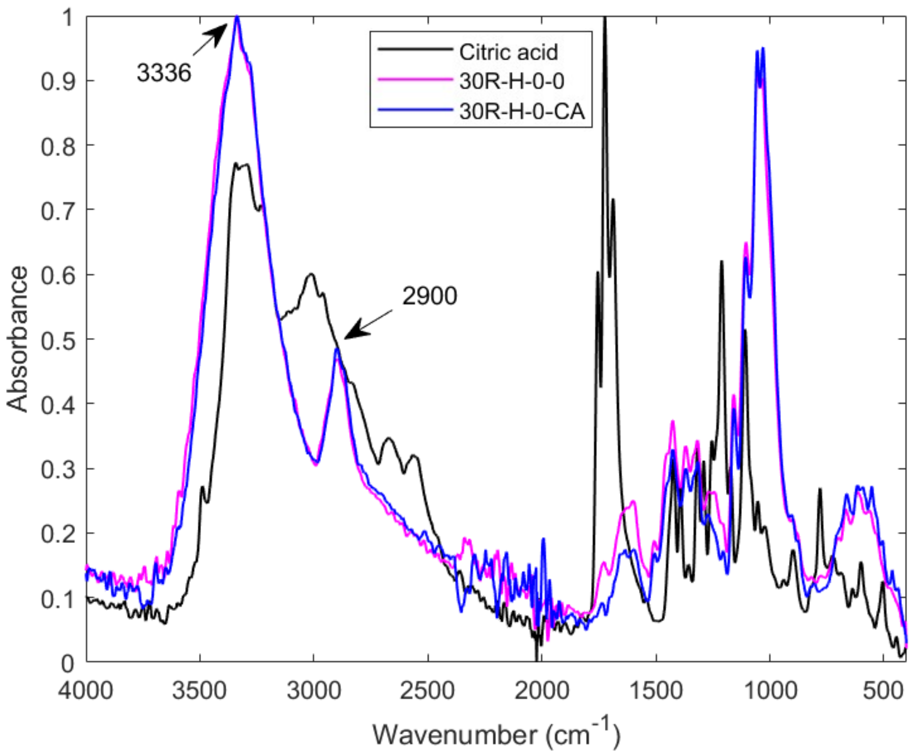


Figure 4.15: Comparison of the spectre of the samples with acid citric

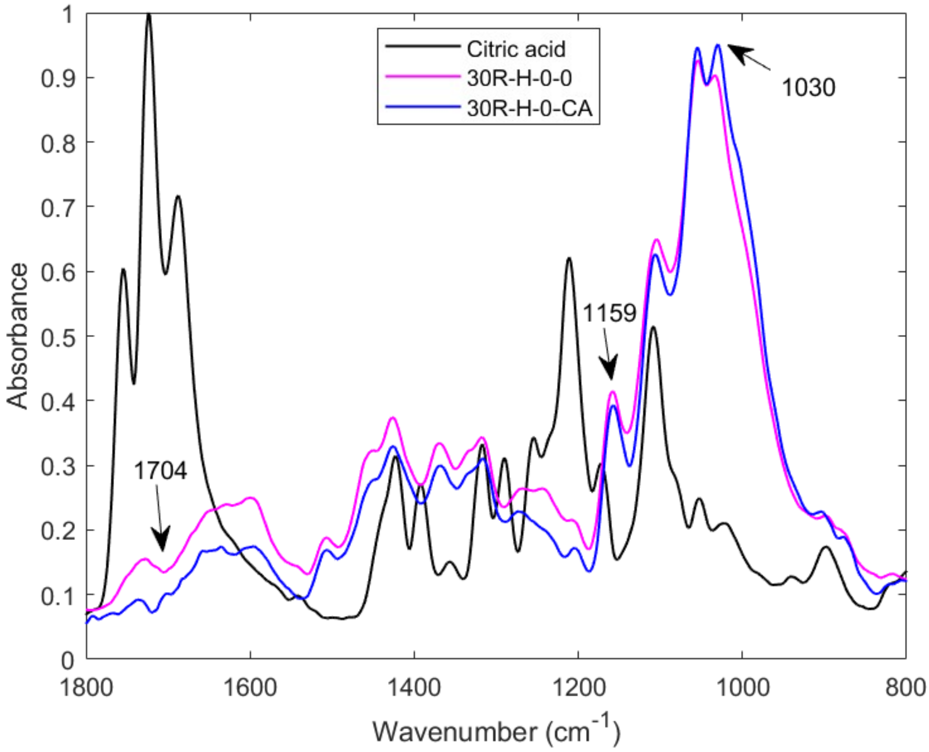


Figure 4.16: Zoom of the spectre of the samples with acid citric

4.9 Acoustic insulation

4.9.1 Methodology

The acoustic properties were assessed using a two-microphone impedance tube, specifically the Brüel & Kjaer type 4206, in accordance with EN 10534 [49]. In the section 3.1.5 more information is detailed. In Appendix A.2, the raw data of the conducted tests are detailed.

4.9.2 Results

To verify the impact of material quantity on the acoustic properties of the mix, the samples in Figure 4.17 can be compared. The comparison of samples with different weights of eucalyptus fiber reveals that higher quantities of hemp stalk lead to improved acoustic insulation properties. This enhancement is attributed to the excellent insulating properties of hemp. The results show that, at lower frequencies, the material exhibits greater absorption than rock wool. However, from 2000 Hz onward, this absorption decreases, although it still remains within an acceptable range (greater than 0.7 dB/dB) for use as acoustic insulation. Upon adding the coating, a noticeable decrease in the acoustic absorption coefficient is observed (0.2-0.4 dB/dB), in samples 30/40E-L-C/GA-0 comparing with 30/40E-L-0-0. This effect can be attributed to the resin curing, it crystallizes on the material's surface, causing sound waves to bounce off the material and also due to a decrease in the material's surface porosity, ultimately reducing the absorption of the waves. Despite the fact that the coating diminishes the acoustic insulation properties of the material, it is essential to add it to prevent degradation caused by humidity in construction applications. Without the protective coating, the bond between the fiber and matrix would eventually break, resulting in loss of structural integrity and acoustic insulation effectiveness.

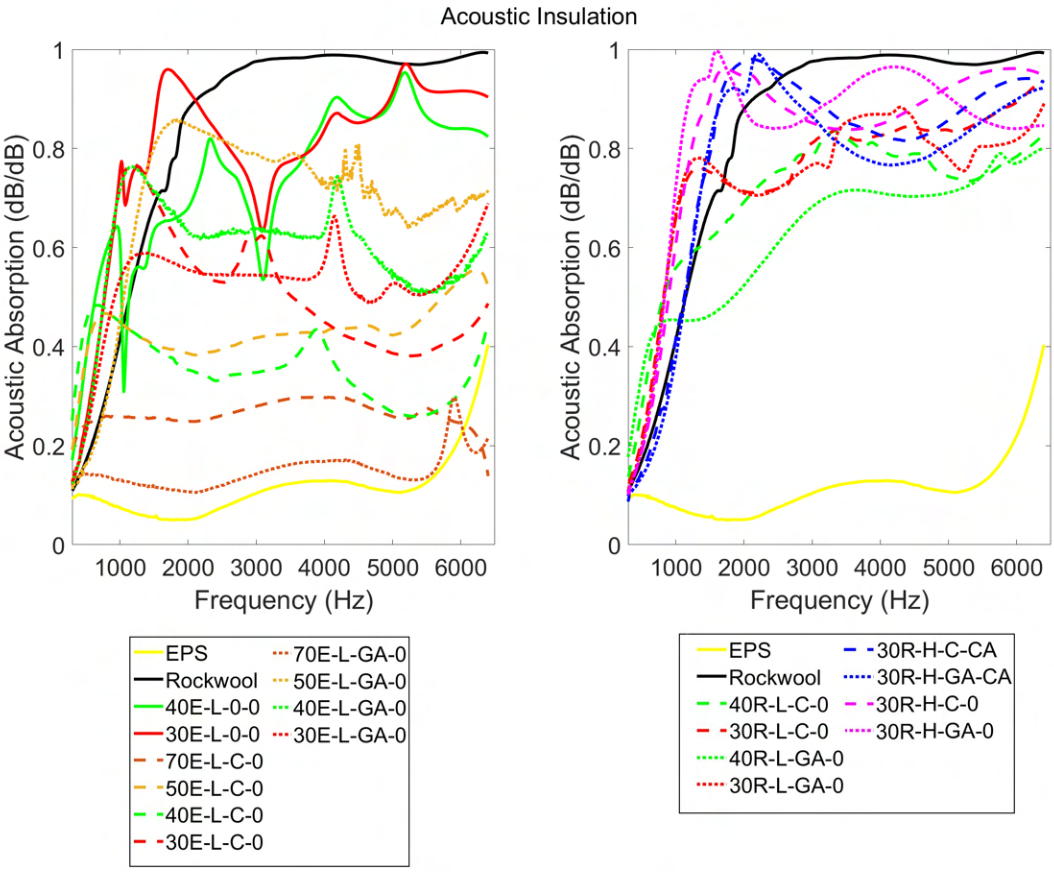


Figure 4.17: Acoustic insulation of stalk-Kraft pulp with coating (left) and hemp stalk-cardboard (right) comparing with other commercial materials

The use of recycled cardboard fiber results in a higher absorption coefficient, as demonstrated in Figure 4.17. Upon comparing the samples made with eucalyptus and cardboard fiber, it is noticeable that the cardboard exhibits superior properties. Although the materials differ, the significant improvement is primarily due to the manufacturing process. In the case of eucalyptus, the internal porosity is lower than the cardboard samples due to the application of vacuum during production. This behaviour is explained in the rigid-framed porous materials model [194]. The non-deforming cavity walls contribute to the increase in acoustic absorption through viscous losses and thermo-elastic damping. This occurs as sound travels through numerous air cavities within the composite material. Increasing the volume of these cavities leads to a higher structure factor and effective porosity, resulting in greater maximum acoustic absorption. This model also explains the improvement in acoustic absorption in cases with more hemp stalk, as observed in the comparison between 70/50/40/30E-L-C/GA. By increasing the quantity of hemp stalk, acoustic absorption also increases due to the interenal porosity of the hemp stalk.

In comparing the samples 30R-L-C/GA-0 and 30R-H-C/GA-0, an increase in acoustic insulation properties was observed (0.2 dB/dB), Figure 4.17. Both samples were produced using the same materials but with different fabrication methods. The best results were achieved with a higher drying temperature and the use of an electric mixing tool for material mixing. The temperature variation during the drying process could potentially influence the crosslinking of fiber, which in turn affects the material's properties. However, it should be noted that the observed improvement in properties appears to be primarily attributed to the mixing process. The mixing process had an impact, as a higher agitation speed led to better distribution of the cardboard fibers. This resulted in greater homogeneity of the samples. In contrast, the manual mixer showed the presence of small unbound cardboard pellets, which negatively affected the properties and resulted in a less uniform distribution of porosity. However, further studies are needed to fully understand the effects of the two manufacturing processes and clarify their impact on the material properties.

Although the acid citric can potentially enhance the crosslinking of the cardboard fiber, which result in modify the size and distribution of internal pores, no significant differences were observed in its use. This suggests that citric acid does not interact significantly with the composite material.

From these results, it is possible to produce an acoustically insulating material using 100% recycled materials, which exhibit competitive acoustic properties (0.85-0.95 dB/dB, 30R-H-C/GA-0/AC) when compared with traditional materials (0.95-0.99 dB/dB rockwool).

Table 4.6: Results of acoustic absorption of vegetal particles with different binders.

Fiber	**Matrix**	**Eco-friendly material**	**Acoustic absorption (α)**	**Pore structure**	**Reference**
Hemp stalk	Cardboard & arabic gum/colophony	YES	0.85-0.95	Hollow microestructure	This study
Hemp stalk	Polycaprolactone	NO	0.6-0.9	Hollow microstructure	[41]
Hemp stalk	Lime	NO	0.6-0.9	Porous material (70-75%)	[59, 87, 125]
Hemp stalk	Porltand & MgO-cement	NO	0.1-0.25	Low porosity	[126]
Hemp stalk	C2-H	NO	0.6-0.8	Porous material	[127]
Hemp stalk	Wheat starch	YES	0.7	Porous material (88-90%)	[84, 128]
Sunflower stalk	Chitosan	YES	0.2	Low porosity	[129]
Sheep wool	Polypropylene	NO	0.3-0.6	Low porosity	[123]

Table 4.6 presents the results of the acoustic absoorption of similar green materials obtained from various studies. It can be observed that materials with lower porosity exhibit lower acoustic insulation properties. On the other hand, materials with higher porosity propose the use of a binder that acts as a solid-state agglomerate, resulting in favorable outcomes due to the combination of high porosity and the inherent properties of hemp. In contrast, the material developed in this study aims to achieve a structure similar to rockwool, where the entire material consists of interconnected fibers. By incorporating recycled cardboard fibers, a similar structure is attained. When combined with the natural porosity of hemp, this material exhibits high-performance acoustic insulation properties.

4.10 Thermal insulation

4.10.1 Methodology

For assessing thermal insulation properties, a square test specimen measuring 10 cm in width and 3 cm in thickness is positioned within a thermal chambe. In the section 3.1.6 more infoormation is detailed. In Appendix A.3, the raw data of the conducted tests are detailed.

4.10.2 Results

The specimens demonstrate superior characteristics compared to commercial materials, despite their individual values being lower (Figure 4.18). This improvement can be attributed to the synergistic effect of combining the two materials, resulting in a new composite material that enhances overall properties. The formation of a new microstructure within the composite material promotes heat attenuation. Thermal insulation materials with low densities are characterized by high porosity, which contributes to their insulation effectiveness. The primary mechanism responsible for insulation is the low thermal conductivity of stagnant gases trapped within the voids of the porous material [131]. Several factors influence thermal conductivity, including the choice of raw materials, temperature, porosity, moisture content, and density. Additional factors such as airflow velocity and material thickness also play a role in determining thermal conductivity [55]. It is important to note that the thermal conductivity of cellulose-based insulation materials is significantly influenced by temperature and moisture content [110, 57]. Therefore, it is necessary to stabilize the material to prevent a decrease in its performance.

By cmparing the samples 40/30E-L-0-0 with 40/30E-L-C/GA-0, can be notice that the addition of a coating further enhances the thermal insulation performance. The optimal combination of materials is achieved with a sample containing 70% stalk, due to the insulating properties of the hemp, Figure 4.18.

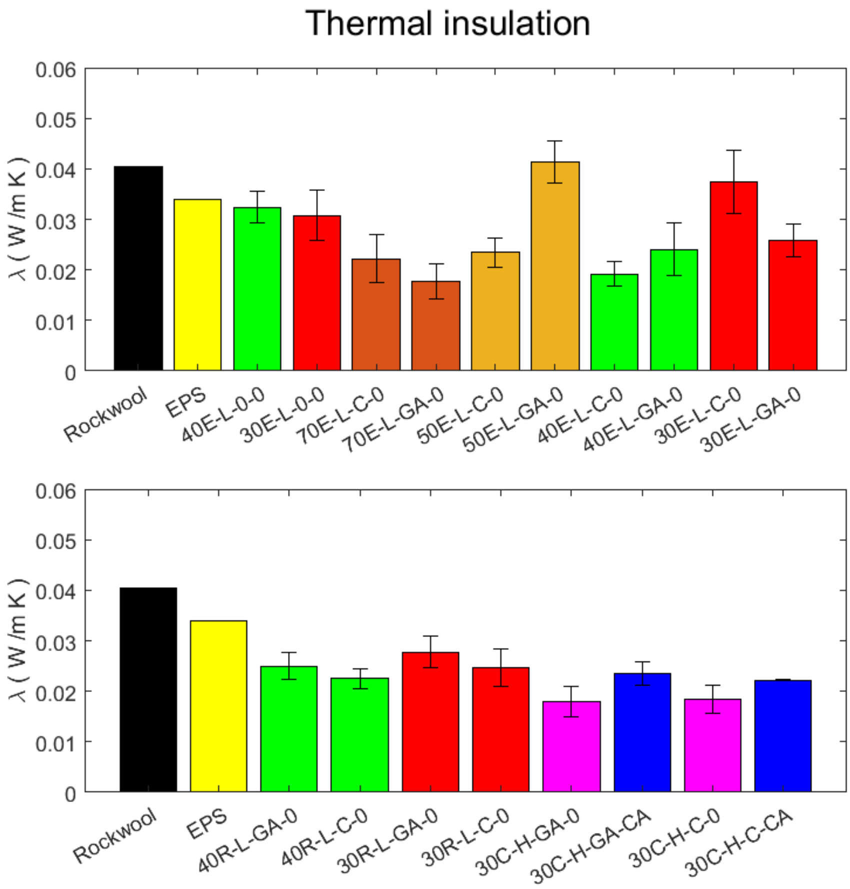


Figure 4.18: Thermal conductivity of stalk-Kraft pulp with coating (up) and hemp stalk-cardboard (down) comparing with other commercial materials

During the material development process, the addition of acid citric did not result in any significant changes when compared to the material produced without it. Although the acid citric can potentially enhance the crosslinking of the cardboard fiber, which result in modify the size and distribution of internal pores, the final density and thermal properties of the hemp and cardboard fiber, remain unchanged, and these factors are more important factors for the insulating properties. As such, the addition of acid citric does not affect the thermal insulation properties of the material, Figure 4.18.

Both coatings exhibit a notable enhancement in thermal insulation properties, with an improvement ranging from 20-25%. However, the arabic gum coating has a higher susceptibility to moisture absorption from the environment, which can negatively impact the thermal insulation performance in a high moisture ambient.

Table 4.7 presents the thermal conductivity values of several materials investigated for their potential use as insulation materials. The results align with the findings related to acoustic properties, indicating that hemp based materials with a high porosity binder and structure exhibit better performance in terms of thermal conductivity. It is noteworthy that in both thermal and acoustic cases, several proposals have been developed using hemp stalk and a green binder that achieve high performance, making them suitable for use as insulation materials.

Table 4.7: Results of thermal absorption of vegetal particles with different binders.

Fiber	Matrix	Eco-friendly material	Thermal conductivity (W/m K)	Reference
Hemp stalk	Cardboard & arabic gum/colophony	YES	0.02-0.03	This study
Hemp stalk	Lime	NO	0.08-0.13	[87, 137, 138]
Hemp stalk	Portland & MgO-cement	NO	0.08-0.115	[126, 125, 139]
Hemp stalk	Wheat starch	YES	0.06-0.07	[84, 128]
Hemp stalk	Cassava starch	YES	0.026	[140]
Hemp stalk	Reactive vegetable protein	YES	0.078	[141]
Sunflower stalk	Chitosan	YES	0.056-0.058	[129]
Corn stalk	Rice huck ashes	YES	0.06-0.08	[132]
Bamboo powder	Bio-glues	YES	0.10-0.20	[142]
Corn stalk	Epoxy	NO	0.10	[68]
Sheep wool	Polypropylene	NO	0.06-0.10	[123]
Flax stalk	Lignin & and biobased epoxy	NO	0.074	[104]

4.11 Fire resistance

4.11.1 Methodology

In order to assess the fire resistance of the material, a fire resistance test was conducted according to the standard EN 13501 & ISO 11925-2:2020 [54, 76]. The specimens, 250x90x30 mm with different coatings, were subjected to direct contact with a flame to evaluate their behavior, Figure 4.19. The purpose of this test was to determine whether the material met the requirements for type E materials, which indicates the ability to withstand the attack of a small flame for a brief period without significant propagation of the flame. Compliance with the type E requirements is essential for the material to be considered suitable for commercial construction applications, as it ensures minimal flame propagation and contributes to overall fire safety. By conducting this test, the material's fire resistance properties were evaluated, providing valuable insights into its performance in fire scenarios and ensuring compliance with the necessary standards.

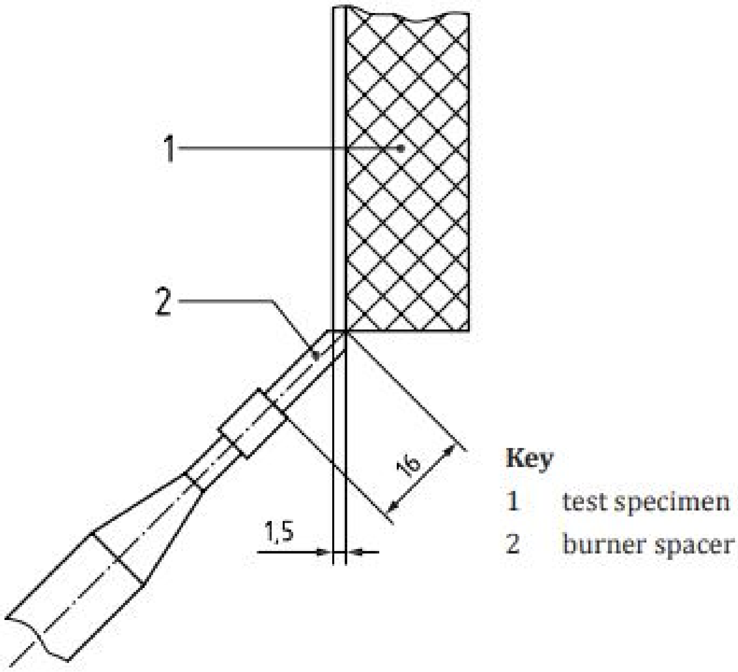


Figure 4.19: Typical flame impingement points for products with thickness greater than 3 mm [76]

Equipment:

- Ignition source: consisting of burner constructed and designed so that it can be used vertically or be tilted at 45° with respect to the vertical axis. The burner shall be mounted onto a horizontal plate so that it moves smoothly forwards and backwards in a horizontal plane along the centreline of the combustion chamber
- Fuel: consisting of commercial propane of 95% minimum purity. In order to obtain flame stability with the burner tilted at 45°, the gas pressure shall be between 10 kPa and 50 kPa
- Support, consisting of a vertical stand to which the specimen holder is attached such that it hangs vertically and exposes its open edge containing the specimen to the burner flame
- Flame-height measuring device: capable of indicating a flame height of 20 mm when located against a fixed point of the burner.
- Timing device

Figure 4.20: Fire test - sample 30R-C-O

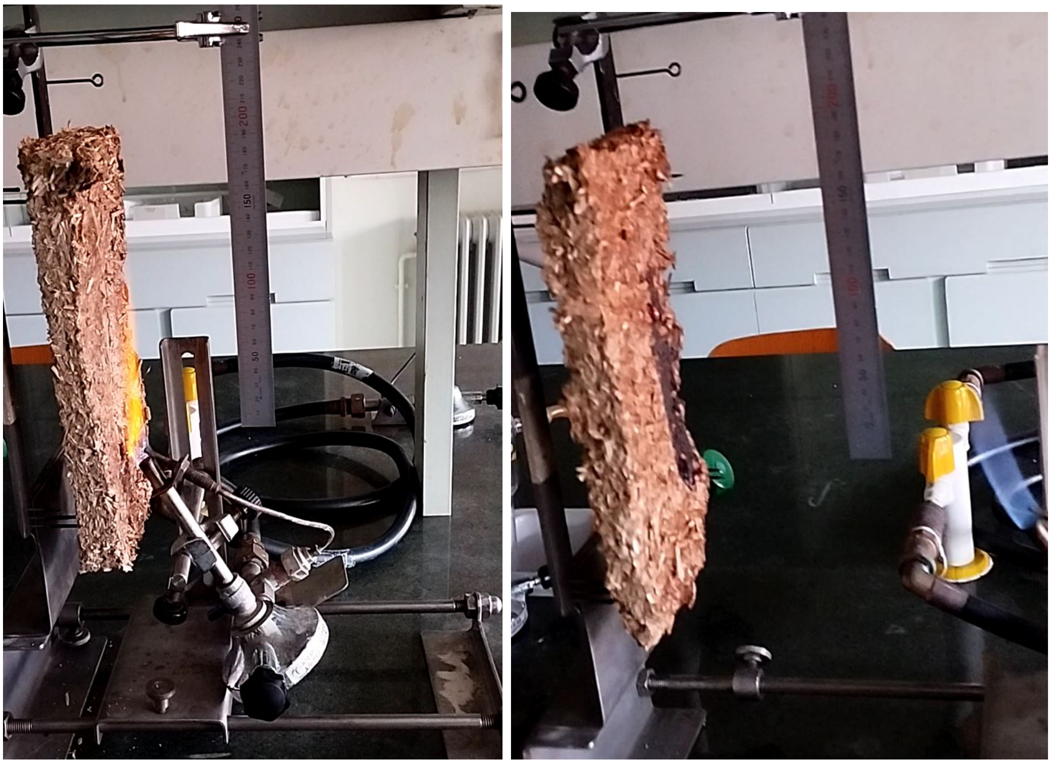


Figure 4.21: Fire test - sample 30R-GA-O

4.11.2 Results

Materials are classified based on their fire resistance according to fire regulations. The minimum required classification for construction materials is Type E. In the test, the material should not ignite, meaning that there should be no presence of a flame for a duration exceeding 3 seconds. Additionally, the flame height (F_s) should not exceed 15 cm from the point of application within a 20 second period.

Table 4.8: Fire resistance

Sample	F_s (cm)	Ignition	Type E
30R-L-0	25	YES	NO
30R-H-C-C-0	> 30	YES	NO
30R-H-C-AC	> 30	YES	NO
30R-H-GA-0	15	NO	YES
30R-H-GA-AC	11	NO	YES

In order to meet the fire resistance regulations, the three coating materials were studied, Table 4.8. Based on the findings from the uncoated specimen (30R-L-O), the material exhibits a low level of fire resistance, it does not meet the necessary criteria for classification in the E category. Consequently, for building applications, the incorporation of a fire-resistant coating is imperative.This coating must provide enhanced fire resistance and also serve to safeguard the interior of the material, effectively preventing the fire from coming into contact with the interior.

Both coatings have achieved the expected results. The colophony coating (30R-H-C-O/AC), Figure 4.20, which is not a good fire-resistant material, and considering that acetone was used for its application, obtained a negative rating in the test. However, when using gum arabic (30R-H-GA-O/AC), Figure 4.21, the results were successful. Only a small and superficial charred area was observed where the flame was applied, while the rest of the material remained intact as the fire did not spread.

4.12 Mechanical tests: compression, shear and bending

In addition to the previous tests, mechanical property tests were conducted to assess the strength and durability of the material. These tests aimed to compare the mechanical properties of the material with those of commercially available materials used in similar applications. The objective was to ensure that the material would withstand its intended lifespan without experiencing any significant breakage or failure. By evaluating its mechanical performance, it can be confirmed that the material is capable of meeting the required standards and specifications for its intended use, providing assurance of its reliability and suitability for the intended application.

4.12.1 Methodology

For the compression test, prismatic specimens with dimensions of 100x100x30 mm were prepared following the EN 826 standard [50]. 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, following ISO 12236 [51]. The bending test was conducted using 250x50x30 mm samples. A 3-point test configuration was employed, with a distance of 200 mm between the supports. The test was performed at a constant speed of 5 mm/min using a MTS insight following the EN 12089 standard [52]. For more details about the methodology see section 3.1.7. In Appendix A.4, A.5 and A.6 the raw data of the conducted tests are detailed.

4.12.2 Results

Bending test in commercial size insulating panels

The bending strength test was conducted using the manufactured panels. This test adhered to EN 12089 standards [52], employing panels with dimensions of 90x42 cm and a thickness of 5 cm, as illustrated in Figure 4.22.

Figure 4.22: Bending test in insulating panels

In Table 4.9, the results are presented, and in all cases, the values surpass the strength of EPS, which is 100 kPa. Consequently, the mechanical stability of the material is confirmed with this data and also corroborate that the result obtained in the small size panels are in the same range of values.

Table 4.9: Bending Strength of Insulating Panels

Sample	Bending test
	Bending strength (kPa) C.V. (%)
40R-L-GA-0	**235.7 (13)**
30R-L-GA-0	**317.7 (16)**
30R-L-C-0	**179.8 (16)**

Mechanical properties of the samples

After confirming the insulation capabilities of the material, mechanical resistance tests were conducted to compare the obtained results with other ongoing developments in insulation materials. Additionally, the results can also be compared with the EPS (in Table 4.10 is highlighting in bold the values that are higher than EPS) so that the material is resistant enough not to be damaged during transport or placement of the panels.

Table 4.10: Compression, shear and bending test results

Sample	Compression test	Shear test	Bending test
	Young Modulus (MPa) C.V. (%)	Shear stress (kPa) C.V. (%)	Bending strength (kPa) C.V. (%)
30E-L-0-0	**0.810 (15)**	15.1 (27)	-
40E-L-0-0	**0.664 (39)**	17.0 (33)	-
40R-L-GA-0	**0.526 (42)**	15.4 (13)	**235.7 (13)**
40R-L-C-0	0.221 (7)	**30.4 (4)**	-
30R-L-GA-0	0.239 (54)	**39.0 (8)**	**317.7 (16)**
30R-L-C-0	**0.541 (43)**	**30.2 (17)**	**179.8 (16)**
30R-H-GA-0	**0.667 (27)**	**41.5 (16)**	**306.4 (16)**
30R-H-GA-CA	**0.676 (14)**	**31.5 (25)**	**230.1 (12)**
30R-H-C-0	**0.960 (28)**	**41.9 (39)**	**431.5 (35)**
30R-H-C-CA	**0.523 (31)**	**37.8 (25)**	**260.8 (10)**

Table 4.11 presents the results from both commercial sources and experimental testing. Nevertheless, for comparing the mechanical properties of the insulating panels, the commercial data will be utilized. It's worth noting that the EPS used in the test had been stored in the laboratory and may have undergone degradation.

Table 4.11: Mechanical properties of EPS

Sample	Compression test Young Modulus (MPa) C.V. (%)	Shear test Shear stress (kPa) C.V. (%)	Bending test Bending strength (kPa) C.V. (%)
Commercial data	0.50	30	150
Experimental results	0.36 (4)	14.9 (13)	110.6 (2)

The first case was a compression test, where the material is compressed between two steel plates, calculating the elastic modulus up to 10% compression strain as indicated by the regulations. EPS has a compressive strength of 0.5 MPa, so in the first case with uncoated Kraft pulp these values are exceeded, Table 4.10. By adding the cardboard fiber, despite being a more resistant fiber, the compressive strength is reduced. When the coating is applied, the surface area of the specimen becomes moistened, causing the bonds of the material to soften. This is more noticeable in compression tests, as if the surface has low resistance, the 10% of strain compression occurs mainly on the surface rather than being evenly distributed throughout the entire thickness. This also leads to an increase in variation between different samples. Comparing the results of the cardboard specimens treated without citric acid (30R-H-C/GA-0), which were cured at a higher temperature (170°C) and the sample with the same composition cured at lower temperature (60°C) (30R-L-C/GA-0), can be observe that they show lower result dispersion and improved mechanical properties. This suggests that the effect of the curing temperature diminishes that effect, as reducing the curing time allows the bonds to have less contact with water, resulting in less weakening. For future samples it will be necessary to study the effect of curing temperature and speed. Furthermore, to minimize the dispersion of the sample, it is recommended to explore the effect of applying pressure during the fabrication process to achieve uniformity. However, it is crucial to avoid applying excessive pressure, as this could compromise the porosity of the material.

In Table 4.10 it is compared the compression young modulus to other proposed insulating material. It can be noted that the mechanical properties of the binder have a important effect on the performance of the composite material.

The shear test involves conducting a static puncture test to compare it with EPS, resulting in a value of 30 kPa. Notably, when considering the fiber length as a significant factor, the utilization of cardboard fiber leads to a substantial increase in shear strength. Eucalyptus fiber exhibits shear strength 50% lower than EPS. However, recycled cardboard demonstrates superior mechanical resistance and possesses longer fibers compared to eucalyptus. Remarkably, the cardboard samples demonstrate a substantial increase in resistance, with fibers that are 2.5 times longer than eucalyptus fiber, resulting in a 40% higher resistance than EPS, Table 4.10. Additionally, the cardboard fibers exhibit a higher lignin content, which enhances the stiffness of the material. In the case of samples with CA, its inclusion does not lead to an improvement in the mechanical properties. These findings align with the results mentioned earlier, indicating that citric acid did not undergo a significant reaction with the hemp stalk.

The results obtained in the bending test follow a similar pattern as the other two tests, Table 4.10, with the notable observation that the range of values obtained is higher than that of EPS (150 kPa) even double it in the best cases (30R-GA/C-O).

In the three mechanical tests conducted, no significant difference was observed between the samples treated with citric acid, with corroboorate that the acid citric do not improve the crosslinking of the cellulose. However, it should be noted that there was a high variance in all the samples, which can be attributed to the significant impact of moisture on the mechanical properties of paper fibers. The mechanical test results demonstrate the enhanced properties of cardboard specimens produced with an electric mixer (30R-GA/C-O) compared to those mixed manually (30R-GA/C). The use of a high-speed mixing tool facilitates better fiber disintegration of the cardboard, leading to a more percentage of bonding among all the materials.

Therefore, a material that meets the mechanical resistance requirements has been obtained using the 70% stalk composition and recycled cardboard, and is also a good thermal and acoustic insulation, using the 2 different coating. The density of the specimens is 130-160 kg/m^3.

The coating plays a crucial role, with improved results seen in cases involving arabic gum. The quantity of hemp is also a significant factor, as increasing its proportion leads to higher bending strength. Based on the three conducted tests, the addition of arabic gum as a coating enhances the biomaterial's mechanical properties. However, an exception is noted in compressive strength.

Nonetheless, for this material, the critical focus is on enhancing its insulating properties, and in this regard, colophony demonstrates superior results. Considering that using colophony aligns with regulatory prerequisites for mechanical strength and offers enhanced resistance to moisture, the configuration comprising 70% hemp, 30% recycled cardboard fiber, and a colophony coating stands out as the most promising choice among the options explored in term of mechanical properties.

Table 4.12 presents a comparison of the compression Young's modulus of the hemp-based materials with other materials. The compression modulus is chosen as a representative and widely studied parameter. The mechanical properties of the composite are significantly influenced by the binder used. In the context of structural applications, it is recommended to incorporate a small amount of hemp stalk into traditional materials like concrete. This addition of hemp particles as aggregates serves multiple purposes, such as reducing the density of the material, enhancing its insulation properties, and increasing its potential for CO_2 storage. However, it should be noted that the mechanical properties of the composite may decrease as a result (up to a 30% reduction when incorporating 8% hemp) [89, 90, 91]. Additionally, the use of cementitious matrices can lead to chemical damage to the hemp stalk, despite the advantages of affordability and adaptability they offer [92].

Table 4.12: Compression young modulus compared with other stalk based materials

Fiber	**Matrix**	**Eco-friendly material**	**Compression Young Modulus (MPa)**	**Reference**
Hemp stalk	Cardboard & arabic gum/colophony	YES	0.67-0.96	This study
Hemp stalk	Wheat starch	YES	0.1-0.15	[84, 128]
Hemp stalk	Reactive vegetable protein	YES	1.1-3.0	[141]
Corn stalk	Epoxy	NO	0.11-0.29	[68, 100]
Hemp stalk	Porltand & MgO-cement	NO	0.4-2.1	[89]
Hemp stalk	Lime	NO	0.3-0.5	[85]

4.13 Durability

In this section, a detailed overview of the durability tests conducted on the developed composite material will be provided. These tests are crucial to ensure the material's stability throughout its expected service life, enabling its use in various applications. For the durability test the sample used were 30R-H-C/GA-CA/0 in order to compare que coating and the acid citric with the best composition obtained. Therefore, in this section, the label will be simplified to the coating, crosslinking agent, and durability time.

4.13.1 Methodology

The durability testing method involves subjecting the material to natural cycles under ambient conditions, replicating the environmental conditions it would encounter during real application use, Figure 4.25. A range of test specimens were prepared and exposed to different durations of ambient conditions. These specimens were then tested to evaluate the potential property degradation.

To conduct this investigation, thermal insulation, acoustic insulation and mechanical properties (compression, shear and bending) were chosen as the key parameters. These properties were evaluated at specific intervals of 0, 3, 6, and 9 months, following the testing procedures outlined in the previous sections (3.1.5, 3.1.6 & 3.1.7.

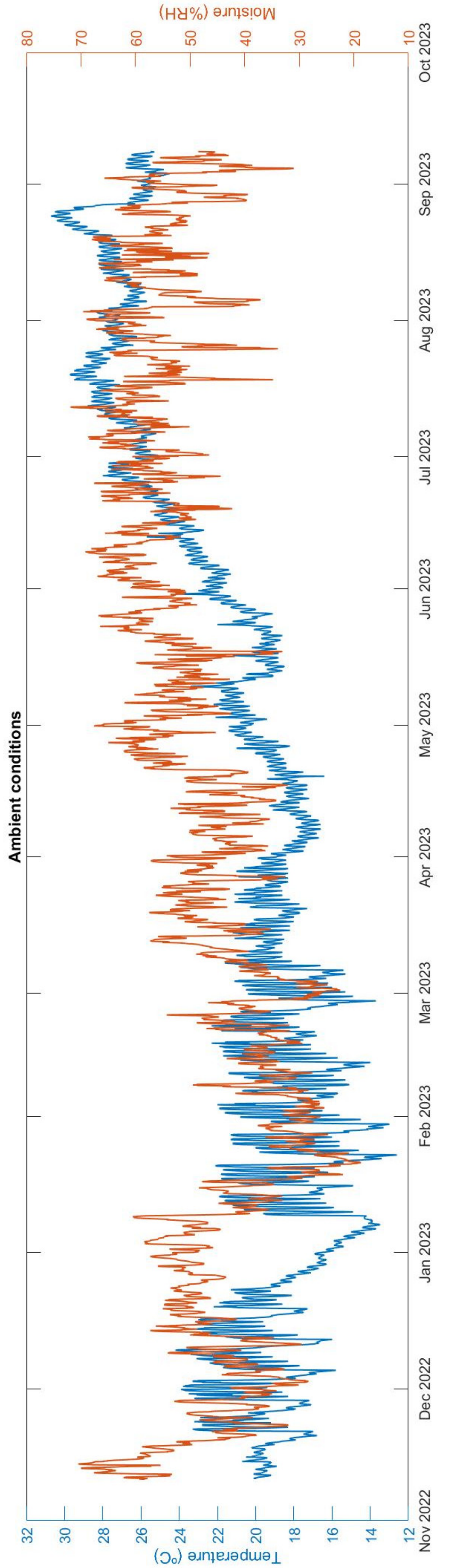


Figure 4.23: Temperature and moisture in the laboratori

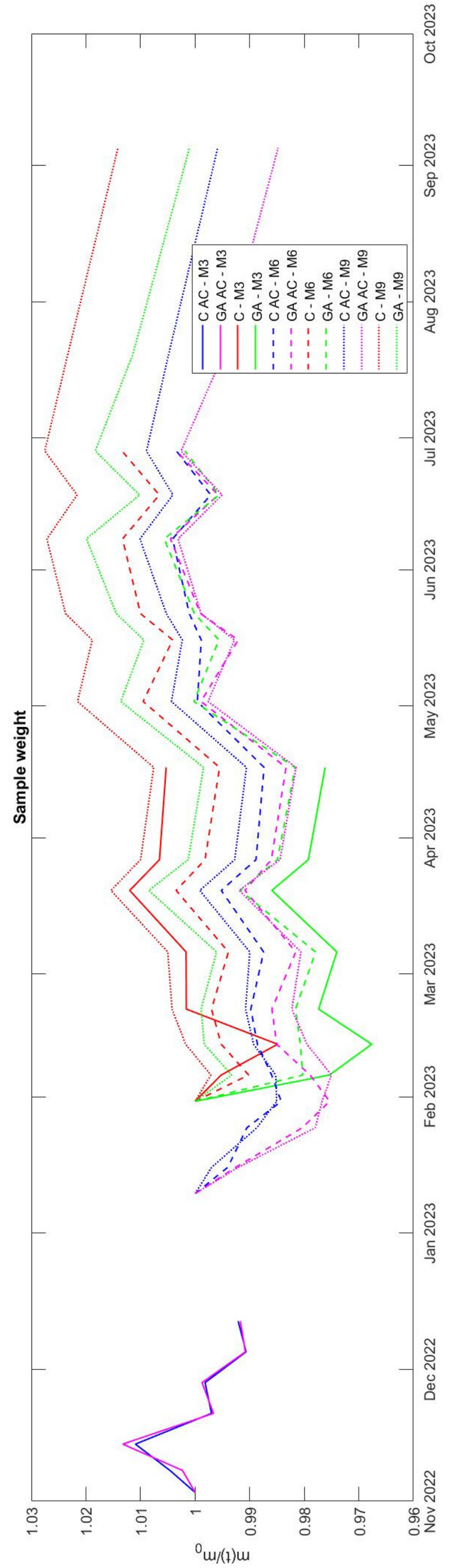


Figure 4.24: Sample weights across durability process

Figure 4.25: Samples storage in laborataory conditions

4.13.2 Results

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, Figure 4.23 and Figure 4.24. The tests began during the winter, the laboratory temperature remained stable due to the heating. This heating was temporarily halted during the weeks coinciding with the New Year holiday. The heating was turned off at the end of winter. 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 arabic gum takes longer to eliminate the water used to form the resin than acetone in colophony. Consequently, its initial weight is higher due to the extra water content that evaporates during the first two weeks. Since the graph is expressed relative to the initial weight of each sample, this difference reflects the initial conditions. However, as the results of the MBV test show, the water diffusion properties are similar, so they follow the same trend, although variability is slightly higher in the case of arabic gum.

These tests also serve to validate the findings from the FTIR analysis, which indicated that the acid does not enhance cellulose crosslinking but may offer a slight improvement in moisture resistance. In other words, degradation over time may be less than in cases where citric acid was used.

Acoustic insulation

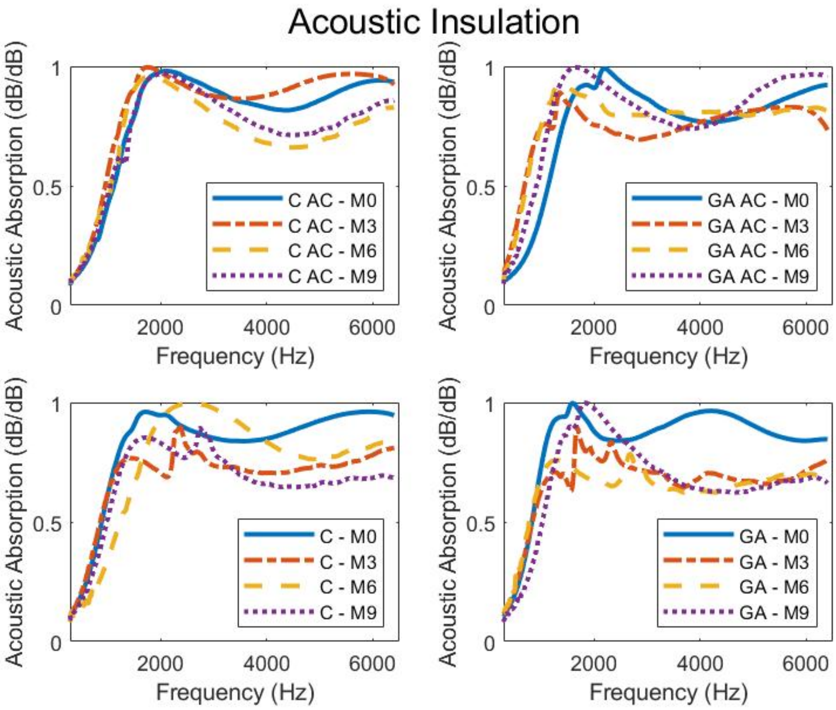


Figure 4.26: Degradation of acoustical insulation properties

From the results shown in Figure 4.26, it can be observed that in the two cases with acid (0-0.2 dB/dB), the degradation of the samples is lower than in 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 degradation corresponds to the reduction of hydrogen bonds in the cellulose molecule, as confirmed in the FTIR test.

Thermal insulation

In the following figures, the coefficient of variation is shown as a percentage on top of each bar.

Based on the results presented in Figure 4.27, it's clear that, similar to the acoustic case, cellulose crosslinking doesn't play a significant role here. However, the ambient humidity level is a critical factor, capable of causing variations of up to 50% in the results. Examining Figure 4.23, it's noticeable that the humidity levels during months 6 and 9 are higher compared to the initial months, leading to a reduction in their thermal properties.

When comparing the results between months 6/9 and months 0/3 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 doesn't enhance thermal properties. It would be beneficial to extend the testing duration to pinpoint when degradation occurs under ambient humidity conditions similar to the initial months, providing insights into potential differences between the samples.

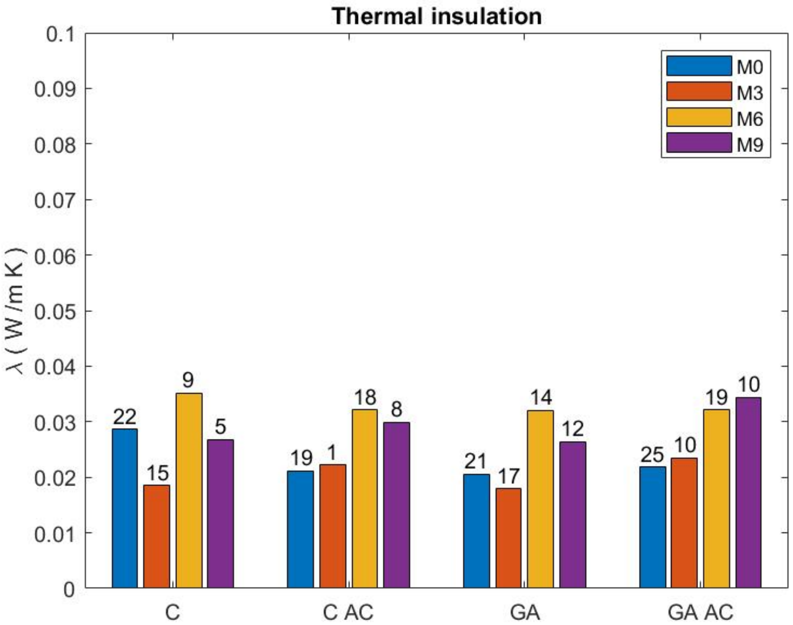


Figure 4.27: Degradation of thermal insulation properties

Mechanical tests: compression, shear and bending

Figure 4.28, Figure 4.29, and Figure 4.30 illustrate the degradation 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 tests were conducted just three days after applying the coating, indicating that it hadn't fully cured yet. Consequently, the samples required more time for proper curing. This lingering moisture within the sample seems to have a more substantial impact on mechanical properties compared to insulating properties. Consequently, degradation will be compared with the properties at month 3 for each characteristic.

These graphs confirm a degradation in the samples. However, much like in the thermal test, these properties are highly influenced by ambient humidity. Therefore, it would be more accurate to increase the duration of environmental durability cycles to validate the results when humidity levels return to lower values. It's worth noting that despite the degradation, these samples still maintain superior mechanical properties compared to EPS. In this application, the samples will already be in place, and there is no need for them to have a structural component as long as the material doesn't disintegrate.

Furthermore, these results align with previous findings in mechanical properties and FTIR analysis. It was confirmed that the inclusion of acid does not enhance the cross-linking of cellulose in the samples, and a catalyst is necessary to initiate the reaction in hemp stalk.

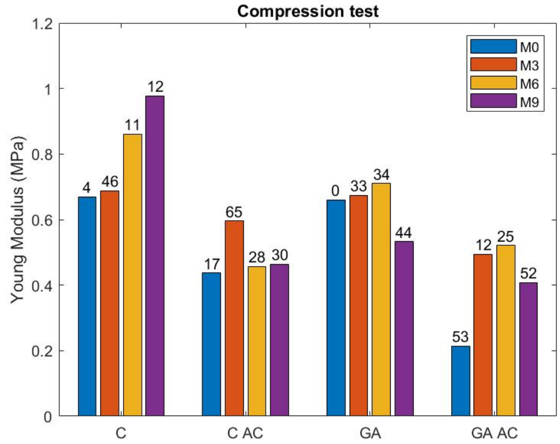


Figure 4.28: Degradation of compression strenght

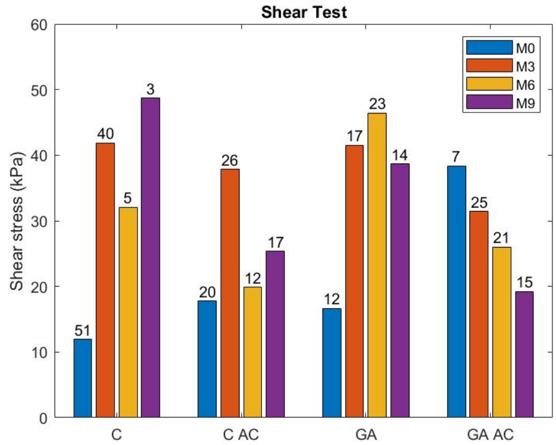


Figure 4.29: Degradation of shear strenght

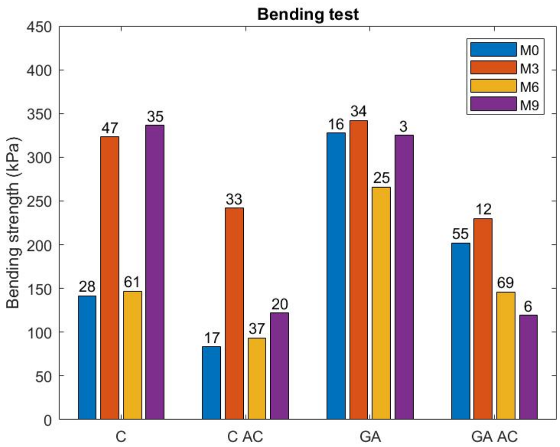


Figure 4.30: Degradation of bending strenght

To compare the influence of moisture in the samples, Figure 4.31 displays the grammage of each sample (with 3 cm of thickness) alongside their compression Young modulus. The figure illustrates that as the grammage increases, the mechanical properties of the samples decrease. This is because the difference in grammage corresponds to a higher moisture content in the sample, which in turn reduces the mechanical properties.

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 stalk 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.

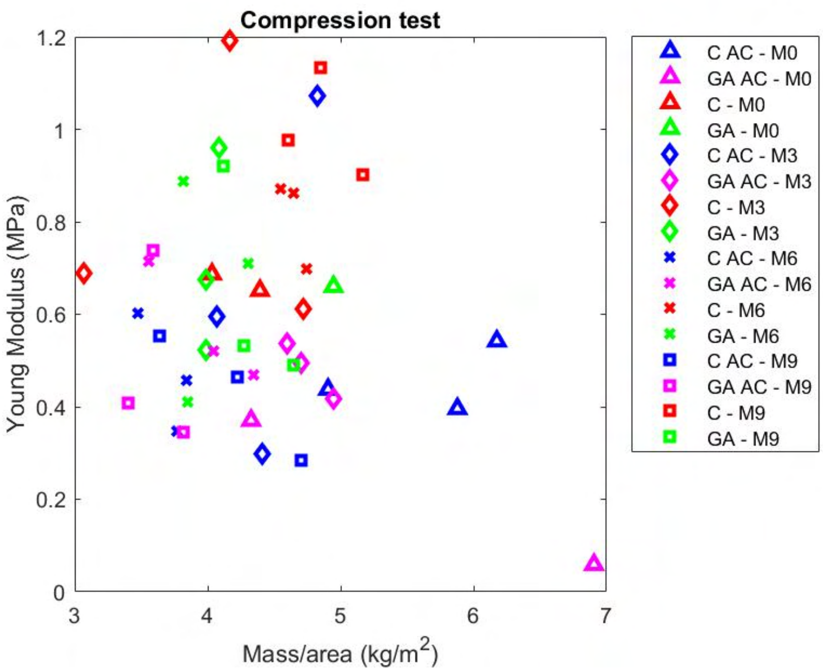


Figure 4.31: Influence of moisture in compression test

4.14 Conclusions

In this study, an experimental investigation was conducted to explore an insulating bio-material based on hemp stalk, eucalyptus pulp, 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:

- The proposed insulating material is predominantly made up of hemp, with 70% of the composition consisting of hemp stalk and the remaining 30% composed of recycled cardboard fiber. Additionally, a surface coating is applied, with options of either colophony or gum arabic depending on the specific application. The material offers excellent insulation properties, positioning it as a viable alternative to conventional inorganic materials.
- The thermal insulation performance of the proposed material surpasses that of commercial materials by 15-40%. This improvement can be attributed to the unique properties of the constituent materials and the synergistic microstructure formed in the composite material.
- In the context of acoustic insulation, it achieves a reduction of 0.8-0.9 dB/dB, which is slightly lower (0.2 dB/dB) when compared to rockwool. The hollow microstructure of the material make a great performance by atenuating the sound energy.
- The material possesses a notable capability for absorbing ambient humidity, thereby enhancing hydrothermal comfort by reducing humidity fluctuations within the building environment.
- To safeguard the composite material from environmental influences, the addition of a protective coating is necessary, as an elevated moisture level can have a substantial impact on its properties. The properties of the material can be customized for specific applications through the selection of appropriate coatings. Arabic gum, with its fire and moisture resistance, is suitable for insulating panels in building construction. On the other hand, colophony, with its hydrophobic properties, is suitable for applications such as building sheds.
- The proposed material meets the necessary mechanical resistance requirements to be used as an insulating panel in construction. It exhibits higher values of compressive, shear, and bending strength compared to EPS (Expanded Polystyrene).
- The experimental tests demonstrate that the inclusion of citric acid does not lead to a significant improvement in cellulose crosslinking within the material. The FTIR test confirms that citric acid does not react with the hemp stalk, it is necesary to study the influence of a catalyst to initiate the chemical reaction. However, despite the absence of a chemical reaction, the bond between the two materials is sufficiently strong to uphold high properties when compared to other insulation materials.
- The inclusion of acid citric enhances the panels' longevity by diminishing the hydrogen bonds among cellulose molecules. Nevertheless, for a more precise quantification of the improvement, it is advisable

to augment the number of cycles.

A recommended future study is to investigate the compatibility of inorganic coatings with the composite material. This research aims to address specific requirements such as protection against direct water contact, moisture resistance, fire protection, and long-term stability.

Recommended areas for future research include further enhancements to the fabrication methods. Additional tests, such as oven curing, could be interesting to carried out to evaluate potential effects of the resin on compressive strength and the occurrence of any softening that might require oven curing. Another aspect to explore in future studies is the influence of different levels of applied pressure on the samples in insulating and mechanical properties.

While the new bio-composite material, based on hemp stalk and eco-friendly binding materials, shows potential as a substitute for conventional inorganic insulating materials in building construction, further research is needed to enhance its long-term stability and moisture resistance. Continued exploration and advancement of this material will be crucial for its successful application as a sustainable building material.

Chapter 5

Life cycle

In this section, the objective is to examine whether the use of the developed material aligns with a sustainable development model: one that satisfies the needs of the current generation without compromising those of future generations. Understood as a necessity, it involves consuming services formed from limited resources. Thus, it should not exceed what can be generated sustainably.

To achieve this, three sustainability indicators are used: carrying capacity, carbon footprint, and life cycle analysis.

- Carrying Capacity: This indicator represents the maximum number of individuals a service can support without jeopardizing future needs.
- Carbon Footprint: This measures the amount of land or sea required to support the human population and absorb the carbon and waste they generate.
- Life Cycle Analysis: This is an objective procedure aimed at assessing the environmental impacts of a product's entire life cycle, from extraction and processing to use, disposal, and even recycling. It quantifies resource consumption, water and energy usage, emissions, and waste generation at each stage, ultimately expressing these processes as the product's environmental impact.

In this section, an exhaustive life cycle analysis of the material will not be conducted as it falls beyond the scope of this thesis. Instead, a short analysis will be performed using the life cycle methodology. These analyses will aid in presenting the material as environmentally friendly and a greener alternative compared to the synthetic materials commonly employed today. Therefore, the analysis is conducted manually without utilizing commercial software databases such as SigmaPro.

5.1 Methodology

The subsequent guidelines prescribe how to conduct a life cycle analysis of materials:

- Environmental management - Life cycle assessment - Principles and framework: ISO 14040:2006 [149]
- Environmental management - Life cycle assessment - Requirements and guidelines Amendment 2: ISO 14044:2006 [150]

In order to be more rigurous methology, the life cicle analyse will follow the guidance outlined in the PCR (Product Category Rules) for insulating materials made of foam plastics [195]. The document contains the requirements on an Environmental Product Declaration (EPD) for the range of environmental product declarations published by Institut Bauen und Umwelt e.V. (IBU). In the document the results of the life cycle are presented in the following stages:

- Product stage
  - Raw material supply
  - Transport
  - Manufacturing
- Construction process stage
  - Transport from the gate to the site
  - Assembly
- Use stage
  - Use
  - Maintenance
  - Repair
  - Replacement
  - Refurbishment
  - Operational energy use
  - Operational water use
- Ends of life stage
  - De-construction demolition
  - Transport
  - Waste processing

  - Disposal

- Benefits and loads beyond the system boundaries

  - Reuse/Recovery/Recycling potential

These phases align with a cradle to cradle system; however, for the purposes of this analysis, it will be simplifyied. Consequently, the emphasis will be on examining until the material's use, excluding considerations of end-of-life phases and recycling, which have not been extensively investigated in the thesis. Corresponding to a cradle to use system.

The material being studied is the insulating panel made up of 70% hemp stalk, 30% recycled cardboard fibers, and an arabic gum coating. This material is fabricated under laboratory conditions. To simplify the analysis, it's important to note that resources related to the construction of the factory, waste generated from personal protective equipment (PPE) like gloves, the production of the panels, packaging, or the initial stages of secondary materials like arabic gum, etc., will not be included in the assessment.

In order to perform the analysis the PCR define that the declared unit for coated insulating materials is 1 m^2. This measured will be use to calculate the inputs and outputs of every stage that are needed.

5.2 Life cycle analysis

In that section the life cycle analysis will be perform taking into account the coonsiderations explaned in the methodology.

5.2.1 Product stage

Raw material supply

In this stage, arabic gum will not be considered for simplification. On the other hand, cardboard and hemp stalk are regarded as recycled and waste materials, so in this case, the inputs and outputs are treated as zero. Regarding hemp stalk, some inputs include water, fertilizer, and other products used in cultivation, while outputs include CO_2 emissions from chemical products, etc. However, since it is considered a waste material, all these contributions are associated with the main products of the crops, which are the textile industry.

Transport

For this part, the fuel consumption of the transport truck to carry the raw material to the factory where the panel is processed is considered. 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 100 kilometers will be assumed. Consequently, it is assumed that a truck carrying 10,000 kg of raw materials consumes an average of 40 liters of fuel for every 100 kilometers traveled.

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 0.056 liters is considered, resulting in the production of 0.148 kg of CO_2 equivalent emissions.

Manufacturing

In the manufacturing stage, the electricity consumption in the laboratory factory will be taken into account. This includes the disintegration of the recycled cardboard fiber, the mixing of raw materials, the preparation of resin with Arabic gum, and the electricity consumed by the oven during the curing process.

The disintegrator machine consumes 0.3 kW h, and it is required for 1 hour to produce the material for one functional unit. The preparation of the coating consumes 0.1 kW h for 15 minutes. However, the oven is the most energy-consuming component, using 0.5 kW h for 48 hours for one functional unit. In total, the energy consumption amounts to 24.3 kW h for manufacturing one functional unit. In the case of Catalonia, the emission are 273 g $CO_{2\ eq}$/kW h considering the data provided by the government for the year 2023. 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 1% loss will be taken into account, 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 arabic gum used for coating the functional unit, 0.8 L of water is needed. In that case, a consumption of 1.8 L of water is considered.

5.2.2 Construction process stage

Transport from the gate to the site

In this stage, the same assumptions made in the previous stage will be applied, as the same inputs and outputs are provided.

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.

5.2.3 Use stage

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

5.2.4 Results

Considering all the simplifications made in this analysis, to prduce a funtional unit oof 1 m^2 of the green insulating panels, is necesary a compsutioon of 6.5 kg of raw material and 1.8 L of water resulting in a emissions of equivalent CO_2.

Considering all the simplifications made in this analysis, to produce a functional unit of 1 m^2 of the green insulating panels, it is necessary to consume 6.5 kg of raw material and 1.8 L of water, resulting in emissions of 6.9 kg of equivalent CO_2.

Furthermore, it's crucial to consider that hemp absorbs CO_2 during its growth, a factor not accounted for in this analysis. In the first stage, a ton of hemp can absorb 325 kg of CO_2 during crop growth. In the functional unit, 4 kg of raw hemp stalk is used, which results in an absorption of 1.3 kg of CO_2. With this data, the insulating panels' final carbon emissions are still positive (5.6 kg of CO_2 emitted into the atmosphere).

Additionally, this analysis highlights that the curing stage in the oven is the phase with the highest emissions, accounting for 95% of the total emissions. This could potentially be improved in a large-scale industrial factory by implementing more efficient machinery, ultimately achieving the desired negative CO_2 emissions. Moreover, the CO_2 absorbtion during the cultivation of arabic gum should be taken into account.

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. However, further studies would be necessary to confirm this and incorporate it into the life cycle analysis.

5.3 Conclusions

The life cycle analysis conducted in this thesis has been simplified to provide a basic technical characterization of the material. A more comprehensive study and the utilization of commercial 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 stalk.

- A functional unit of the green insulating panel is associated with 6.9 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, eventually achieving a negative carbon footprint.

Chapter 6

Conclusions and future research

6.1 Conclusions

Based on the results presented in various studies conducted over the past few decades, hemp stalk emerges as a green material with exceptional insulating properties, positioning it as a replacement for certain inorganic commercial materials. This growing interest in hemp stalk can be observed through the increasing number of research initiatives and the expansion of the hemp industry in the business sector. The insulating panels developed in this thesis improve thermal insulation and acoustic comfort in buildings, thereby aiding in the creation of non-structural materials for eco-friendly construction.

Here are some key conclusions and recommendations based on the findings of this thesis:

- **Hemp stalk applications:** Based on the initial studies conducted, hemp stalk exhibits significant thermal and acoustical insulating properties for the production of eco-friendly insulation materials, which is the primary focus of this thesis. Moreover, coffered ceilings are also an application where hemp stalk can be a substitute for synthetic materials.
- **Variability in hemp stalk properties:** Different hemp varieties and positions on the stem can result in significant variations in the elastic modulus of hemp stalk, up to 60%. These variations can be attributed to the distinct sizes of cell walls in different hemp varieties, as segments with larger cell walls exhibit a higher elastic modulus. Nonetheless, it is still uncertain whether these fluctuations in cell size directly affect the insulating properties of hemp. To enhance the material's performance, it is crucial to confirm the stability of the raw material's properties. It's possible that to achieve higher quality, the hemp stalk should be provided from a specific section of the stem.
- **Importance of binders:** The choice of binder plays a critical role in creating a low-density and porous material, which is essential for achieving optimal insulating properties. A notable gap in the existing literature is the scarcity of studies employing recycled cardboard fibers as a binder in combination

with other eco-friendly particles to fabricate insulating panels. Recycled cardboard exhibits great compatibility with hemp stalk, resulting in a stable material comprising a minimum of 30% cardboard fiber. This manufacturing process does not require high temperatures or pressure, reducing the energy consumption during production while also yielding a low-density material with high porosity, thus enhancing its insulation properties.

- **Green insulating panels:** The proposed insulating material is primarily composed of hemp, with 70% of the composition consisting of hemp stalk and the remaining 30% comprising recycled cardboard fiber. Increasing the quantity of hemp enhances the acoustical and thermal insulating properties, surpassing the performance of both materials individually due to the synergistic qualities of the hollow microstructure it creates. Additionally, a surface coating is applied, with the choice between colophony or gum arabic depending on the specific application. The material offers excellent insulation properties, positioning it as a viable alternative.
- **Insulating properties:** Hemp-based insulating materials demonstrate competitive insulating properties (0.02-0.03 W/m K and 0.85-0.95 dB/dB). Surpassing some commercial alternatives in thermal insulation by 15-40% and closely matching them in acoustic insulation (0.2 dB/dB lower) when compared to rockwool. The exceptional insulating properties are achieved through the intrinsic characteristics of the materials themselves and their synergistic interactions, creating a hollow microstructure that forms air gaps. These air gaps effectively reduce heat transfer and dampen sound waves.
- **Protection against ambient conditions:** Hemp-based composite materials are susceptible to environmental degradation. The green insulating panel disintegrates in just 3 minutes upon direct contact with water. To address this, a protective coating is essential. Arabic gum is effective in shielding the material from moisture, while a colophony coating renders the material hydrophobic.
- **Ambient confort:** The material possesses a notable capability for absorbing ambient humidity, thereby enhancing hydrothermal comfort by reducing humidity fluctuations within the building environment. By adding a coating of arabic gum, the MBV increased in a 30% due to its capacity to dissolve in water, resulting in a significant uptake of ambient moisture.
- **Mechanical strength:** The proposed materials meet the necessary mechanical strength requirements for insulating panels in construction. They have a compression elastic modulus of 0.65-0.95 MPa, shear strength of 40.0-42.0 kPa, and bending strength of 310-430 kPa in the best cases studied, surpassing the performance of Expanded Polystyrene (EPS). In the case of mechanical properties, the binder has a significant influence. The sample with cardboard increased the shear strength of the eucalyptus by 100% due to the longer fiber length and improved mechanical properties. Additionally, applying pressure in the fabrication method can enhance the mechanical properties; however, it also reduces the material's porosity, resulting in lower insulation performance. As a result, this approach was ultimately discarded.

- **Influence of citric acid:** The inclusion of citric acid enhances the material's durability by reducing hydrogen bonds within cellulose molecules in the cardboard fibers.
- **Fire resistance:** The insulating panel developed cannot withstand direct contact with an open flame. However, this limitation can be addressed by applying a coating. When coated with arabic gum, the material can come into contact with a flame for a short duration without igniting or propagating the flame.
- **Life cicle analysis:** A functional unit of the green insulating panels (1 m^2), produced in laboratory conditions, is associated with 6.9 kg of equivalent CO_2. However, it's important to note that hemp has the capability to sequester carbon dioxide (CO_2) during its cultivation, which results in a net carbon footprint of 5.6 kg emitted into the atmosphere. The negative result could potentially be reversed with the use of an efficient industrial manufacturing process that could achieve a total negative carbon footprint. Additionally, opting for biomaterials like cardboard fiber and arabic gum can significantly reduce the carbon footprint. However, these factors were omitted in the analysis.
- **Recycling and circular economy:** Once hemp-based insulating materials reach the end of their life cycle or are damaged, they can be disintegrated with water and reused as raw materials. This offers significant potential for recycling and supports a circular economy.

In conclusion, the research conducted thus far highlights the promising potential of hemp-based composite materials for sustainable insulation and construction applications. However, continued research and development are essential to optimize these materials for real-world use, enhance their properties, and ensure their long-term durability. This ongoing exploration will play a crucial role in advancing sustainable building materials and reducing the environmental impact of construction practices.

6.2 Future research

As a result of the research conducted in this thesis, several questions have arisen, identified as gaps in the current state of knowledge, and potential avenues for future research. These may prove to be compelling areas for future studies that will contribute to a deeper understanding within the scientific community on this topic.

Future research should investigate the relationship between hemp stalk properties, including variety and stem position, and their effect on insulation properties.

Plant-based composite materials, although significant for reducing the planet's negative impact, may not always meet the mechanical property criteria for non-structural applications. To tackle this challenge, researchers can investigate the incorporation of inorganic components or a proportion of green materials into the resin to improve mechanical performance, all while adhering to their commitment to environmentally friendly solutions. Moreover, further research is required to investigate the acoustic properties, fire resistance,

resistance to fungal growth, and long-term durability of 100% green composite materials. The existing literature contains limited information on these topics.

Future research should explore the compatibility of inorganic coatings with the composite material to meet specific requirements such as moisture resistance, fire protection, and long-term stability. Additionally, investigations into fabrication methods and the influence of different levels of applied pressure on insulating and mechanical properties should be conducted. An extended durability and end-of-life test could also be of interest to comprehend how the material performs over an exceptionally long period, such as 20 or 50 years.

Coffered ceilings presents itself as an intriguing avenue for further investigation. This could involve the utilization of colophony as a binder or potentially employing the same approach as with the insulating panels, using recycled cardboard as a binder and incorporating colophony as a protective coating.

Further research is necessary to accurately measure the enhancement in the durability of insulating panels through the addition of citric acid. Furthermore, exploring various chemical catalysts to activate cellulose crosslinking in hemp stalk and assessing their impact on insulating properties is an intriguing area of study.

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Appendices

Appendix A

Experimental campaign

In the appendix section, the process conducted in each of the tests will be detailed. Including: regulations, any modifications made compared to the standard procedures, the equipment and instrumentation used, as well as the raw results obtained from all the specimens tested throughout the document. Additionally, the behavior of the material in each of the tests will be explained.

A.1 Compatibility of hemp stalk and eucalyptus fibber

In this section, the results obtained during the initial manufacturing phase for each specimen are elaborated. Different proportions of cellulose fiber and hemp were employed, and their suitability was determined through characterization. Furthermore, an investigation will be conducted to assess the appropriateness of employing manual pressing as a drying method, and a determination will be made regarding whether soaking the hemp in water 24 hours prior to specimen fabrication is a viable approach. The various variables presented in the sample include:

- %wt. of eucalyptus fiber (100/80/70/60/50/40/30/20/0E)
- Wet or dry hemp stalk (W/D)
- Dry process by ambient or apply pressure in the sample (A/P)

The characteristic of every sample will be presented, along with the relevant dosage, an accompanying table will be included to categorize the fabricated specimens according to the condition of the hemp and the utilized drying method. Images of both the upper and lower sides of each specimen will be displayed. The upper surface corresponds to the area exposed during the vacuum process, while the lower surface pertains to the region in contact with the filter paper positioned in the Buchner funnel when vacuum was applied.

The sample 100E-P, depicted in Figure A.1, is produced using only disintegrated raw kraft eucalyptus pulp. This specimen type has been crafted to understand the manufacture process and methodology that should be employed for subsequent specimens containing a certain quantity of hemp.

Figure A.1: 100E-P

The 80E sample comprises 80% cellulose fiber and 20% hemp, illustrated in Figure A.2. Four sample variations have been created, where two incorporate wet hemp, while the other two involve dry hemp using two distinct manufacturing methods. This strategy facilitates the characterization of the samples to ascertain which of the two hemp states interacts more effectively with the binding material. Due to the significantly greater cellulose fiber content, a more uniform distribution of hemp within the sample is produced, resulting in a well-defined circular shape without any signs of fracturing.

In the case of 70E sample, Figure A.3. A lower amount of hemp leads to improved adhesion to the paper fibers, resulting in a more uniform specimen. In the case of sample 70E-W-P, upon removal from the press, the filter paper could not be taken off due to the risk of it breaking, attributed to its thinness.

In the case of 60E sample, Figure A.4. Continue the same behaviour.

In the case of 50E sample, Figure A.5. It was observed in 50E-W-P sample, that the hemp remained on top and struggled to blend with the cellulose fibers during vacuum filtration, especially for the coarser shreds. Nevertheless, in the remaining specimens, a strong adhesion between the hemp and paper fibers has been observed.Continue the same behaviour.

In the case of 40E sample, Figure A.6. A non-homogeneous sample in the case of dry hemp stalk is noted.

In the case of 30E sample, Figure A.7. The sample specimens produced with dry hemp have not been well compacted with the cellulose fiber, leading to non-homogeneity in the mixture and insufficient adhesion between the two raw materials. It can be observed that in the pressed specimen, a uniform circular shape has not been achieved, resulting in losses of raw material.

In the case of 20E sample, Figure A.8. Regarding the specimens manufactured with dry hemp, it was observed that when pouring the mixture into the Buchner funnel for filtration, most of the hemp remained afloat and didn't bind well with the disintegrated kraft pulp. As a result, the mixture was not homogeneous. Upon completing the filtration and removing the specimen from the Buchner funnel, pieces of hemp broke off, with the majority remaining on the surface without adhering to the cellulose fiber. The 20E-D-P/A specimens broke when removing the filter paper. Therefore, when using dry hemp in this dosage, it's evident that a proper bond wasn't formed, and the compactness wasn't achieved. On the other hand, the specimens manufactured with wet hemp, 20E-W-P/A, mixed more easily with the disintegrated Kraft pulp, resulting in a more homogeneous mixture.

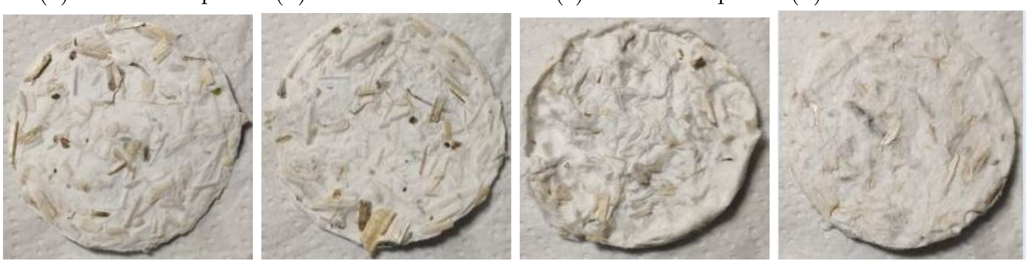
In the case of 0E sample, Figure A.9, all the sample is unestable due to the absence of binder material.

(a) 80E-D-P top (b) 80E-D-P bottom (c) 80E-D-A top (d) 80E-D-A bottom

(e) 80E-W-P top (f) 80E-W-P bottom (g) 80E-W-A top (h) 80E-W-A bottom

Figure A.2: Sample 80E

(a) 70E-D-P top (b) 70E-D-P bottom (c) 70E-D-A top (d) 70E-D-A bottom



(e) 70E-W-P top (f) 70E-W-P bottom (g) 70E-W-A top (h) 70E-W-A bottom

Figure A.3: Sample 70E

(a) 60E-D-P top (b) 60E-D-P bottom (c) 60E-D-A top (d) 60E-D-A bottom

(e) 60E-W-P top (f) 60E-W-P bottom (g) 60E-W-A top (h) 60E-W-A bottom

Figure A.4: Sample 60E

(a) 50E-D-P top (b) 50E-D-P bottom (c) 50E-D-A top (d) 50E-D-A bottom

(e) 50E-W-P top (f) 50E-W-P bottom (g) 50E-W-A top (h) 50E-W-A bottom

Figure A.5: Sample 50E

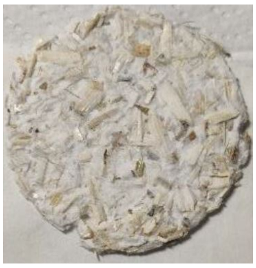

(a) 40E-D-P top


(b) 40E-D-P bottom


(c) 40E-D-A top


(d) 40E-D-A bottom


(e) 40E-W-P top


(f) 40E-W-P bottom


(g) 40E-W-A top


(h) 40E-W-A bottom

Figure A.6: Sample 40E


(a) 30E-D-P top


(b) 30E-D-P bottom


(c) 30E-D-A top


(d) 30E-D-A bottom

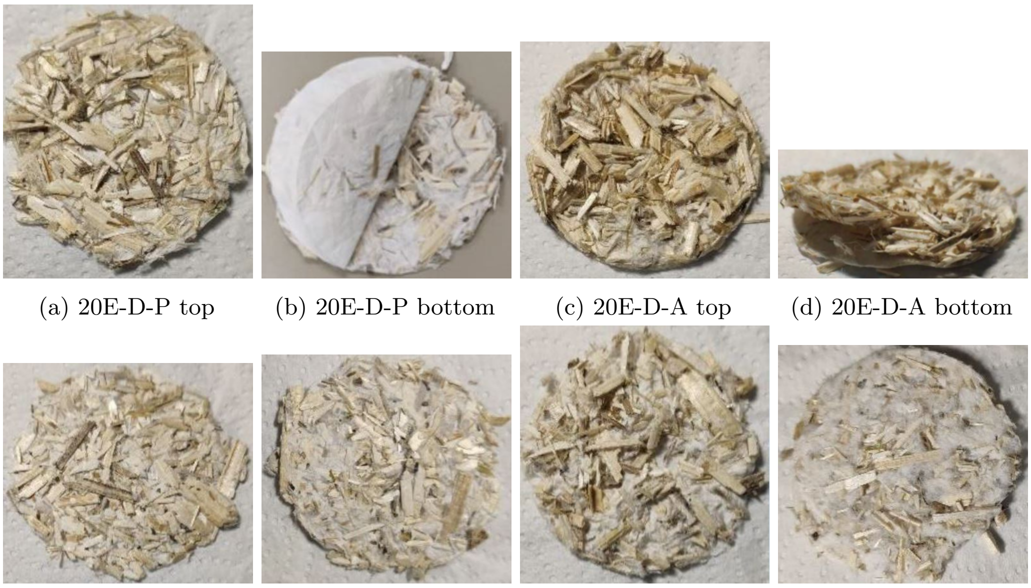

(e) 30E-W-P top


(f) 30E-W-P bottom


(g) 30E-W-A top


(h) 30E-W-A bottom

Figure A.7: Sample 30E



(a) 20E-D-P top (b) 20E-D-P bottom (c) 20E-D-A top (d) 20E-D-A bottom

(e) 20E-W-P top (f) 20E-W-P bottom (g) 20E-W-A top (h) 20E-W-A bottom

Figure A.8: Sample 20E

Figure A.9: Sample 0E

After establishing the dry weight percentage of each component, the next phase entails evaluating the weight per unit area necessary to attain a 5 cm thickness for the insulation material formation. This is done while ensuring that compatibility with the existing behavior is maintained.

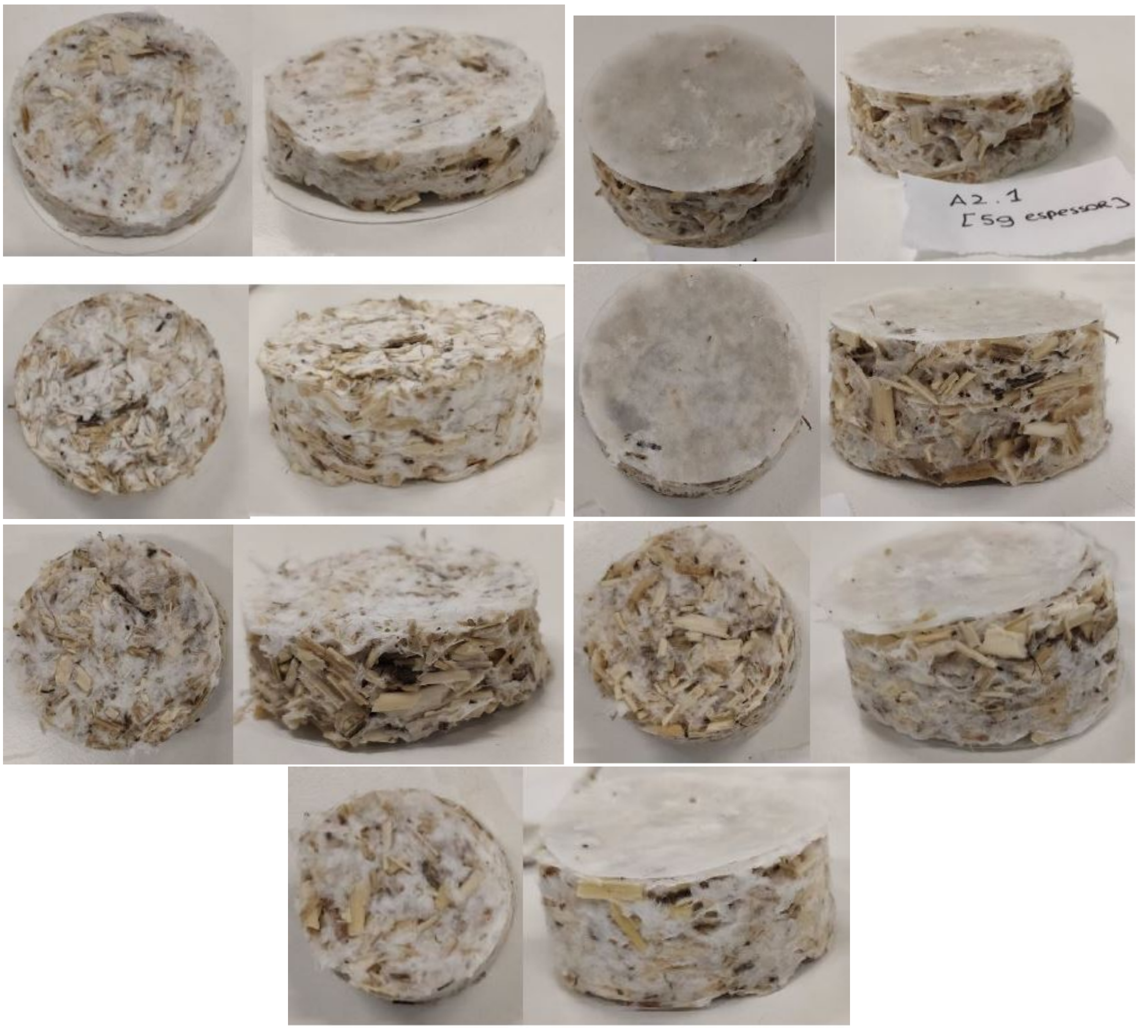


Figure A.10: Sample 30E with different weight

A.2 Acoustic test - ISO 10534

The raw data off the acoustic test is presented. The following figures represent the sample tested for the characterization process of the insulating panels.

For rockwool, the chosen samples include an aluminum layer on one side for fire protection. Testing both sides revealed that without the aluminum layer, better results were achieved. The presence of aluminum leads to sound waves bouncing within the impedance tube, influencing the final outcome.

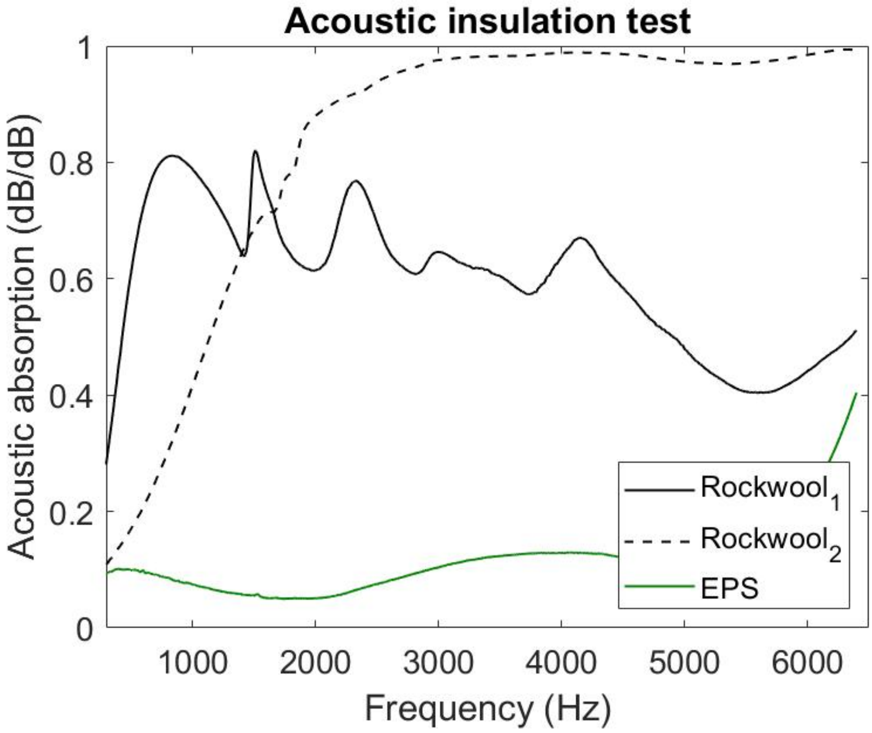


Figure A.11: Acoustic absorption of commercial materials

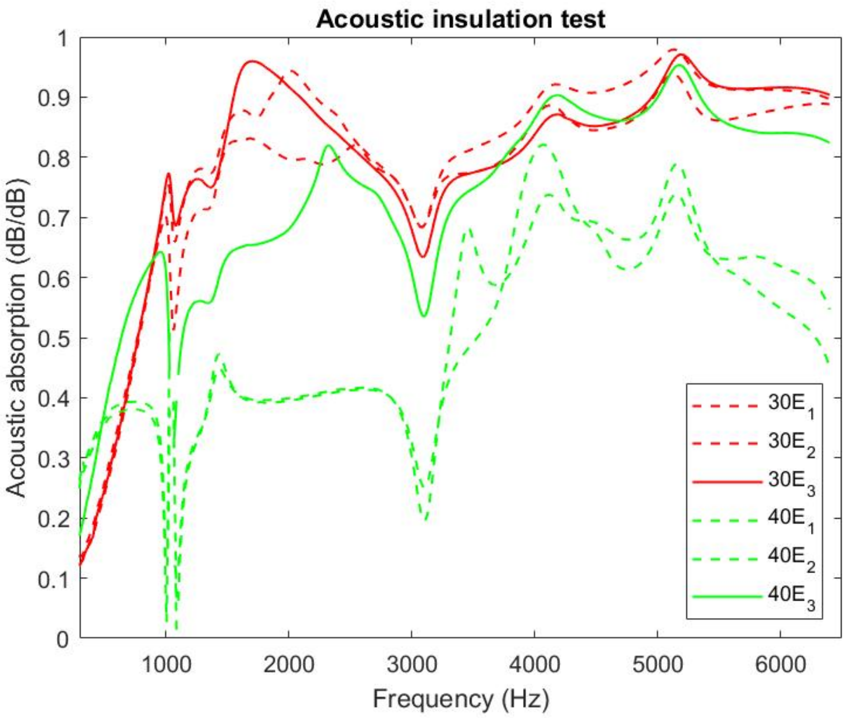


Figure A.12: Acoustic absorption of eucalyptus-hemp stalk samples along the thickness

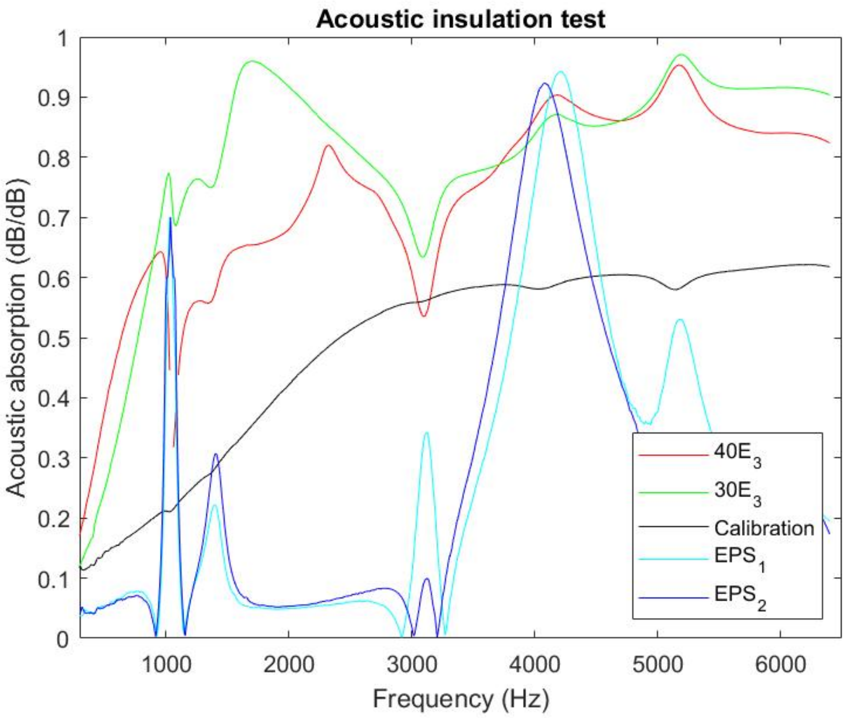


Figure A.13: Comparison of acoustic absorption

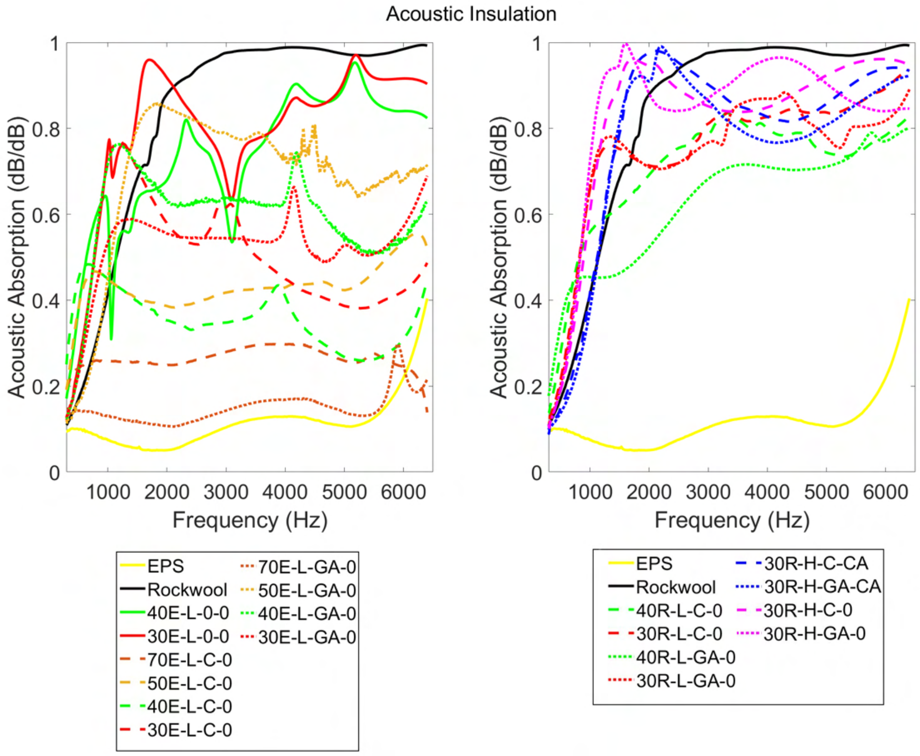


Figure A.14: Comparison of acoustic absorption of cardboard-hemp stalk samples

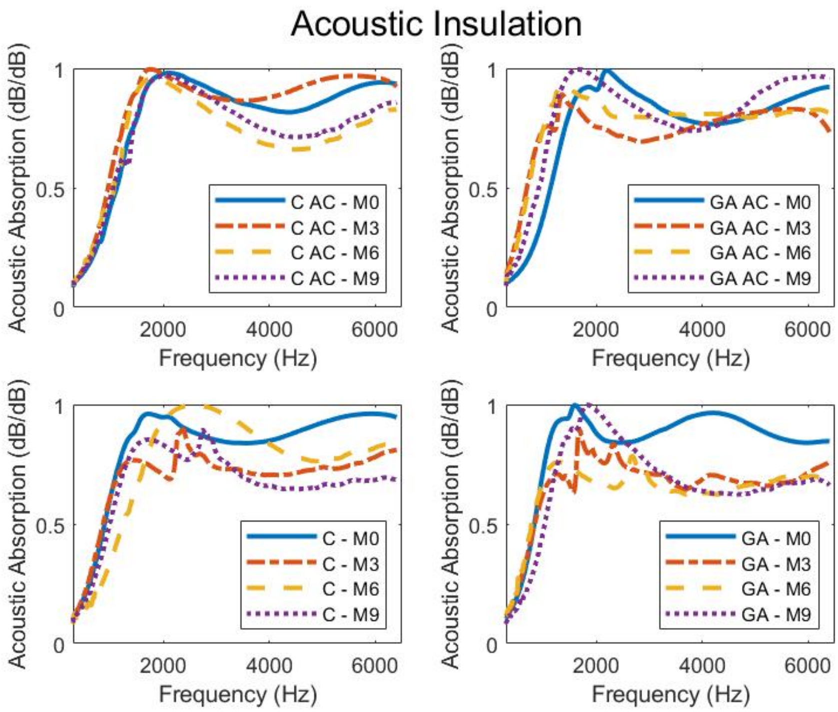


Figure A.15: Comparison of acoustic absorption of durability samples

A.3 Thermal test - ISO 12664

In this section the figures of the raw data of the thermal insulating test performed to the insulating panels will be presented.

The following visuals illustrate the temporal temperature gradient of the samples, which is crucial to ensure the achievement of the quasi-stationary phase required for calculating the thermal insulation of the specimens.

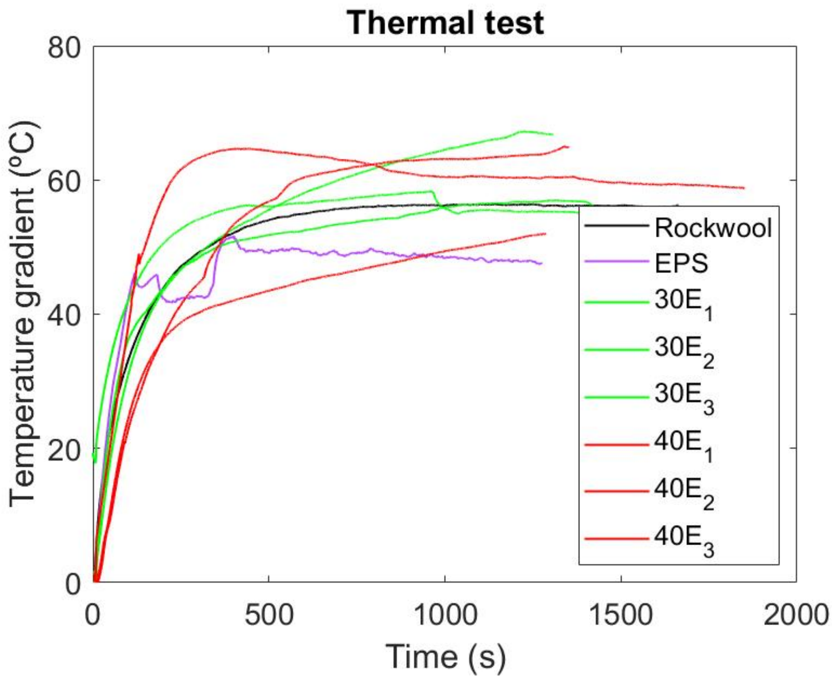


Figure A.16: Temperature gradient-time of eucalyptus-hemp stalk samples

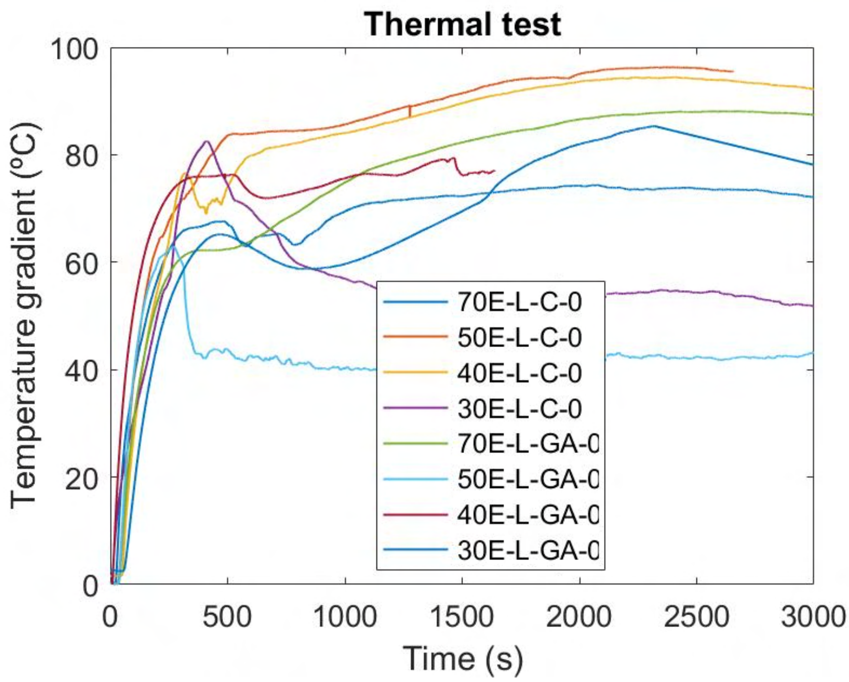


Figure A.17: Temperature gradient-time of eucalyptus-hemp stalk samples with coating

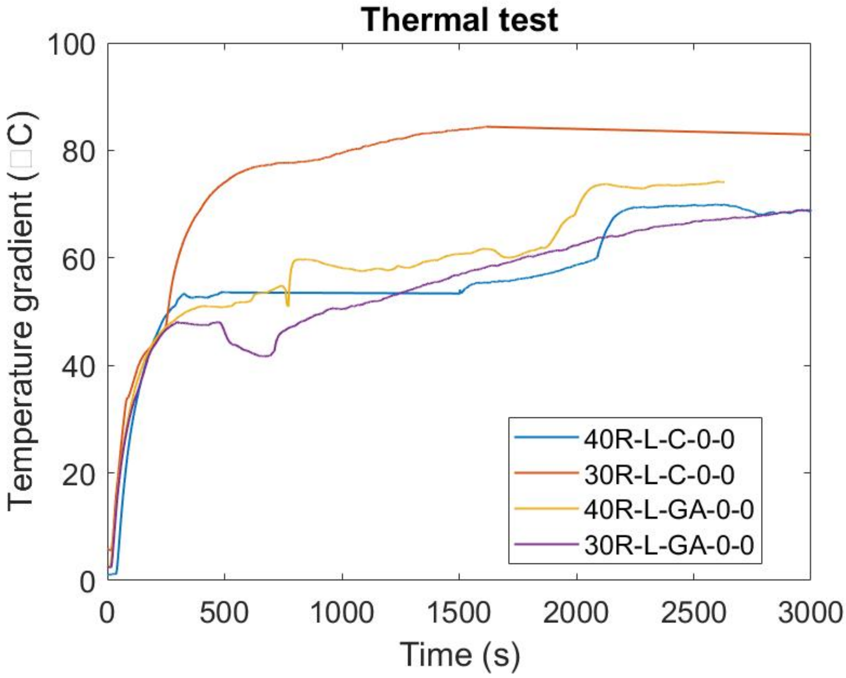


Figure A.18: Temperature gradient-time of hemp stalk-cardboard samples

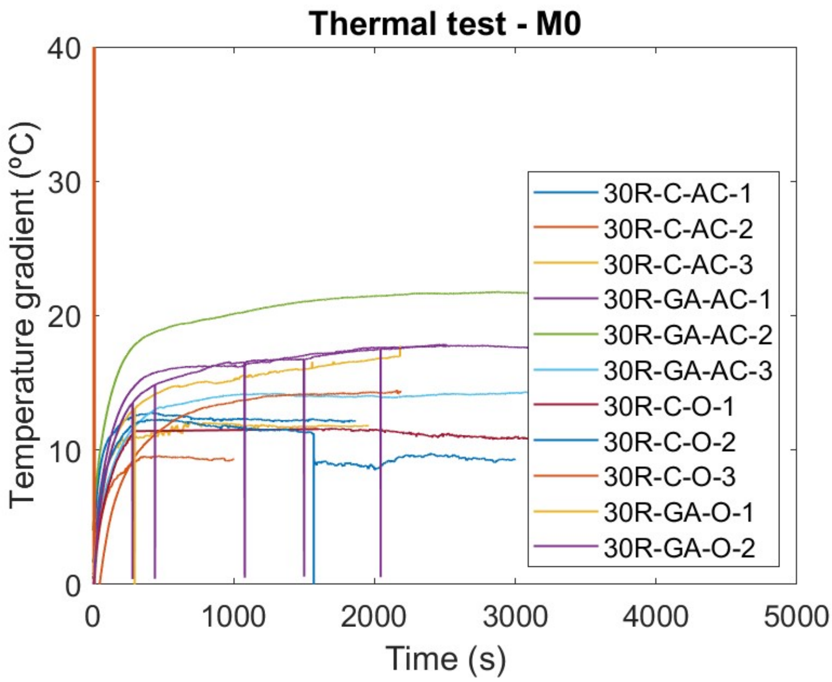


Figure A.19: Temperature gradient-time of hemp stalk-cardboard durability M0 samples

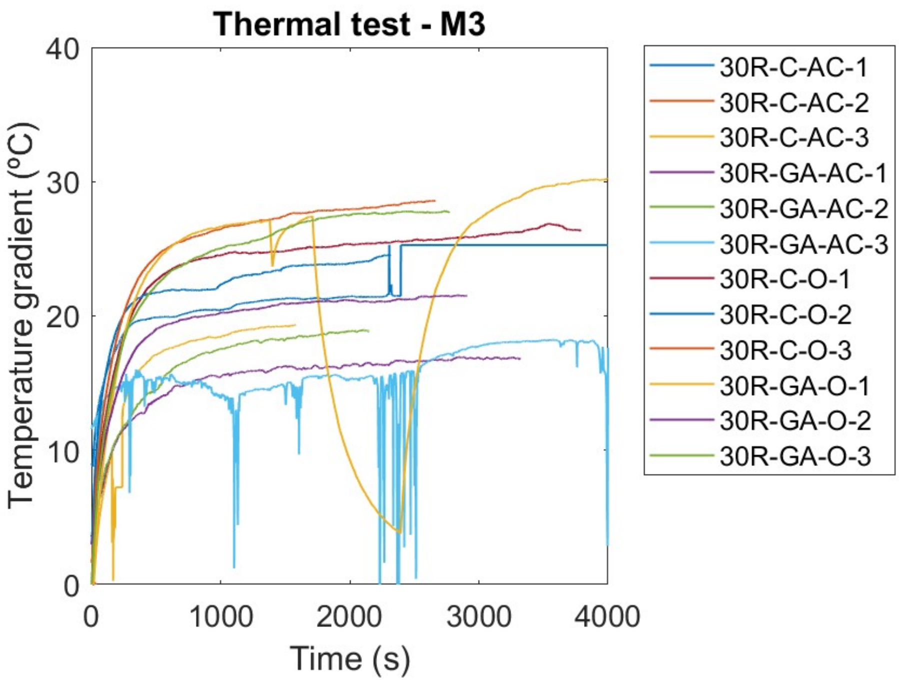


Figure A.20: Temperature gradient-time of hemp stalk-cardboard durability M3 samples

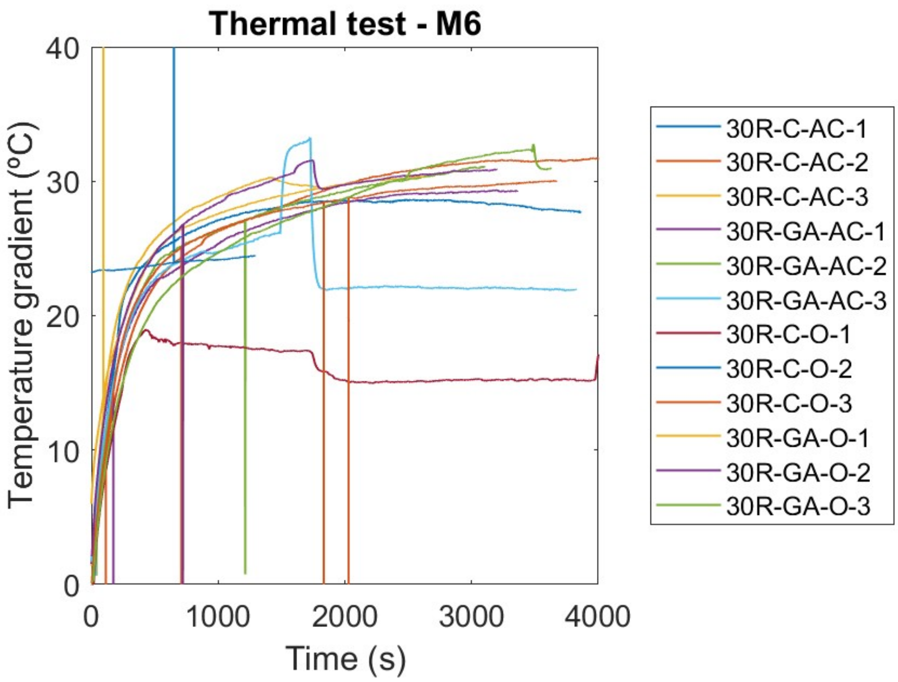


Figure A.21: Temperature gradient-time of hemp stalk-cardboard durability M6 samples

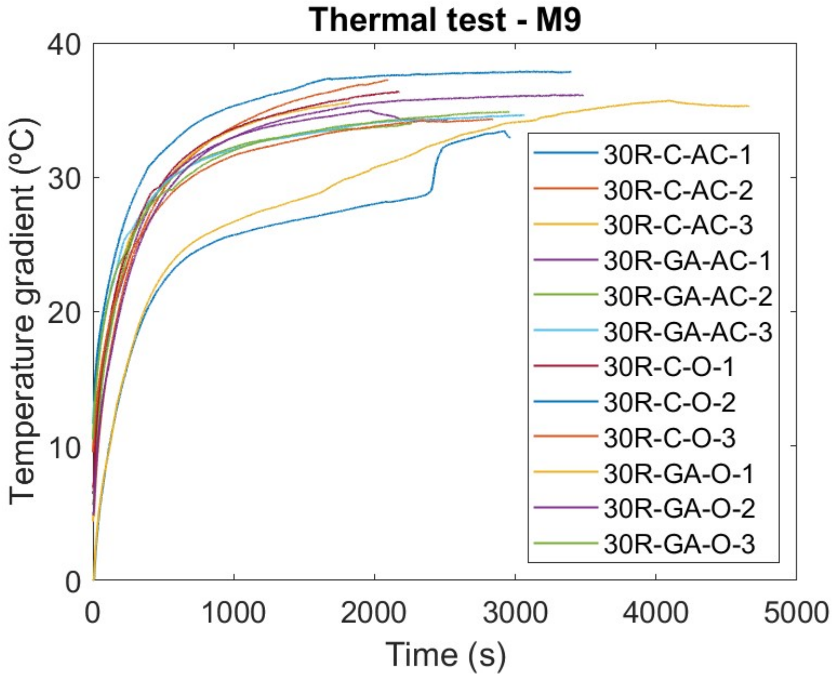


Figure A.22: Temperature gradient-time of hemp stalk-cardboard durability M9 samples

A.4 Compression test - EN 826

The next figures represent the raw data of the compression test performed to the insulating panels.

The behavior can be categorized into three phases. Initially, the curve exhibits curvature attributed to both the mechanism and material adjustment, as the sample isn't entirely flat. The second phase corresponds to the linear behavior of the material, enabling mechanical calculations. The third phase signifies the inelastic material behavior; due to porosity, the material doesn't break under compression, resulting in an exponential curve for the plastic zone.

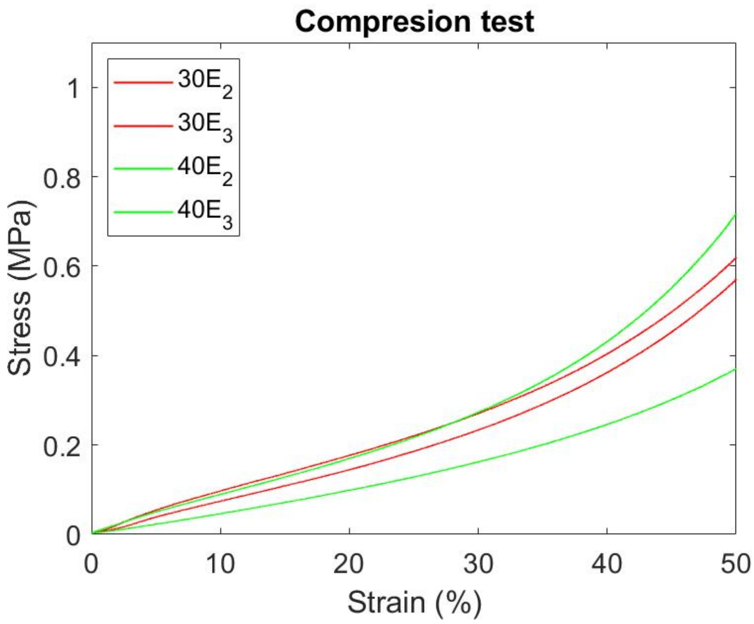


Figure A.23: Compression test of eucalyptus-hemp stalk samples

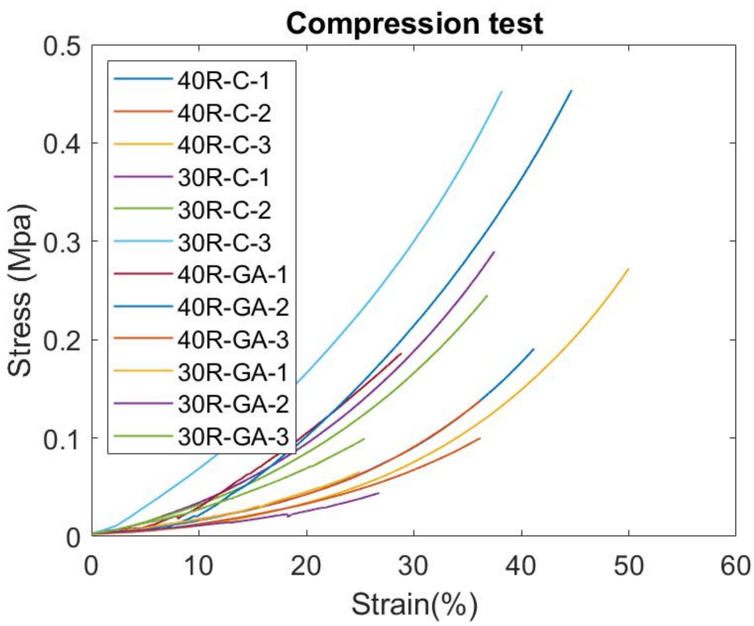


Figure A.24: Compression test of hemp stalk-cardboard samples

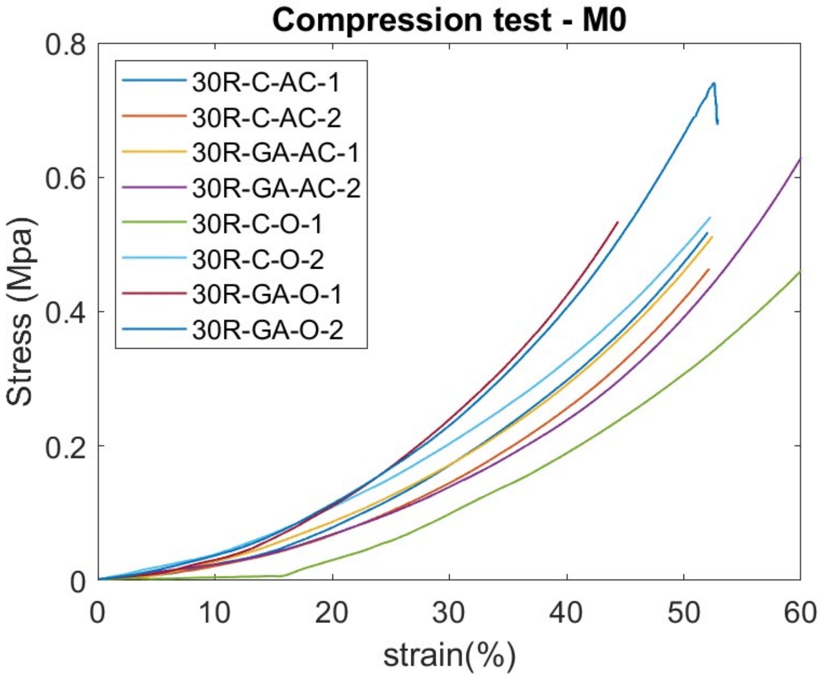


Figure A.25: Compression test of hemp stalk-cardboard durability M0 samples

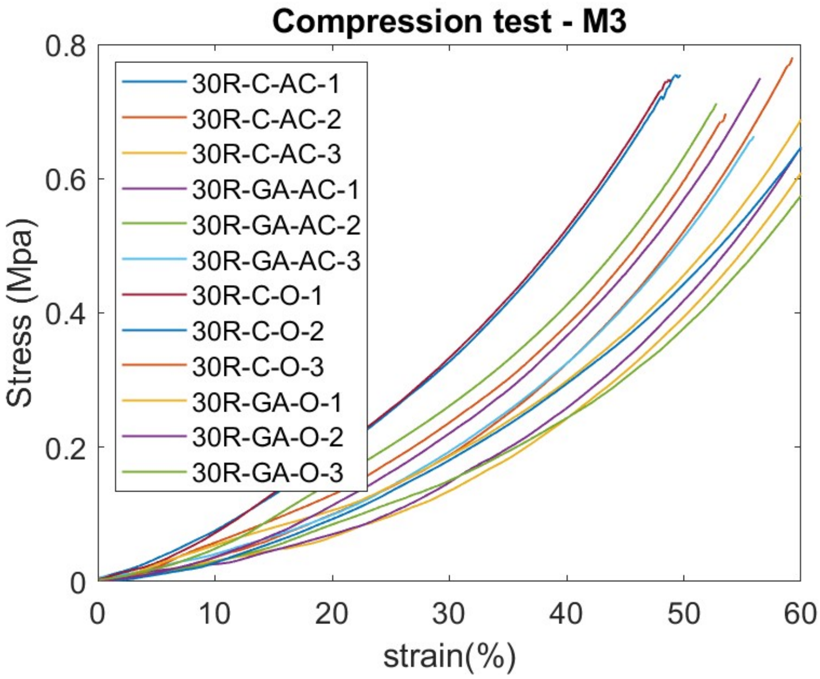


Figure A.26: Compression test of hemp stalk-cardboard durability M3 samples

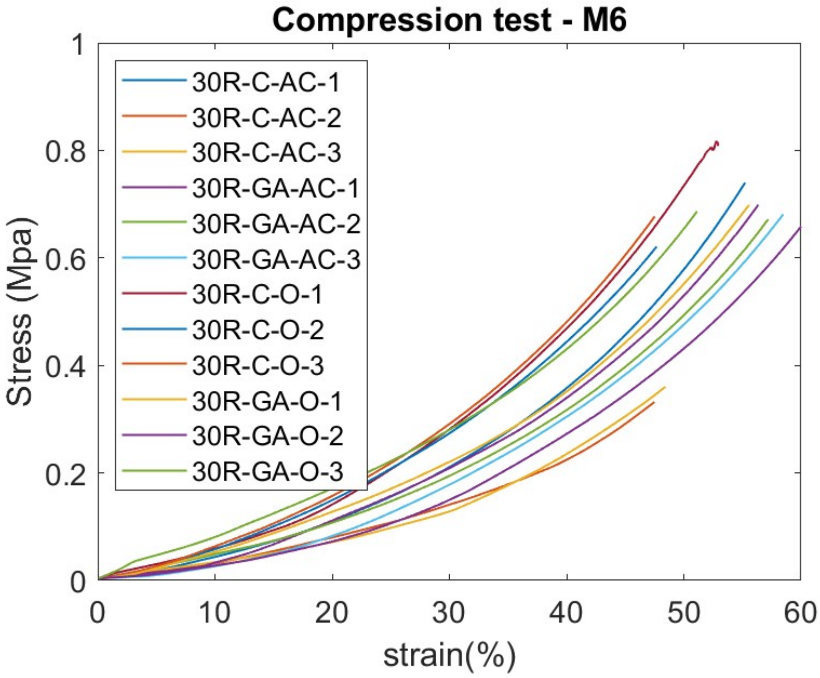


Figure A.27: Compression test of hemp stalk-cardboard durability M6 samples

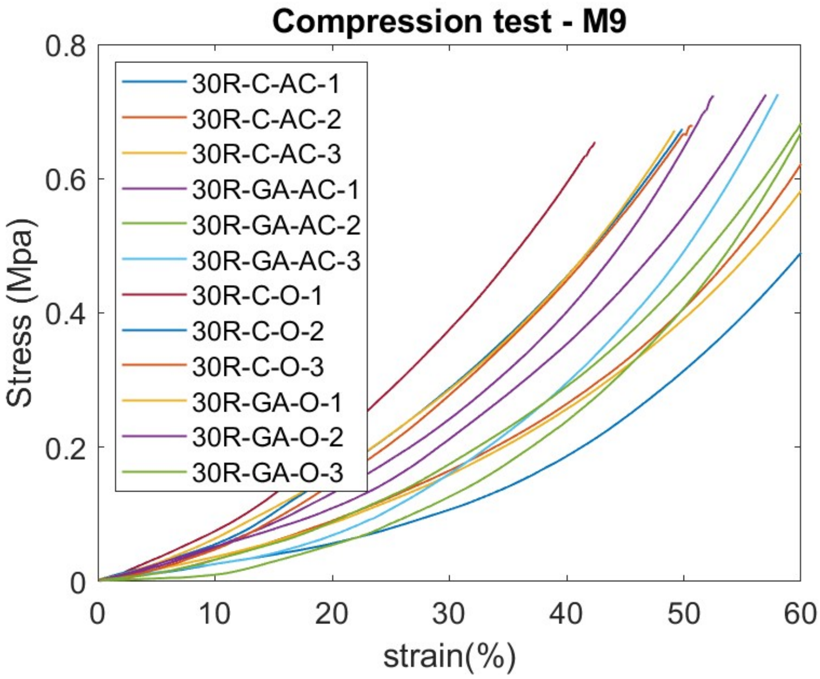


Figure A.28: Compression test of hemp stalk-cardboard durability M9 samples

A.5 Shear. Static puncture test - EN 12236

The next figures represent the raw data of the shear test performed to the insulating panels.

The behavior can be categorized into three phases. Initially, the curve exhibits curvature attributed to both the mechanism and material adjustment, as the sample isn't entirely flat. The second phase corresponds to the linear behavior of the material, enabling mechanical calculations until it reaches its maximum load and fractures. The third phase depicts material sliding resulting from its breakage across the thickness until the plunger completely traverses the material.

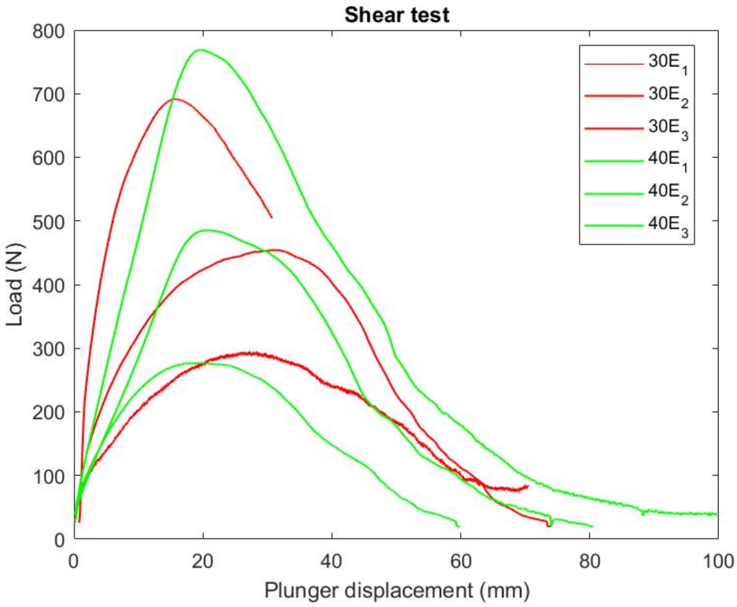


Figure A.29: Shear test of eucalyptus-hemp stalk samples

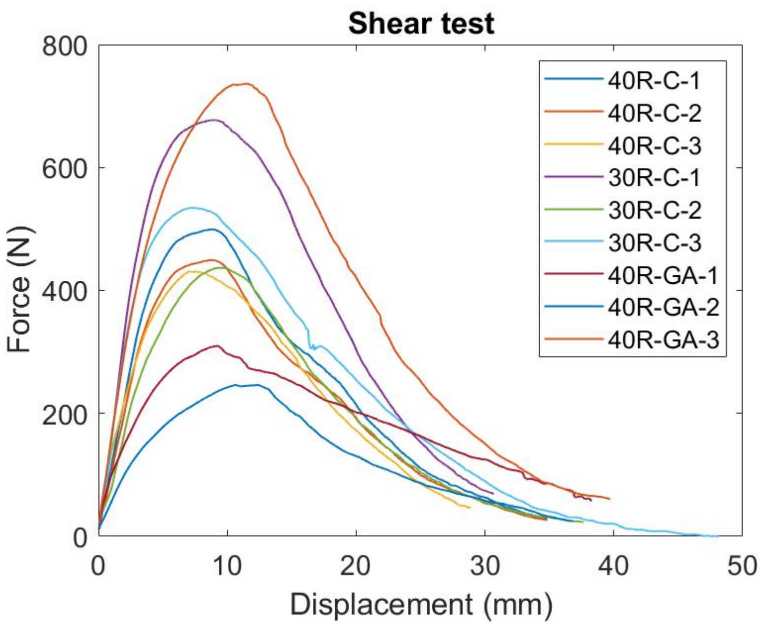


Figure A.30: Shear test of hemp stalk-cardboard samples

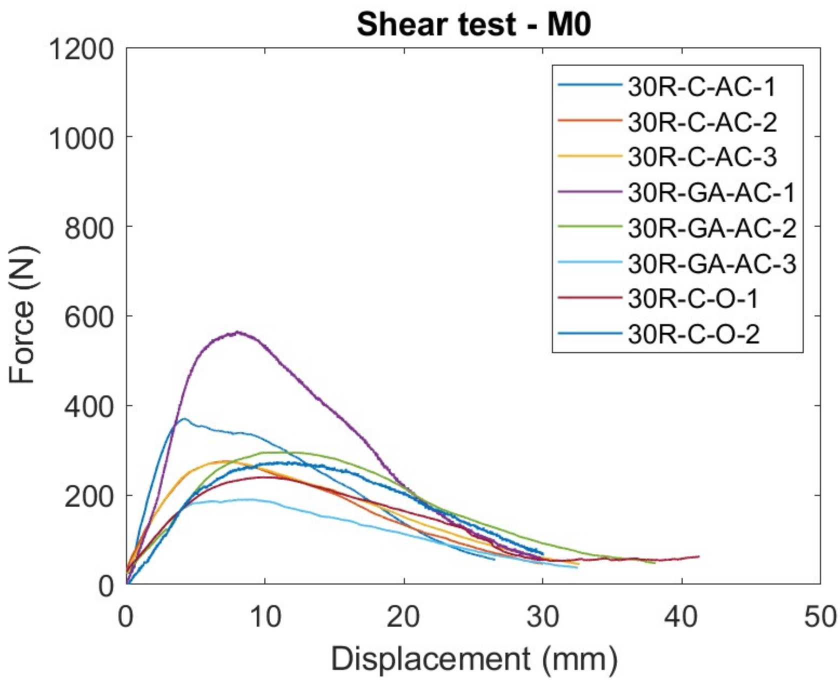


Figure A.31: Shear test of hemp stalk-cardboard durability M0 samples

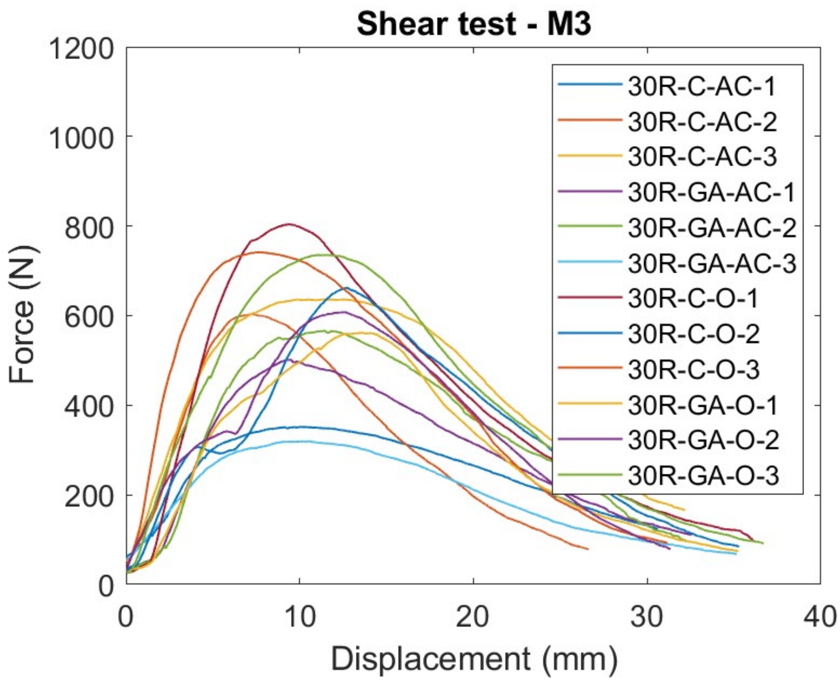


Figure A.32: Shear test of hemp stalk-cardboard durability M3 samples

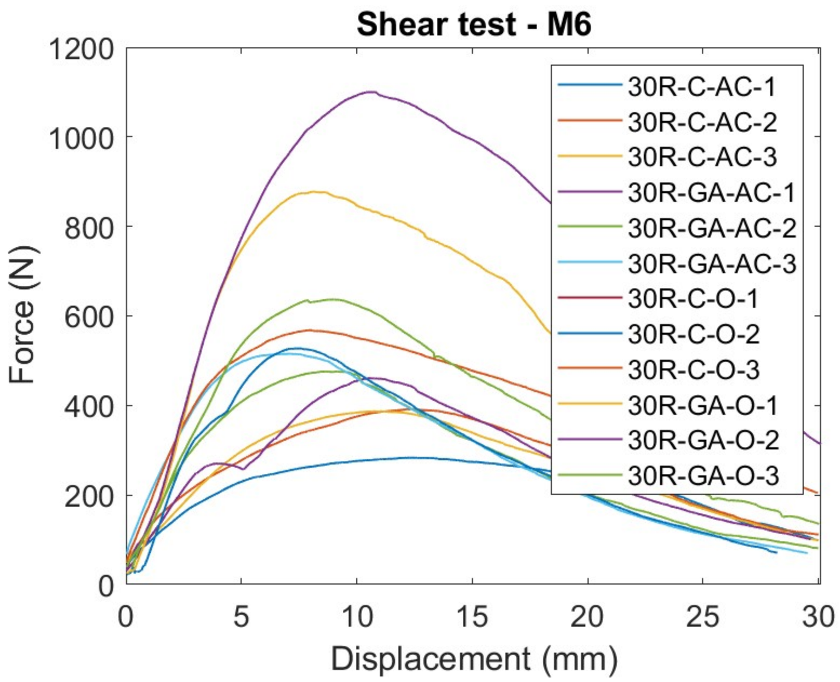


Figure A.33: Shear test of hemp stalk-cardboard durability M6 samples

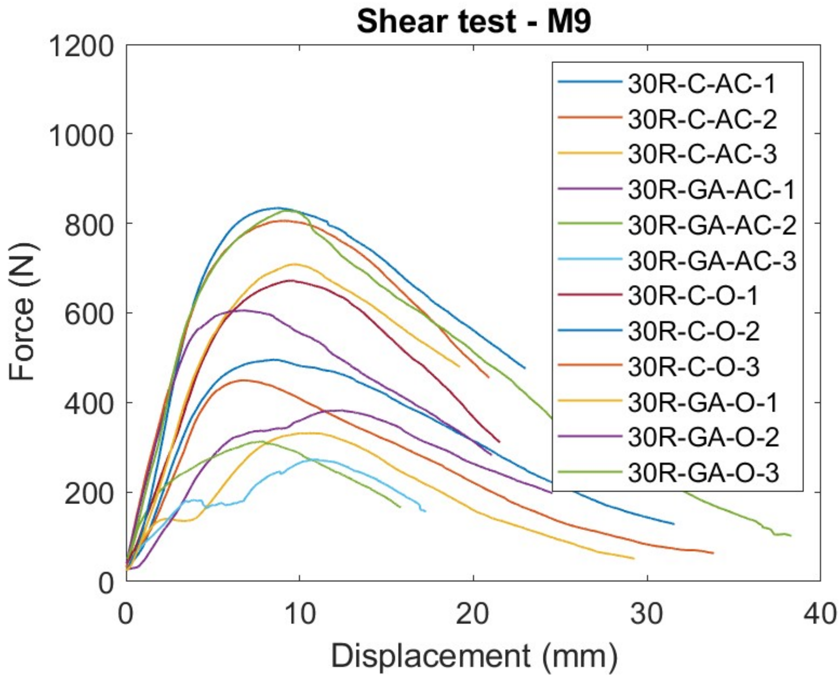


Figure A.34: Shear test of hemp stalk-cardboard durability M9 samples

A.6 Bending test - EN 12089

The next figures represent the raw data of the bending test performed to the insulating panels.

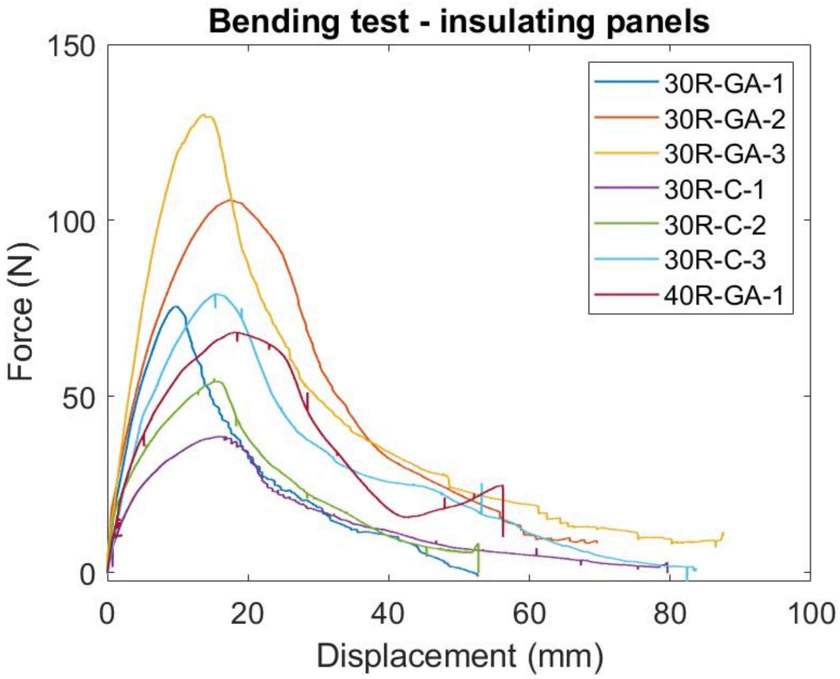


Figure A.35: Bending test of hemp stalk-cardboard samples

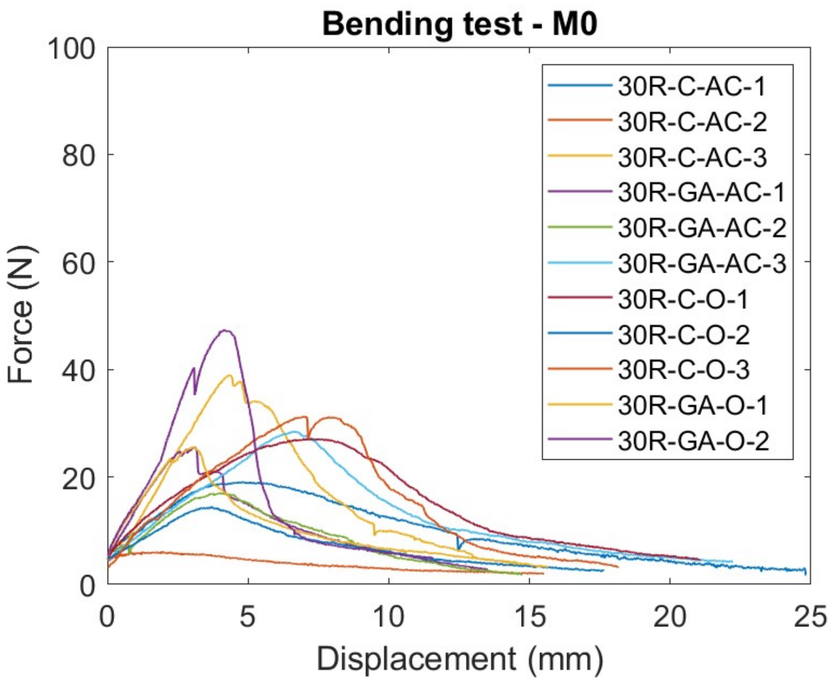


Figure A.36: Bending test of hemp stalk-cardboard durability M0 samples

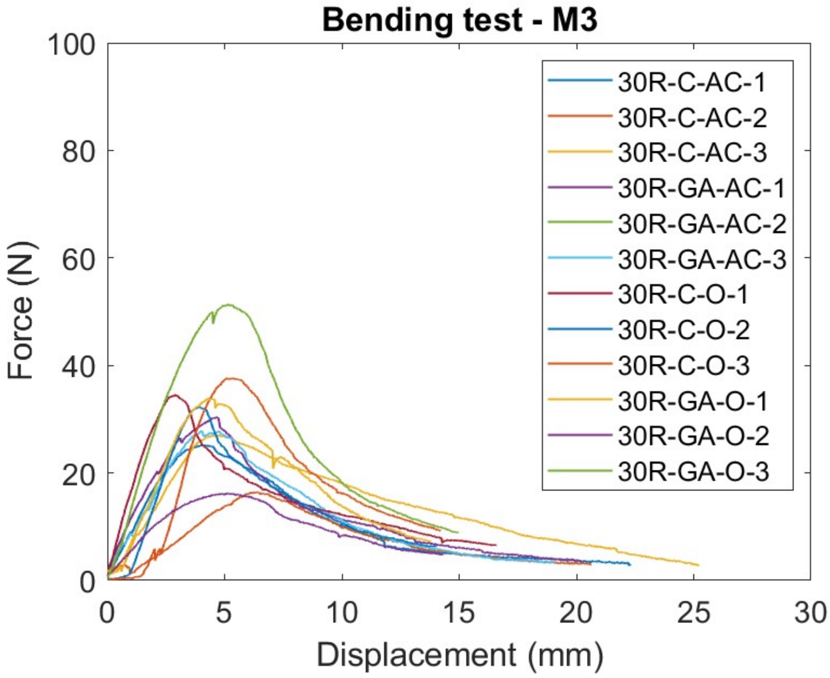


Figure A.37: Bending test of hemp stalk-cardboard durability M3 samples

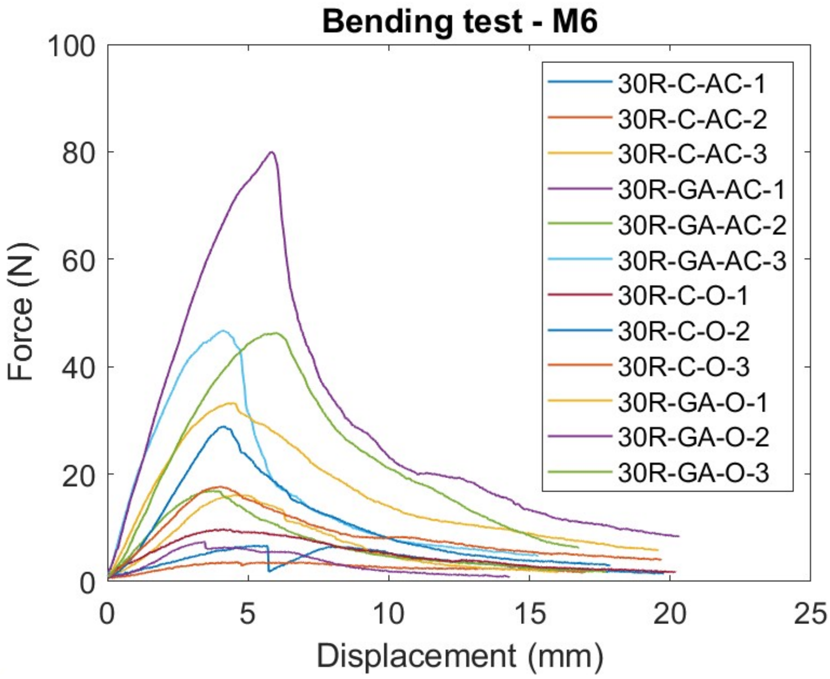


Figure A.38: Bending test of hemp stalk-cardboard durability M6 samples

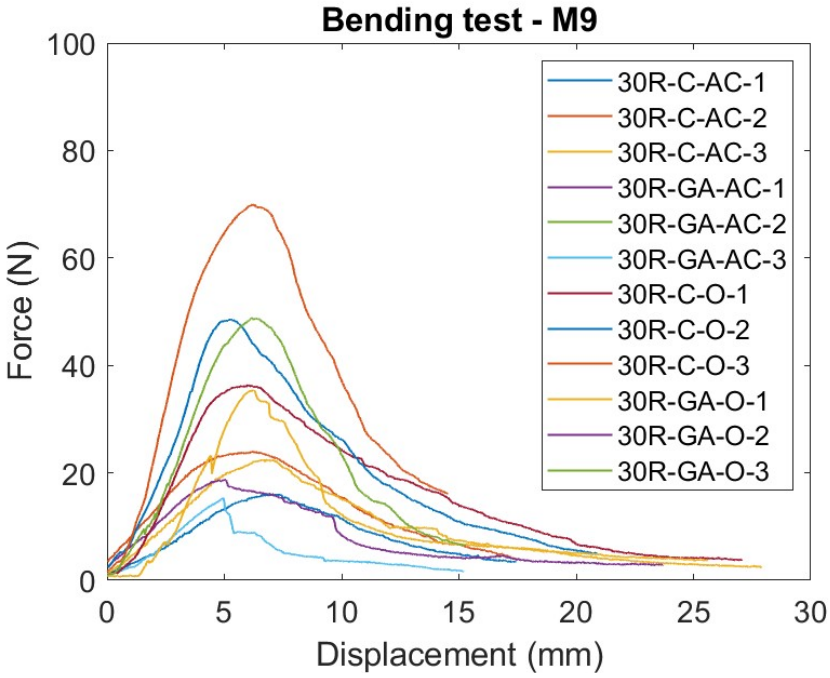


Figure A.39: Bending test of hemp stalk-cardboard durability M9 samples

A.7 Tensile test - ISO 1607

The next figure represent the raw data of the tensile test performed to the insulating panels.

The curve represents the material's elastic behavior until it fractures. However, for sample t1, post-processing data was not included due to an identified pre-failure of the material, evident in the curve by anomalous data points.

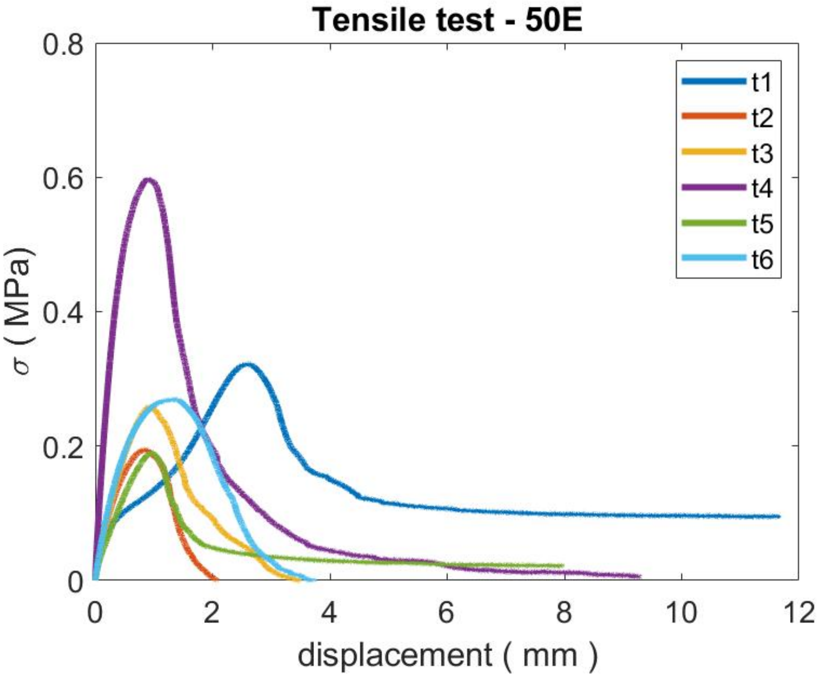


Figure A.40: Tensile test of eucalyptus-hemp stalk samples

A.8 Microscopy

This section will showcase all the figures depicting the material's microstructure, aiming to ensure the consistency of the presented results across all samples rather than being limited to a specific point. To obtain the figure a x4 objective lens was used.

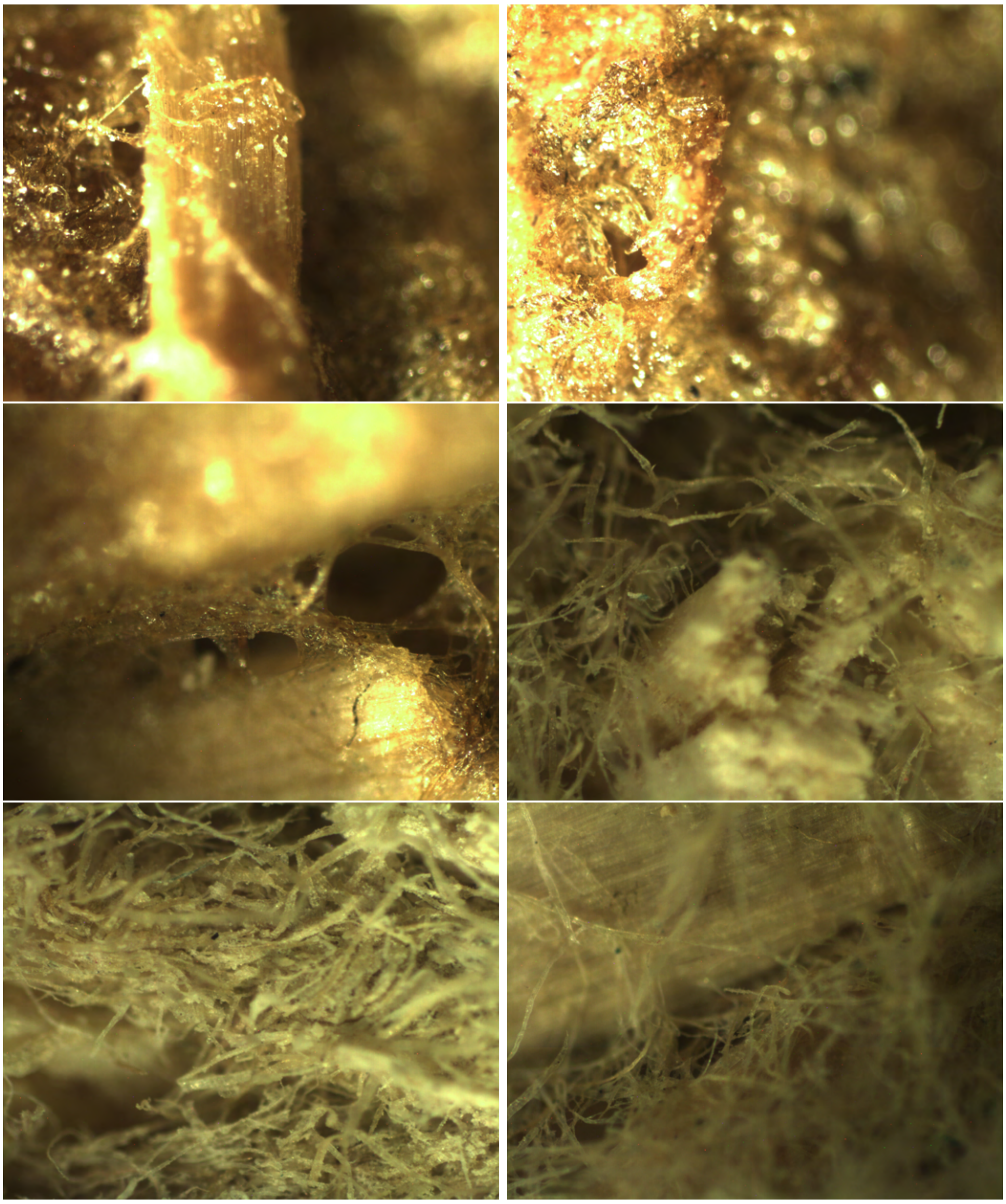


Figure A.41: Sample 30R-H-C-AC

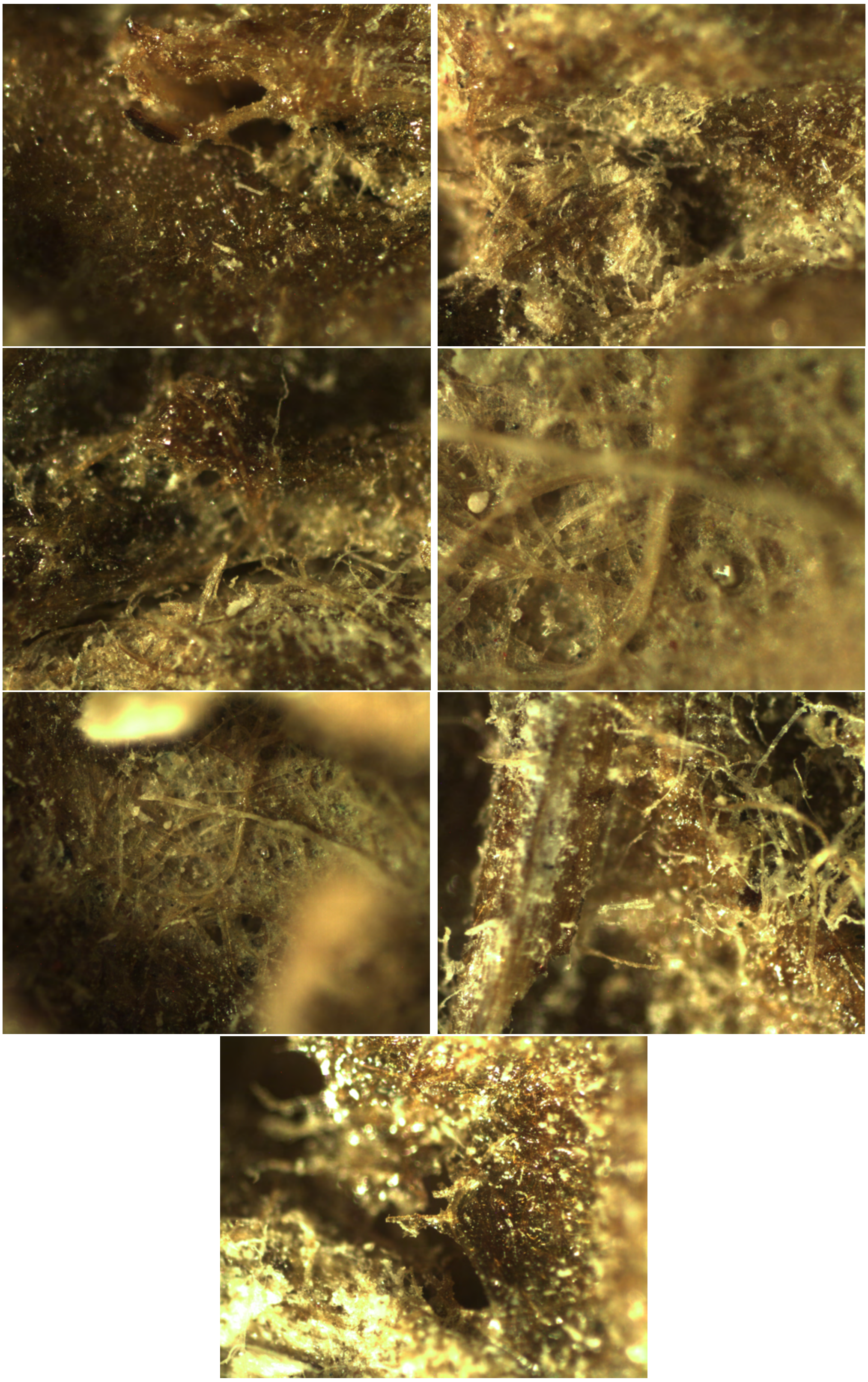


Figure A.42: Sample 30R-H-C-0

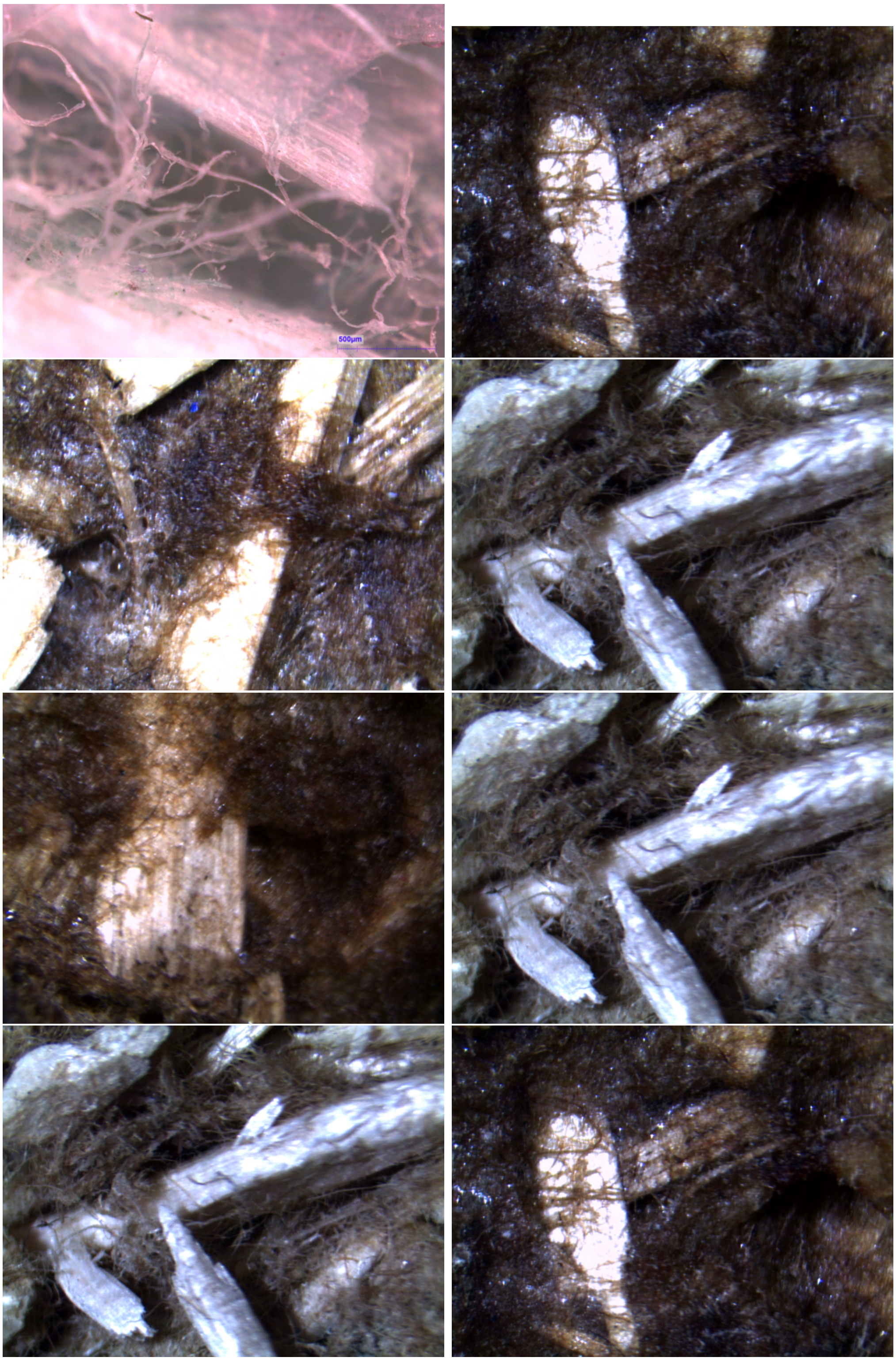


Figure A.43: Sample 30R-H-GA-AC

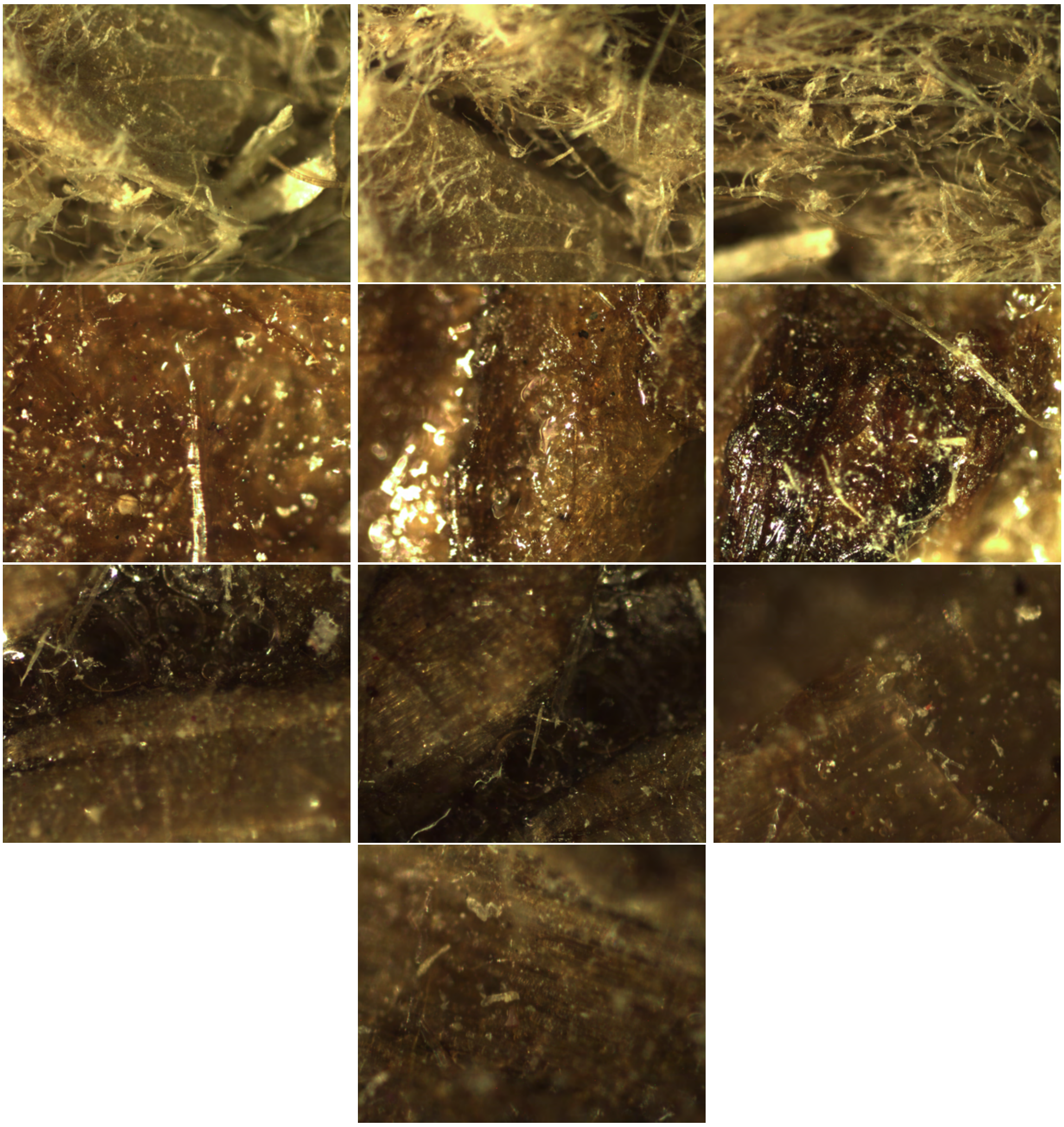


Figure A.44: Sample 30R-H-GA-0

A.9 Moisture Buffer Value - NORDTEST project

The next figure represent the raw data of the compression test performed to the insulating panels.

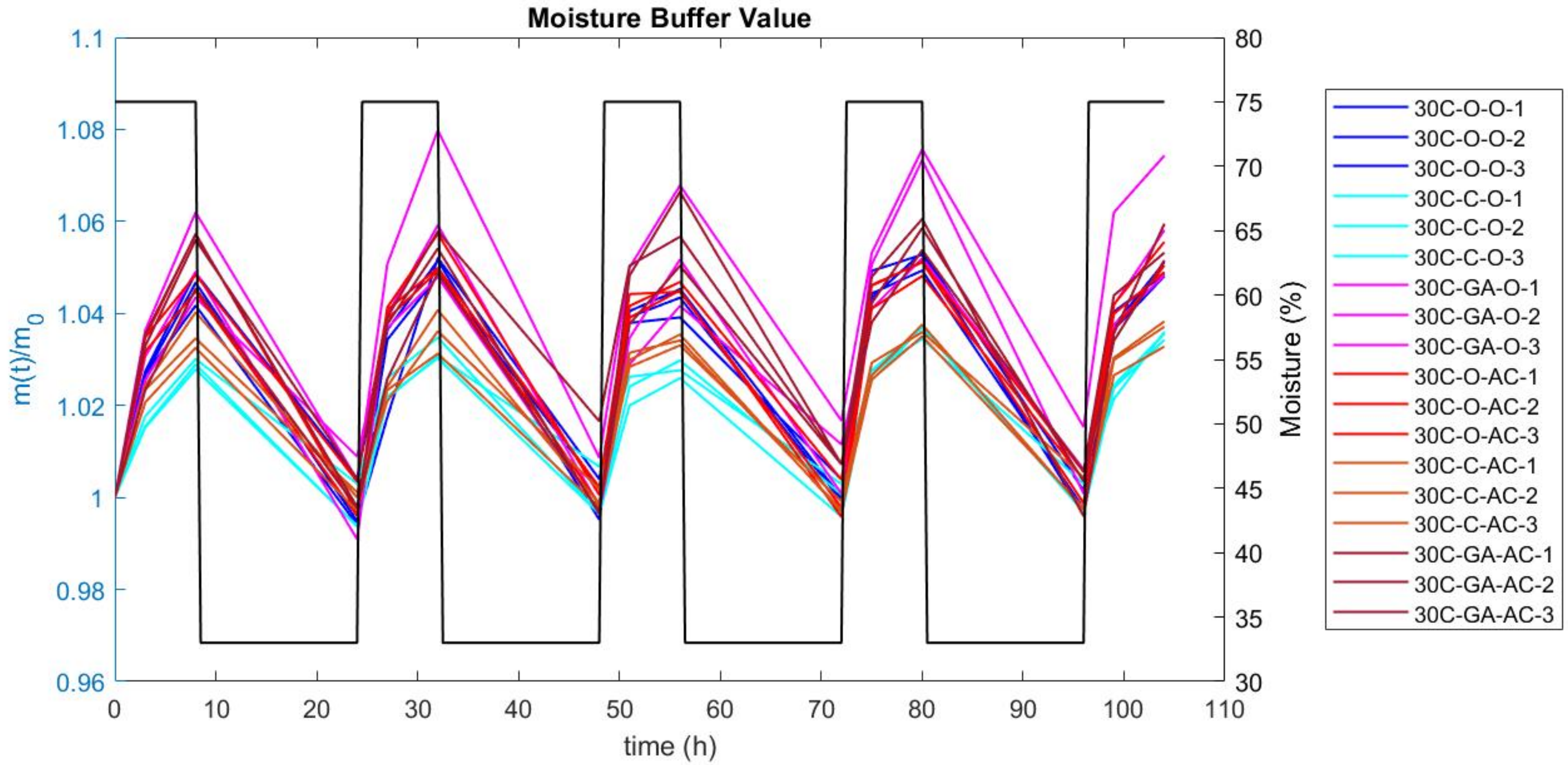


Figure A.45: Weight of the different samples during the MBV test

A.10 Tensile test - EN 319

In this section, we will present the raw data from the tensile tests conducted as part of the characterization process for the medium-density sample. Initially, all the resins used will be introduced, which include:

- White glue
- Bioepoxy
- Colophony
- Arabic gum
- Corn Starch
- Formaldehyde

After the preliminary studies have been presented, the focus will shift towards optimizing the manufacturing process of the samples. The samples that demonstrate the most effective manufacturing process for each type of resin will be highlighted. Ultimately, a comprehensive comparison of all the best-case scenarios will be provided.

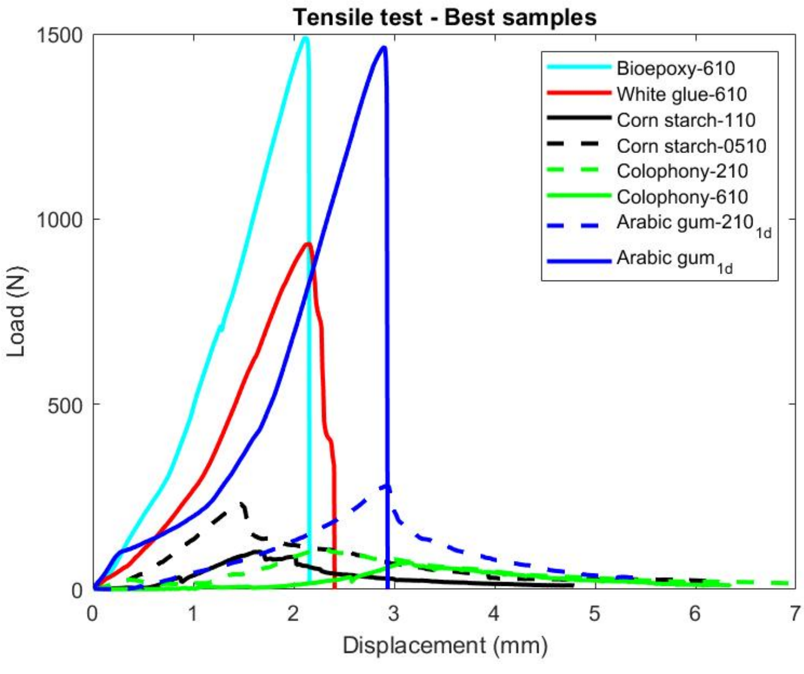


Figure A.46: Tensile test

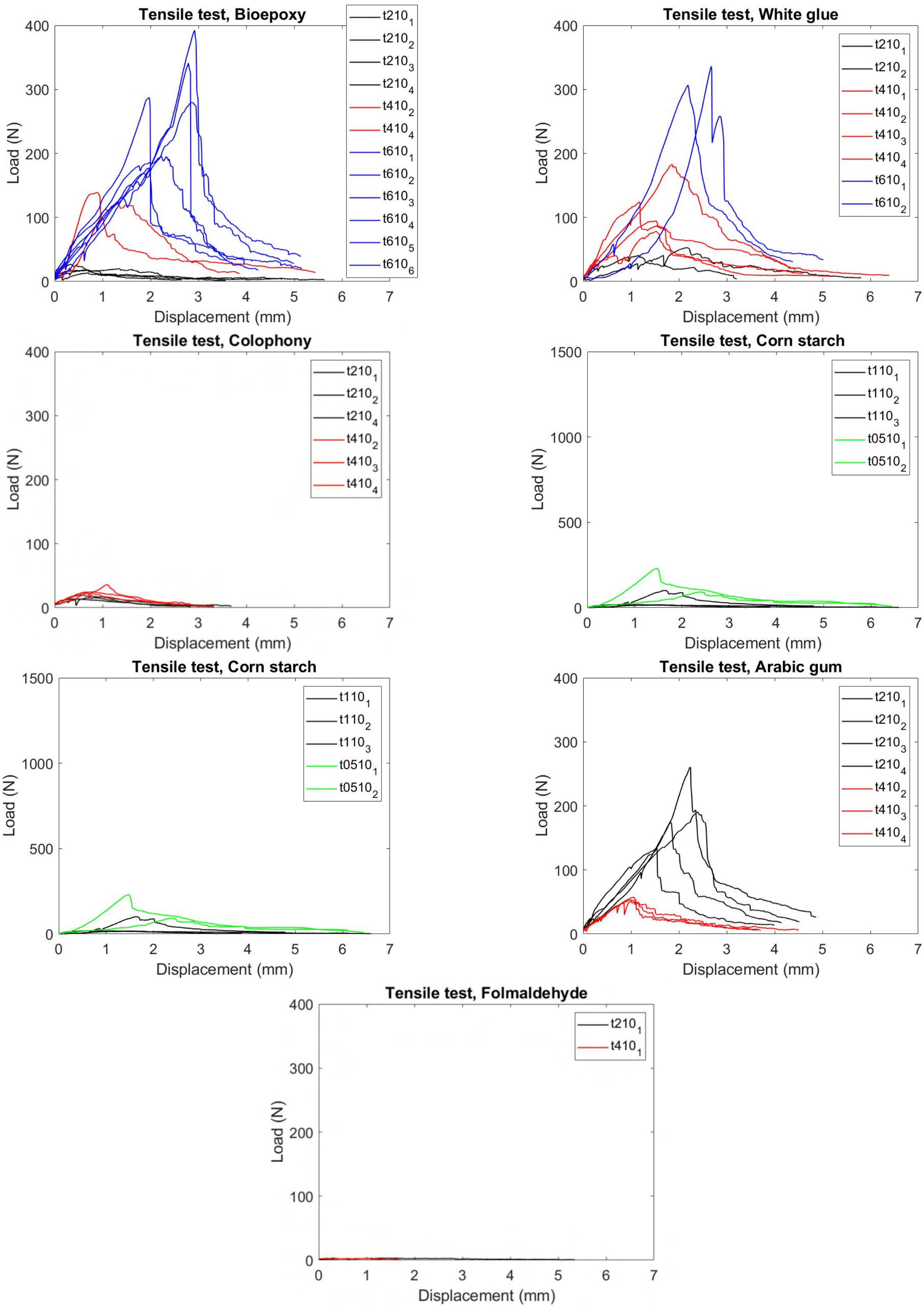


Figure A.47: Tensile test - first samples

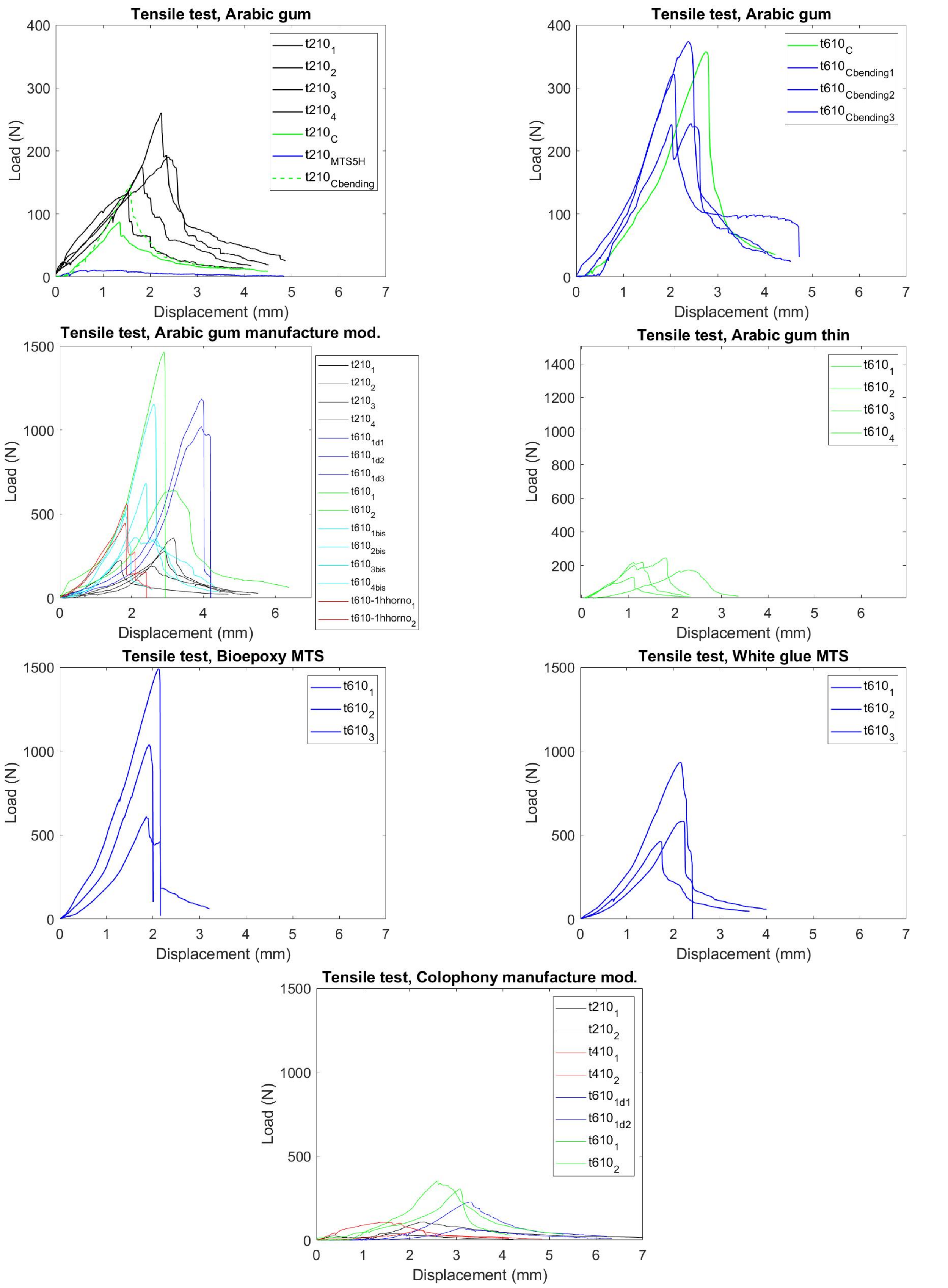


Figure A.48: Tensile test - manufacture modifications

A.11 Bending test - EN 310

In this section, we will present the raw data from the bending tests conducted as part of the characterization process for the medium-density sample. Initially, all the resins used will be introduced, which include:

- White glue
- Bioepoxy
- Colophony
- Arabic gum
- Corn Starch
- Formaldehyde

After the preliminary studies have been presented, the focus will shift towards optimizing the manufacturing process of the samples. The samples that demonstrate the most effective manufacturing process for each type of resin will be highlighted. Ultimately, a comprehensive comparison of all the best-case scenarios will be provided.

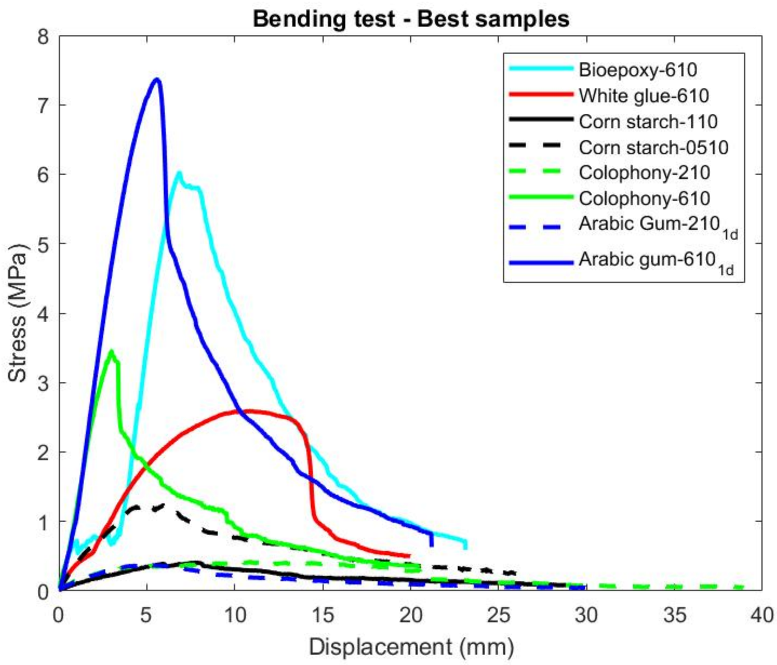


Figure A.49: Bending test - Best samples

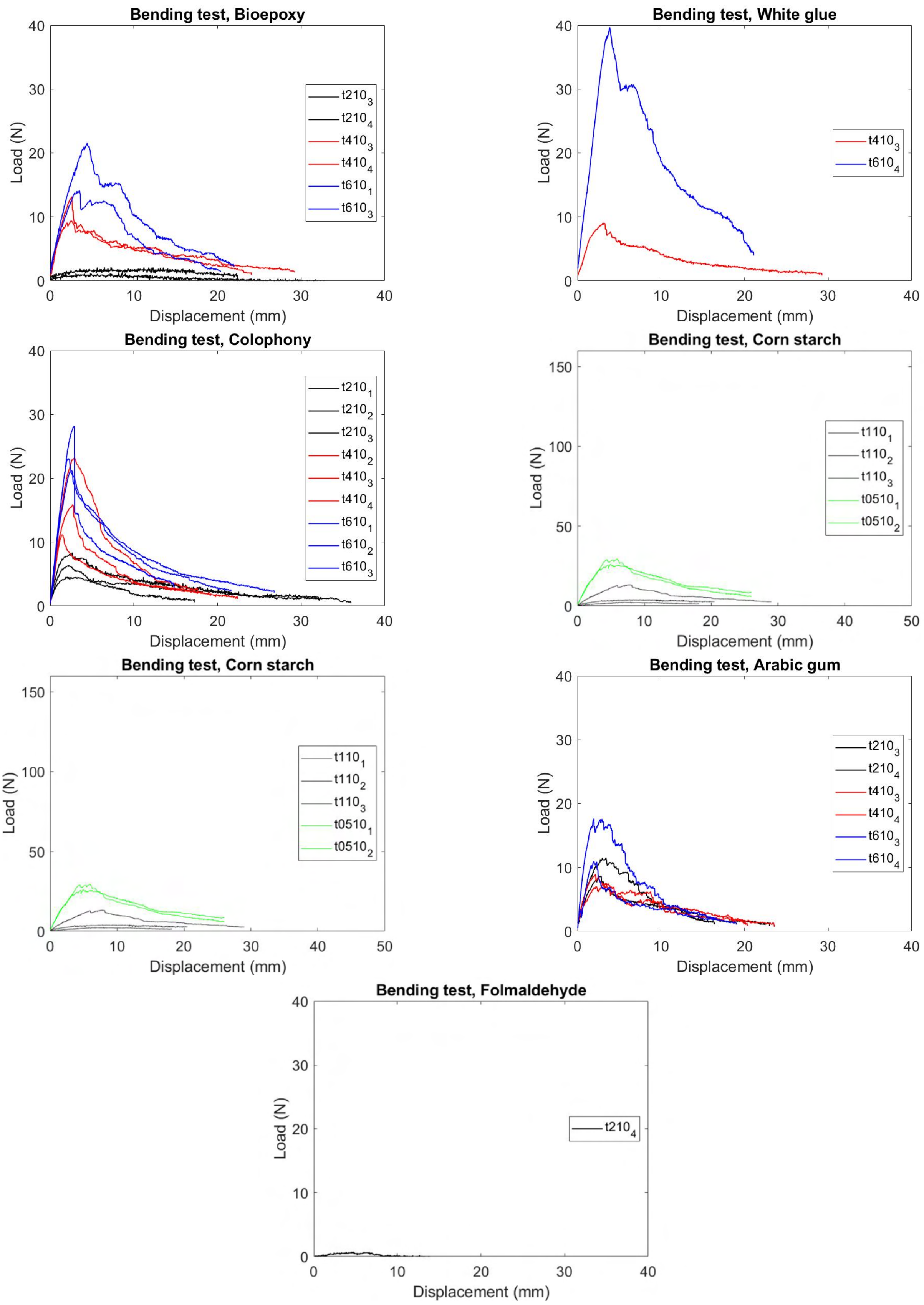


Figure A.50: Bending test - first samples

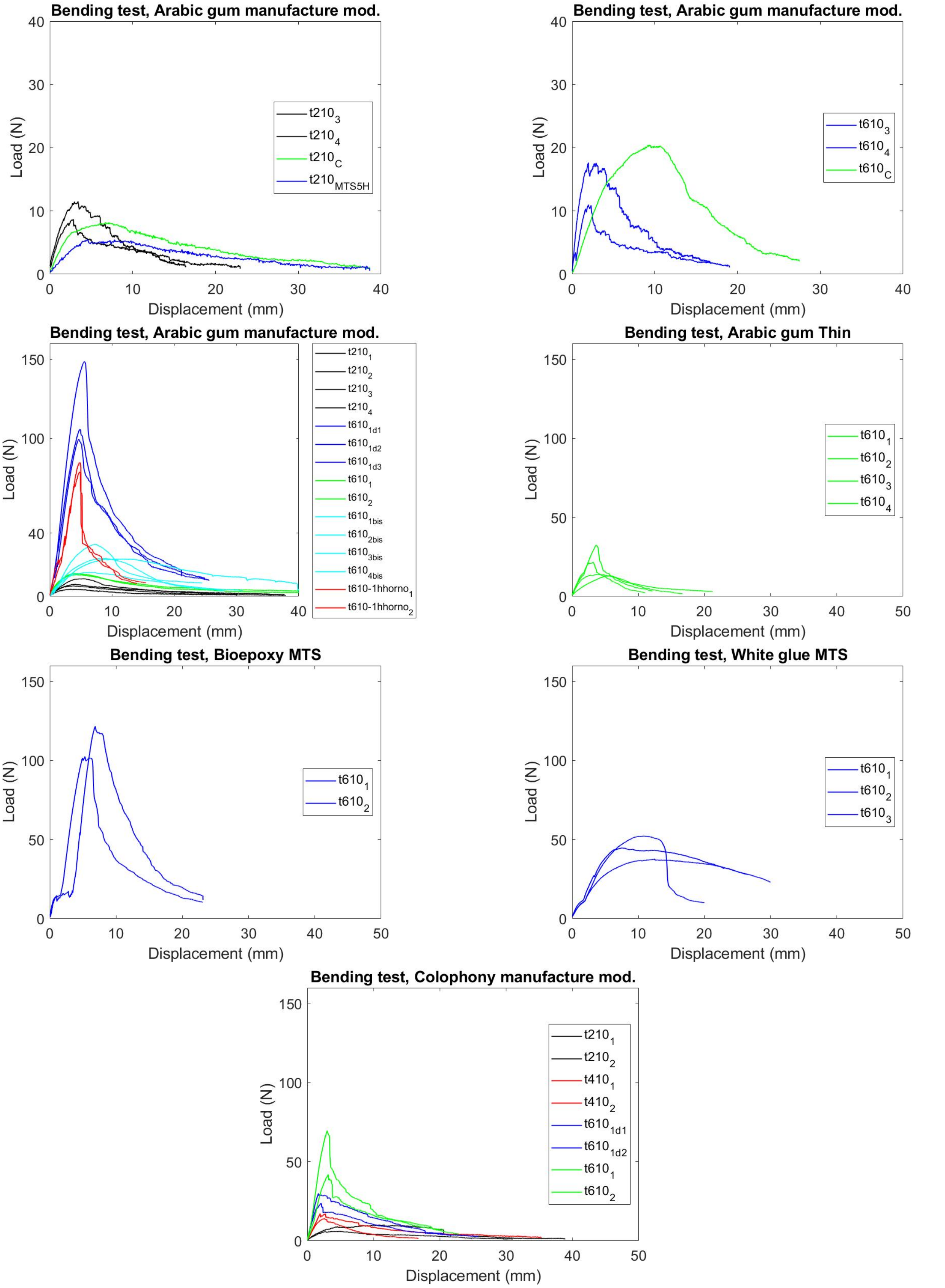


Figure A.51: Bending test - manufacture modifications

Appendix B

Technical data sheet

B.1 Bioepoxy ONE

	ONF FAST	ONS SLOW
MECHANICAL DATA		
Tensile Modulus (ASTM D638)	2.7 GPa	3.2 GPa
Tensile Strength (ASTM D638)	53.2 MPa	67.6 MPa
Elongation (ASTM D638)	6%	6%
Flexural Modulus (ASTM D790)	2.5 GPa	3.0 GPa
Flexural Strength (ASTM D790)	82.1 MPa	100.5 MPa
Compression Strength (ASTM D695)	77.9 MPa	86.3 MPa
Tg Ultimate (DSC, midpoint)	63°C	53°C
Hardness (Shore D)	70-80	70-80
PROCESSING DATA		
Mix Ratio (by volume)	2:1	2:1
Mix Ratio (by weight)	100:43	100:43
Viscosity (A/B/Mixed @ 25 °C)	1870/120/1020 mPas	1870/140/1060 mPas
Component Density (specific density @ 25°C)	1.14 (resin), 0.98 (hardener) gcm^{-3}	1.14 (resin), 0.98 (hardener) gcm^{-3}
Mixed Density (specific density @ 25°C)	1.09 gcm^{-3}	1.08 gcm^{-3}
Pot Life (@ 25°C)	18 min	43 min
Tack Free Time (@ 35°C)	3 hrs	8 hrs
Recommended Full Cure	7 days @ 25°C	7 days @ 25°C, Post cure recommended
ENVIRONMENT DATA		
VOC Content (ASTM D2369)	21.0 g/l	7.7 g/l
Biobased Carbon Content (ASTM D6866)	28%	21%

Figure B.1: Datasheet Bioepoxy ONE

B.2 White glue, HM-425

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Adhesivos, Selladores, Siliconas e Impermeabilizantes

AENOR ER Empresa Registrada ISO 9001 — AENOR Gestión Ambiental CGM-02/048 — IQNet Certified Management System

BRIK CEN HM-425

DESCRIPCIÓN

Cola sintética de poliacetato de vinilo.

PROPIEDADES

- Temperatura de aplicación +5ºC a +25ºC
- Alta calidad
- Resistencia a la tracción elevada
- Secado rápido

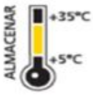
APLICACIONES

- Uso general.
- Industria del mueble.
- Ensamblado y trabajos de carpintería y
- ebanistería.
- Fabricación de puertas: armado, rechapado, etc.

* Consultar disponibilidad de otros colores y formatos

Envases	Capacidad	Caducidad
Autoaplicador	100 g/250 g/500g /750 g	24 meses
Tarro	1 kg	24 meses
Garrafa	6kg, 12 kg y 23 kg	24 meses
Contenedor	1000 kg	24 meses

* Conservar en lugar fresco y seco



CARACTERÍSTICAS TÉCNICAS

Característica	Método	Unidades	Valores
Aspecto	VISUAL		Líquido blanco
Contenido en sólidos	UNE 53356	%	57 ± 2
Viscosidad	UNE EN ISO 2555	mPa.s	15.000 ± 2.000
Resistencia a la tracción	EN 205	N/mm2	10 ± 2
pH			6± 1

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Figure B.2: Datasheet white glue HM-425 1/2

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SOLUCIONES PROFESIONALES
Adhesivos, Selladores, Siliconas e Impermeabilizantes

MODO DE EMPLEO

Remover el producto antes de su utilización. A continuación, aplicar una capa fina de adhesivo sobre una de las superficies a unir, se puede aplicar mediante métodos manuales (brocha, llanas, etc..) o también mecánicamente (encoladoras de rodillos). Someter ambas partes a presión (el tiempo abierto y de prensado varía según las condiciones ambientales). Las superficies para unir deben estar exentas de polvo, aceites y grasas.

Producto cuyo prensado se puede realizar tanto en prensa fría como de plato caliente, adjuntamos tabla orientativa de temperatura tiempo.

TIPO DE PRENSA	HOJA DE MADERA	ESTRATIFICADO MELAMINICO
FRIO	2 h	2 h y 30 min
80ºC	3 min	3 min
100 ºC	1 min	1 min y 30 seg

.

ADHESIÓN

Excelente adhesión sobre la mayoría de los diferentes tipos de madera, tableros de madera, DM, aglomerado, contrachapado, cartón.

LIMPIEZA

Limpieza con agua cuando el producto esté sin endurecer.

Una vez endurecido la limpieza es de forma mecánica.

SEGURIDAD E HIGIENE

Ficha de seguridad a disposición del cliente.

NOTA

La información proporcionada en esta ficha técnica y en particular las recomendaciones relativas a la aplicación , uso final del producto y asesoramiento del departamento técnico (de forma verbal o escrita) son dadas de buena fe y basadas en nuestro conocimiento actual y experiencia (cuando los productos son correctamente almacenados, utilizados y aplicados en condiciones óptimas dentro de su vida útil).
En la práctica , las posibles diferencias en los materiales, soportes y condiciones reales en el lugar de aplicación son tales, que no se puede deducir de la información ,de este documento ni de cualquier recomendación escrita o verbal, ninguna garantía en términos de comercialización o idealidad para propósito particulares ni obligación alguna fuera de cualquier relación legal que pudiera existir.
El usuario de los productos debe realizar las pruebas para comprobar su idoneidad de acuerdo al uso que le quiere dar. Nuestra garantía se limita exclusivamente a asegurar la calidad del producto suministrado conforme a nuestros estándares de calidad declinando toda responsabilidad en lo que se refiere a resultados obtenidos y a posibles perjuicios procedentes de un uso incorrecto o no adecuado.
En el caso que Quiadsa fuera considerada responsable en virtud de cualquier fundamento jurídico, la responsabilidad de la misma en ningún caso superará el importe de la entrega correspondiente. Quiadsa se reserva el derecho de cambiar las propiedades de sus productos. Los usuarios deben de conocer y utilizar la versión última y actualizada de las fichas técnicas de los productos, mediante su solicitud a nuestro departamento o consulta en la web www.quiadsa.com

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Figure B.3: Datasheet white glue HM-425 2/2

B.3 Citric acid

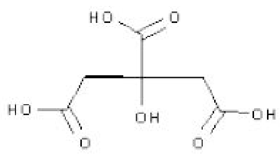
Ed.: 4 . Vig.: 14.05.2019 . Prod.: 131018

PanReac AppliChem ITW Reagents

PRODUCT CODE: 131018

Citric Acid 1-hydrate (Reag. USP) for analysis, ACS, ISO

$C_6H_8O_7.H_2O$



M.= 210,14 CAS [5949-29-1] EINECS 201-069-1
TARIC 2918 14 00 00

SYNONYMS: 2-Hydroxy-1,2,3-Propanetricarboxilic Acid

PHYSICAL DATA: Small crystals, in water at 20°C D 1,542 • M.P.: 135 °C • pH(50g/l)1,8 •
BIBLIOGRAPHY: Merck Index **13,** 2.350 Safety **2 , 892 D** • Römp **8 , 879** • Beilstein **3 , 556 IV , 1272** • BRN 4018641 • ACS **XI** • ISO 6353/2-1983 R - 8 , 10 • BP.**2018** • USP **42** • Ph. Eur. **8.0** (2014) **9.0** (2017) • F.C.C **11** • BOE **243**(8-10-2009) • Regulation (EU) nº 231/2012 •

HAZARDOUS: RTECS: GE 7810000 • LD50 ipr rat 375 mg/kg

H: H319 •
P: P264 • P280 • P305+P351+P338 • P337+P313 •

SPECIFICATIONS:

Assay (Acidim.)	99,5-102,0%
Identity :	

Figure B.4: Datashhet citric acid 1/2

Identity	IR passes test
Maximum limit of impurities	
Insoluble matter in H2O	0,005 %
Darkened substances by H2SO4	passes test
Residue on ignition (as SO4)	0,02 %
Chloride (Cl)	0,0005%
Sulfur compounds (as SO4)	0,002 %
Phosphate (PO4)	0,001 %
Oxalate (C2O4)	0,05%
As	0,00001%
Heavy metals (as Pb)	0,0005%
Metals by ICP [in mg/Kg (ppm)]	
Al	2
Au	2
B	2
Ba	2
Be	2
Ca	20
Cd	2
Co	2
Cr	2
Cu	0,5
Fe	3
Ge	2
In	2
K	50
Mg	5
Mn	2
Mo	2
Na	50
Ni	2
Pb	2
Pt	2
Sb	2
Si	10
Sn	5
Sr	2
Ti	2
Tl	2
V	2
Zn	2
Zr	2

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Figure B.5: Datasheet citric acid 2/2