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LIGNOCELLULOSE AND POLYLACTIDE FIBRES IN ACOUSTIC NONWOVENS AND COMPOSITE MATERIALS

Summary of the Doctoral Thesis



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Faculty of Materials Science and Applied Chemistry Institute of Design Technologies

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DOCTORAL THESIS PROPOSED TO RIGA TECHNICAL UNIVERSITY FOR THE PROMOTION TO THE SCIENTIFIC DEGREE OF DOCTOR OF SCIENCE

To be granted the scientific degree of Doctor of Science (Ph. D.), the present Doctoral Thesis has been submitted for the defence at the open meeting of RTU Promotion Council on 22 November 2023 at 10.00 at the Faculty of Materials Science and Applied Chemistry of Riga Technical University, 6 Ķīpsalas Street 6, Room 206.

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DECLARATION OF ACADEMIC INTEGRITY

I hereby declare that the Doctoral Thesis submitted for review to Riga Technical University for promotion to the scientific degree of Doctor of Science (Ph. D.) is my own. I confirm that this Doctoral Thesis has not been submitted to any other university for promotion to a scientific degree.

Name Surname (signature) Date:

The Doctoral Thesis has been written in Latvian. It consists of an Introduction, 3 chapters, Conclusions, 109 figures, 40 tables, 1 appendix; the total number of pages is 160, including the appendix. The Bibliography contains 165 titles.

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INTRODUCTION

Due to European Parliament and Council directives such as 2000/53/EC [1], 2018/849 [2], 2018/850 [3], 2018/851 [4] and people's "green thinking", the demand for materials is increasing, which are made from renewable resources and would be able to biodegrade after their end of use. The usefulness of the newly obtained materials has so far been mainly evaluated by their performance and costs, underestimating the aspect of environmental protection. For the creation of a sustainable material, not only the origin of the raw materials is important, but also the resources involved in their extraction (including the distance from the place of extraction of the raw materials that make up the material to the place of manufacture of the finished product), for the operation and management of the finished product at the end of operation.

In the development of materials that promote consumer demand for higher comfort during use, the increasingly dominant tendency is to increase their overall dimensions, mass, and installation without reducing technical performance or even making it better. At the same time, the replacement of non-renewable resources in the composition of many materials with renewable resources is facilitated by the requirements established by the directives of the European Parliament and the Council. Plant fibres such as hemp and flax, which are useful for reducing product mass, are a renewable resource. Due to the climatic conditions, both plants are suitable for industrial cultivation in Latvia; however, cultivation is not common. One of the main obstacles to the industrial cultivation of hemp and flax fibres in Latvia is that the most modern and the nearest primary and secondary processing factory is located in Lithuania.

This does not allow the assessment of the practical use of hemp and flax fibres.

Referring to the above-mentioned, **the aim of the Doctoral Thesis** is to create a soundabsorbing, environmentally friendly nonwoven of lignocellulose origin and polylactide fibre arrangement and composite materials based on them.

The part of the Doctoral Thesis related to the creation of the material composition and the production of samples was carried out at the Institute of Textile Technology of the Rhine-Westphalia University of Technology Aachen (Institut für Textiltechnik of RWTH Aachen University), which specialises in the production of composite materials for use in car interior decoration (ITA 2021), so in the the doctoral work, the possibility of using the developed materials in car construction is discussed.

Directive 2000/53/EC of the European Parliament and of the Council [1] regulates the life cycle of vehicles and has set minimum goals for the reuse of vehicle components and deadlines for achieving them, while Directive 2018/850 [3] on landfills determines a reduced amount of waste to be deposited in landfills.

In order to implement the set goal, the following tasks have been defined:

- Collect information about the most frequently used textile materials in the interior of a passenger car, traditional materials used, production technologies and technical requirements.
- 2. Collect and compare information about the possibilities of using hemp and flax fibres in nonwoven and composite materials.

- To collect information about partly and fully biodegradable thermoplastic polymers and compare them: incorporation technology into nonwovens, quality of polymer and natural fibre interlinking, and market price.
- 4. Choose the most suitable polymer matrix and the most suitable proportion of fibres for the production of nonwoven and composite materials, and plan the most appropriate structure of the nonwoven material.
- 5. Make samples of nonwoven and composite materials.
- 6. Analyse the technical, visual, mechanical, acoustic and other properties of nonwovens.
- Analyse technical, visual, and mechanical properties of composite materials and analyse their practical useability (including for use in the automotive industry – interior parts) and carry out an environmental impact assessment.

Actuality of the Thesis

The European Council (EP) Directive 2000/53/EC [1], which regulates the life cycle of vehicles, has set the minimum goals for the reuse of vehicle components and the deadlines for their achievement. Achieving the goals set by the directive promotes the usage of natural fibres in construction materials. Use of natural fibres, such as hemp, flax, etc. in constructions, replaces fibres from petroleum products, promotes the use of renewable resources, and reduces the mass of the material. Reducing the total mass of the vehicle reduces fuel consumption, which would be a step towards the Regulation of the European Parliament and the Council of May 30, 2018, No. 2018/842/EU [5] on binding annual reductions in greenhouse gas emissions that the Member States must achieve from 2021 to 2030. Savings in fuel consumption also provide significant financial savings.

Scientific novelty

The nonwoven (NM) and the composite materials based on it are made from renewable resources (plant fibres from hemp and flax) and polylactide, the production of which requires 20–30 % less energy than petroleum-based polymers. The raw materials of the manufactured materials are fully biodegradable; the structure of the materials consists of multi-layered layers prepared by mixing the raw materials using the low-pressure laying (airlaid) method (not the carding method) and the mechanical needle punching method. The hot-pressing method is used in the production of the composite materials. The production technology does not involve the use of water in any step.

The novelty of the Doctoral Thesis is not only the development technology of the fully biodegradable material production within the framework of the doctoral research, but also the assessment of its impact on the environment.

Due to the components and construction structures of the nonwoven material, the variety of the developed material applicability is expanding. The same nonwoven material can be used in various car parts that need to provide sound absorption and damping. The same nonwoven material can be used to make composite material parts, which have greater rigidity, shape retention and load-bearing properties. The novelty is the reduced mass of composite materials using the plant based fibres in it.

The use of light materials also in the car interior to reduce the total vehicle mass is considered a priority, as it improves the overall energy efficiency of the vehicle. Composite materials made from nonwoven materials of natural origin are becoming a common interior decoration material due to their properties (light material, sound insulation) [6].

Practical significance of the Thesis

Within the framework of the Doctoral Thesis, nonwoven and composite material was developed. It consists of two components: 40 wt. % of plant fibres containing cellulose and 60 wt. % – polylactide fibres. Latvia has suitable climatic conditions for obtaining these plant fibres, thus promoting the development of agriculture in the country. The production of nonwoven and composite materials can be considered as a creation of added value for plant fibres grown in Latvia. A new nonwoven material and composite from raw materials obtained from renewable resources was developed in the Doctoral Thesis. The obtained materials can be used to make certain parts for the automotive industry.

Theses to be defended

- The blend of fibres created from renewable resources and the structure of the multilayer material created from it allow the production of two materials that differ in consistency and purpose of usage: nonwoven acoustic materials and composite materials based on them.
- 2. In the developed fibre blends of both compositions (Hemp-PLA and Flax-PLA) for the lignocellulose component, 40 wt. % is a sufficient mass proportion so that the nonwoven and composite materials made from it can be integrated into the passenger car interior structure.
- The manufacturing process of the components and materials making up the Hemp-PLA (or Flax-PLA) fibre blend produces a lower total amount of harmful emissions than composites, the production of which traditionally uses polymer fibres from fossil resources.

Approbation of the results

The results of the Doctoral Thesis have been published in peer-reviewed scientific journals and conference proceedings and reported and discussed at international and local conferences.

The author's reports at scientific conferences

 1. 11th International Scientific Practical Conference "Environment. Technology. Resources", Rezekne Academy of Technologies, Rezekne, Latvia, June 15–17, 2017.

- 2. Riga Technical University 57th International Scientific Conference "Materials Science and Applied Chemistry" (MSAC 2016), Riga, Latvia, October 21–22, 2016.
- 3. 15th International Scientific Conference "Engineering for Rural Development", Jelgava, Latvia, May 25–27, 2016.
- 4. RTU 56th International Scientific Conference, Riga, Latvia, 14-16 October 2015.
- RTU 56th International Student Science and Techology Conference, Riga, Latvia, 8 May 2015.
- 6. RTU 55th International Scientific Conference, Riga, Latvia, 17 October 2014.
- 13th International Conference on Global Research and Education "Inter Academia 2014", Riga, Latvia, September 10–12, 2014.

Publications of the author of the Thesis on the topic of the Doctoral Thesis

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- Seile, A., Beļakova, D. Usage of Hemp Fibres in Nonwoven Sandwich Type Structure and Composite Material Production. In: *Abstracts of the Riga Technical University 56th International Scientific Conference: Section: Materials Science and Applied Chemistry*, *October 14–16, 2015, Riga, Latvia.* Riga: RTU Press, 2015, pp. 57–57. ISBN 978-9934-10-733-7.

Publications of the author of the work unrelated to the topic of the Doctoral Thesis

- Seile, A., Kukle, S. Sustav zbrinjavanja rabljenog tekstila. *Tekstil*, 2012, 61 (7–12), str. 333–339. ISSN 0492-5882. (SCOPUS)
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Other activities related to the Doctoral Thesis

ERASMUS internship from 01.10.2013 until 31.05.2014 at the Institute of Textile Technology (ITA) of the Aachen Rhine-Westphalia University of Technology, Germany.

Participation in the Riga Technical University scientific research project for young scientists No. ZP-2016/31 (researcher).

Participation in SAM 8.2.2. In the 3rd round of project "Strengthening of doctoral students and academic staff of Riga Technical University and Banking Graduate School in areas of strategic specialization".

Participation in the academic year grant of Riga Technical University doctoral programme 2021/2022.

1. LITERATURE OVERVIEW

Nonwovens (NM) in passenger car interior

Textile materials in passenger car construction

Textile materials in the car body make up more than 20 kg and 43 parts [7]. In the car interior, the areas of use of textile materials are divided into three categories: passenger compartment (example.g., floor carpet, control panel insulation, ceiling lining, door decoration, and seats), luggage compartment (side decoration, luggage compartment floor, floor decoration, luggage compartment parcel tray (shelf panel)) and engine compartment (e.g. bonnet insulator and absorber). Whereas, textile materials used in car construction are divided into two categories: cabin interior surface materials and cabin decoration materials [6]. The car's parcel tray (Fig. 1.1) is also one of the interior details – interior decoration material.



Fig. 1. 1. The structure of the passenger car and its main elements [8].

Technical requirements of passenger car interior parts

One of the factors characterising textile materials is its surface density. The surface density of textile materials used in car construction depends on the material's function, raw materials, and manufacturing technology. For example, surface density for fabrics is 200–400 g/m², for velvet 360–450 g/m², and for knitted fabric 160–370 g/m² [7]. The car interior ceiling upholstery PET and PP nonwovens surface density is usually around 200 g/m²[7], [9].

When introducing new materials and solutions in the automotive industry, they must meet a number of technical requirements [10]:

- materials in the cabin are visually harmoniously coordinated and give a quality impression;
- the interior of a new car must look the same after three years of operation under normal conditions as it did at the time of purchase;
- the cabin should be easy to clean;
- materials must age uniformly;
- materials must not smell unpleasant;
- surface materials serve flawlessly for four years of warranty;
- materials do not make noise when in contact with each other;
- interior finishing materials are arranged in such a way that they do not create noise.

All textiles and leathers used in car interiors must be certified according to OEKO TEX 100, product class 4 [10].

Application of natural fibres in the production of car interior parts

The choice in favour of natural fibres usage in the production of textile parts of the vehicle interior is driven by environmental and economic factors. Environmental factors include the EP directive 2009/28/EC [11], which determines the use of energy from renewable resources. On the other hand, the demand for fuel consumption reduction promotes the use of light materials in car construction [12].

Noises in a passenger car

The manufacturing method of textile materials used in the interior of passenger cars is mainly determined by the function of the manufactured part. The materials used in the interior of the car also have a sound-absorbing function, which is best provided by materials with a porous structure. The structure with air inclusions can be provided by the structure of the nonwoven.

Figure 1.2 shows the noise sources of a moving car, arranged in order of highest noise level: tires, exhaust system, intake system, and engine. The analysis of frequency content helps to better understand the nature of noise sources. A detailed analysis of each major noise source is necessary to identify and reduce critical noise drivers.



Fig. 1.2. Layout of noise sources according to the noise test of a moving car [13].

Hemp and flax fibres as components of nonwoven and composite materials

Hemp and flax are similar in their mechanical properties. The chemical composition of both fibre plants is also similar (Tables 1.1 and 1.2). In the structure of both hemp and flax fibres, the main component proportionally by mass is cellulose, since it is the main component of the cell sheaths. Cellulose in plants (both hemp and flax) is related to hemicellulose and lignin. The fibre structure also consists of wax, protein, water, minerals and water-soluble compounds [14]–[15]. Higher lignin content makes the fibres less sensitive to chemicals. For example, hemp fibres are resistant to bases and acids [16].

Table 1.1

Cellulose	Hemicellulose	Lignin	Pectin	Wax, protein, water,	Reference
				ash, etc.	
(%)	(%)	(%)	(%)	(%)	(%)
67–78	17	3.5-5.5			[17]
77				10 % hygroscopic water	[18]
				0.5 % ash, 1.7 % water-	
80.2	12	2.6	3	soluble compounds, 65 %	[19]
				moisture	
64.2-70.5	16.99-23.79	5.68-7.96	1.37-1.64	0.52-0.73	[20]
66.20	10.04	5 60	1.54	0.52	Bialobrzeskie
00.38	10.04	5.08	1.34	0.32	(B LV) [20]
70.54	16.00	6 10	1.64	0.72	Bialobrzeskie
/0.54	10.99	0.18	1.04	0.73	(B PL) [20]

Chemical Composition of Hemp Fibre

The main differences are observed when the growing conditions are changed. The advantage of hemp is the length of the plant, which allows to obtain a higher yield of green mass per unit area than in the case of flax. The advantage of flax is the tradition in its harvesting technology, including the availability of these technologies.

Table 1.2

Cellulose	Hemicellulose	Lignin	Pectin	Wax, protein, water,	Reference
(%)	(%)	(%)	(%)	ash, etc. (%)	
45-76	13–22	0.60-13	0.6–5	0.2–1.7	[21]
64.57-75.38	12.97-26.07	4.78-7.44	0.45-3.23	0.83-1.9	[20]
69.9	21.3	7.26	0.45	1.28	T36-5-7-94 1. līnija [20]
75.38	12.97	5.56	1.10	0.97	Ilona [20]
71.98	16.21	6.8	0.73	0.83	Nike [20]

Chemical Composition of Flax Fibre

Partially and fully biodegradable polymer matrices

For the selection of the composite matrix among the various polymers, the compatibility of the properties of the specific polymer with the intended function, economic and environmental factors are considered.

In Table 1.3, the properties of competing polymers are compared according to the function of application. Of the polymers compared in the table, only PLA is obtained from renewable resources and is completely biodegradable. The other three polymers (PP, hard PVC, and PET) are obtained from non-renewable petroleum products. All the synthetic polymers mentioned above have a high recycling potential. Products from recycled PP and PET are becoming more and more popular in industrial production.

Among the compared polymers, PLA is the most expensive, and its economic cost (in May 2018, the price of PLA fibres varied from 2.69 EUR/kg to 3.20 EUR/kg (3.20 USD/kg to 3.80 USD/kg [22]) is one of the main factors inhibiting its wider use. In terms of density, PLA is close to rigid PVC and PET. Among all four polymers, PLA has the lowest crack fracture resistance, and the particle hardness according to the Vickers hardness test is the second highest after PET. PP is a widely used polymer in the automotive industry; however, the comparison of its technical properties shows lower values than PET, hard PVC and PLA.

PLA fibres have several properties similar to the other polymers discussed above. The mechanical properties of PLA are similar to those of PET. Due to the low melting and softening temperature, PLA is comparable to PP [23].

The tensile properties of PLA are very different from the high strength PET. The initial tensile modulus of PLA at 2 % elongation is very similar to other textile fibres. The stress yield point is very pronounced, so its fibres stretch easily.

Compared to natural plant fibres, PLA has a higher elasticity, which, combined with low density, gives textiles lightness and a sense of airiness.

Table 1.3

Physical-mechanical and	Thermo-physical	Properties	of Polymers	[24]
5	1 2	1	2	L 1

Attributes	PET PP		PVC	PLA
Tashniaal Attributes			hard	PLLA
Demaiter (lag/ard)	1100 1010	800.020	1200 1500	1210 1240
Density (kg/m^3)	1190-1810	890-920	1300-1580	1210-1240
Elastic modulus (GPa)	1.6-4.4	2 [25]	2.14-4.14	3.45-3.8
Elongation (%)	1.30-5.00	100-600	11.93-80	5-7
(MPa \cdot m ^{0.5})	1.05-9.16	3.00-4.50	1.46-5.12	0.7-1.1
Vickers hardness (Hv)	11-40	6-11	10-15	14-18
Yield strength (MPa)	30-40	20.7-37.2	35.4-52.1	48-69
Tensile strength (MPa)	55 [25]	26 [25]	14-58 [25]	45 [26]
Service temperature (°C)	-20-160	-40-37.2	-20-70	70-80
Melting point $t_{kuš}$ (°C)	267 [27]	160–165	210	160 [23]–180 [25]
Glass-transition temperature t _{st} (°C)	67–81	-10 [28]	85	60–65 [29]
Specific heat capacity (J/(kg·K))	1160–1587	1870–1956	1355–1445	1180–1210
Thermal conductivity (W/(m·K))	0.28-0.58	0.11-0.17	0.15-0.29	0.12-0.15
Thermal expansion (10 ⁻⁶ /K)	115–170	122–180	1.80–180	126–145
Eco Attributes				
Energy content (MJ/kg)	89-95	76-84	77-83	52-54
Recycle potential	High	High	High	High
Technical Attributes				
Low to high pitch	7–8	6–7	7—7	Semi-crystalline
(0-10)				[29]
Transparent to opaque	Transparent to	Transparent	Transparent to	Transparent
Glossiness (%)	opaque	20–94	opaque	
		20 91		
Features Relative to Oth	ner Polymers			
Corrosion resistant	+ (By adding	+	+	
Corrosion resistant	additives)	I	I	
Amortization		+	+	
Approval from the U.S.		1	1	
Administration	+	Ŧ	— —	Т
Flame retardant	+		+	
Heavy		Light	Heavy	
Impact resistance		+	_	
Resilient	+	+	+	
Stiff			+	+

Table 1.3 continued

Attributos	DET	DD	PVC	PLA
Attributes	TEI	11	hard	PLLA
Strong	+		-	
UV resistant	+ (By adding additives)	-	+	
Smoothness		+		

Physical property studies [23] confirm the commercial potential of PLA fibres as textile fibres. The ecological and aesthetic properties of PLA are also not far behind and can compete with other polymers used so far.

Summary of Chapter 1

The main issue in writing the literature review was obtaining of the necessary data on the technical requirements in the auto industry, because every company has the right to accept its internal standards as commercial information.

The automotive industry in Europe is regulated by EU and EP regulations and directives. The development of the European Green Deal defines a strategy aimed at a modern transformation of the EU in terms of resource efficiency and a competitive economy, with special emphasis on a holistic approach to climate and environmental challenges. For example, the EP Directive 2009/28/EC sets the percentage of recycling and reuse of components that make up new cars. On the other hand, EU regulation 540/2014 regulates the noise level of the car. The reduction of the total mass of the car allows to save fuel or electricity consumption. For cars powered by fossil fuels, reduced fuel consumption also reduces CO_2 emissions, particulate matter, etc. volume of emissions.

In order to achieve the above-mentioned requirements dictated by the regulations, it is necessary to replace the raw materials used in the auto industry with raw materials of more environmentally friendly origin. It is necessary to reduce emissions by improving economic indicators. For example, by choosing plant fibres obtained in Latvia as the substitute raw materials, it is possible to solve the previously mentioned environmental and economic issues. It is necessary to create versatile materials, such as acoustic nonwovens, based on which composite materials can be made. Acoustic nonwoven and composite materials made from environmentally friendly raw materials could be used for the production and construction of car interior parts.

2. RESEARCH METHODOLOGY

The two-composition nonwoven and composite materials created within the framework of the Thesis are made of cellulose-containing plant fibres and PLA. For both material compositions, the same total amount of fibre by mass was used within one sample, keeping the proportion of fibres at 40 mass.% hemp or flax fibres and 60 mas.% PLA fibres. The manufactured NMs are labeled as Hemp nw for NM containing hemp and PLA fibres and

Flax_nm for NMs containing flax and PLA fibres. The manufactured composite materials are marked by analogy: Hemp_comp for the composition containing hemp and PLA fibres and Flax_comp for the composition containing flax and PLA fibres. The numbers attached to the labels represent the numbering of the sample.

Technology of production of nonwovens

If one fibre blend is planned to be used in both nonwovens and composite materials production, the structure planning plays an important role. In the composite structure, plant fibres form the framework of the material and absorb most of the applied load. Whereas the polymer matrix binds the material together, supports the fibres and distributes the load between the layers of the composite. Obtaining the composite requires the formation of nonwoven materials of both compositions from several layers of fibre coating, which are gradually combined into multilayer structures during the production of the material. In the production of multi-layer composite materials, recommendation has been followed to avoid an even number of layers of fibre covering, because under the influence of loading, the use of even layers can contribute to the delamination of the composite materials in the middle of the material layers.

Extraction of nonwovens

The process of manufacturing nonwovens includes a set of several work operations (Fig. 2.1): preparation of fibres for work (dissolving), mixing of fibres, production of layers of fibre covering (5 rounds), fixing of layers of fibre covering against disintegration by needling and with repeated needling of the covering of all fibres combining the rounds into one nonwoven material.



Fig. 2.1. Scheme of nonwoven material production processes.

The nonwovens of both compositions Hemp_nw and Flax_nw are made of a multilayer or so-called sandwich-type structure (Fig. 2.2), which consists of five layers of fibre covering.



Fig. 2.2. Multilayer structure of nonwoven material.

The ratio of natural fibres and polymer fibres in the nonwoven layers was chosen based on the previous experience of the Institute of Textile Technology (ITA) of the Aachen Rhine-Westphalia University of Technology in research on the production of natural fibre-reinforced composite materials [30] and considering the experience of other researchers in the production of plant fibre and polymer composite materials. Hargitai has researched that the optimal mechanical properties in hemp and PP fibre blend composite materials are achieved with a proportion of plant fibres of 40–50 % [31] by mass. Calculation of fibres to produce materials of both compositions was carried out according to Eqs. (2.1) and (2.2).

$$\frac{Mass_{PLA}}{Mass_{Hemp}} = \frac{\rho_{PLA}}{\rho_{Hemp}} \times \frac{V_{PLA}}{V_{Hemp}} \to \frac{1,27_{PLA}}{1_{Hemp}}$$
(2.1)

$$\frac{Mass_{PLA}}{Mass_{Flax}} = \frac{\rho_{PLA}}{\rho_{Flax}} \times \frac{V_{PLA}}{V_{Flax}} \to \frac{1,34_{PLA}}{1_{Flax}},$$
(2.2)

where

$$\begin{split} &\textit{Mass}_{PLA} - \text{the calculated mass of PLA, g;} \\ &\textit{Mass}_{Hemp} - \text{calculated hemp fibre mass, g;} \\ &\textit{Mass}_{Flax} - \text{calculated flax fibre mass, g;} \\ &\textit{P}_{PLA} - \text{density of PLA, kg/m^3;} \\ &\textit{P}_{Hemp} - \text{hemp fibre density, kg/m^3;} \\ &\textit{P}_{Flax} - \text{flax fibre density, kg/m^3;} \\ &\textit{V}_{PLA} - \text{PLA volume, 60 wt. \%;} \\ &\textit{V}_{Hemp} - \text{hemp fibre volume, 40 wt. \%;} \\ &\textit{V}_{Flax} - \text{flax fibre volume, 40 wt. \%.} \end{split}$$

The average length of the hemp fibres used in the study varies from 50-80 mm, the moisture content at the time of delivery is up to 12 %, while the impurity of straw is less than 6 %. Chemical composition of hemp fibre supplied: cellulose 60–72 %, hemicellulose 11–19 %, and lignin 2.3–4.7 % [32].

The fineness of the flax fibres used in the samples of the linen composition is 3.87 dtex, the average length is 139.23 mm.

 7.22 ± 0.55 den fine and 64 ± 4 mm long PLA fibres were used in the production of both compositions.

Fibre opening

The structure of the material is influenced by the technologies of nonwoven and composite materials manufacturing. In this case, the used fibre separation and mixing machine (Fig. 2.3) with three parallelly arranged work rollers and the vertical vacuum tunnel connected to the machine with the low-pressure conditions present in it are suitable for performing three consecutive work operations: fibre opening, mixing and production of finished fibre layers.



Fig. 2.3. Working principle of the TRÜTZSCHLER CVT3 1200 fibre opening and mixing machine [33].

Mixing of fibres and creation of fibre covering by low-pressure airlaid method

The fibre blend is mixed and the fibre layer is formed in the vertical vacuum tunnel attached to the fibre separation and mixing machine *TRÜTZSCHLER CVT3 1200*, which ensures the separation of the fibres from each other and uniform arrangement in different, random directions. With each repeated loading of the mixed fibre blend into the machine, the fibres are mixed more evenly. This machine is used for uniform mixing of hemp and PLA fibres for Hemp-PLA fibre layers and for flax and PLA fibres for Flax-PLA fibre layers. The mass distribution for nonwovens of Hemp_nw and Flax_nw fibres according to their constituent layers is shown in Tables 2.1 and 2.2.

Table 2.1

Fibre Mass Distribution by Layers for the Production of one Sample of Hemp_nw Nonwoven

	A layon of fibro	Fibr		
No.	covering	Hemp fibre (g)	PLA fibre (g)	Total (g)
1	PLA		87	87
2	Hemp-PLA	113	86	199
3	Hemp-PLA	113	86	199
4	Hemp-PLA	113	86	199
5	PLA		87	87
			Total (g):	771

For better mixing quality of two types of fibre, the fibres can be placed alternately on the feeding belt: 1 dose of natural fibres, 1 dose of PLA fibres, 1 dose of natural fibres, etc. (Fig. 2.4 a) – the first time of mixing Flax-PLA fibres). During the second time of mixing of fibres or/and during the production of a layer of fibre coating, the pre-mixed fibre blend is evenly distributed on the feed belt (Fig. 2.4 b)).

		Fibre		
No.	covering	Flax fibre (g)	PLA fibre (g)	Total (g)
1	PLA		88.5	88.5
2	Flax-PLA	110	88	198
3	Flax-PLA	110	88	198
4	Flax-PLA	110	88	198
5	PLA		88,5	88,5
			Total (g):	771

Fibre Mass Distribution by Layers for the Production of One Sample of Flax_nw Nonwoven



a)



b)





Fig. 2.5. Schematic of the fibre mixing process.

The fineness of the fibres within the formation of layer composition affects the number of processing times required in the machine (Fig. 2.5) for sufficient mixing of the mixed fibre blend. After opening the fibres and obtaining the mixed blends, the amount of fibres intended for each layer of the fibre coating is put into the machine for the last time to obtain the ready fibre layer.

First needle punching of the fibre layers

Fixing the layers of fibres and making the nonwoven material by the mechanical needling method is a productive and environmentally friendly method because, unlike other nonwoven material production methods, no water is used. Fixation provides protection of the prepared fibre layers from accidental disintegration, preparing the layer structure for further nonwoven material extraction.



Fig. 2.6. The principle of operation of a simple needle punching machine [34].

The needling of the fibre layers was performed on the DILO LBM 6 needling machine in the ITA laboratory. Figure 2.6 shows the role of the elements of the needling equipment in the work process. The fibre layers prepared for needle punching are fed to the punching work area (width is 600 mm). Hardened felting needles (2000–3000 needles/m) are embedded in the needlebeam (1 and 2). The needlebeam is attached to the upper crank (3), while it rotates, the needle plate pierces the fibre coating at a speed of 3000 stitches/min [35]. The needled nonwoven material moves between two horizontal plates: the lower bed plate (4) supports the needled material, the upper perforated (stripper) plate (5) ensures that the needles are lifted from the needled material. The finished perforated nonwoven material is compacted by two draw-off nip rollers (6).



Fig. 2.7. The principle of operation of the needle when punching [34].

In the needle punching process, the needles pierce all the fibre layers. As the needlebeam moves behind the needle barbs, the fibres in its path are caught, stitching through the vertical layer between the plates (Fig. 2.7).

The 5 layers of fibre, intended to produce each nonwoven material composition sample, turn into 3 layers after the first needling (Fig. 2.8): the outer PLA fibre layer is fixed together with the next layer of plant and PLA fibre blend. When needle punching or fixing these layers, observe the order of the layers: on the bottom – a layer of Hemp-PLA or Flax-PLA fibre blend, on top – a layer of PLA fibre.



Fig. 2.8. First needle punching of the fibre layers.

Second needle punching of the fibre layers

Needle punching of nonwoven material was performed on the same DILO LBM 6-needle punch machine as fixing of the fibre layers. Each ready-made nonwoven sample consists of 3 layers of needle-punched fleece, see Fig. 2.9.

After fixation, the layers of fleece obtained are trimmed, adjusting their dimensions to the needling machine, and arranged for the second needling, or nonwoven production. Place a 3rd fleece layer at the bottom (for the Hemp-PLA composition, PLA needle-punched together with the Hemp-PLA fibre layer for the Hemp_nw sample; for the Flax-PLA composition, PLA needle-punched together with the Flax-PLA fibre layer) with the PLA side down, a 2nd fleece layer (Hemp-PLA or Flax-PLA) is placed on top, with a 1st fleece layer on top (for the Hemp_nw composition, PLA needle-punched together with Hemp-PLA fibre layer; for the Flax nw composition, PLA needle-punched together with Flax-PLA fibre layer), see Fig. 2.10.



Fig. 2.9. Preparation of all fibre layers for the second needle punching.



Fig. 2.10. Arranging the fibre layers for the second needle punching.

Production of samples for testing nonwoven and creating composite materials

Six material sample strips, each 5 cm \times 30 cm, have been press-cut from the centre of each nonwoven sample. According to the strip cutting schemes of the nonwoven material samples of both compositions (Fig. 2.11), four strips (Nos. 1–4) are cut parallel to the working direction of the machine, two (Nos. 5–6) are cut perpendicular to the working direction of the machine.



Fig. 2.11. The scheme of cutting out the sample strips: for the Hemp_nw nonwoven sample (on the left); for the Flax_nw nonwoven sample (on the right).

Manufacturing technology of composite materials

The construction of the material structure considers the composite material manufacturing method (hot-pressing performed in the ITA laboratory with the hot press – C Press), as a layer of PLA polymer fibres is placed both on the top and bottom. In nonwoven material heat pressing, the melting PLA fibres cover the plant fibres and protect them from direct contact with the heating surfaces of the heat press. The technological parameters of the equipment were determined experimentally, based on the settings determined within the framework of the study *Development of Bio-composites for the Automotive Industry* (developed by ITA [30]). The final accepted working modes of the hot press are reflected in Table 2.3.

Table 2.3

Composition of composite material	Time (min.)	Pressure (bar)	Temperature (°C)
Hemp_comp	15	3.75	162
Flax_comp	15	3.75	162

Technological Parameters of the Hot Press

Test methods

Determination of geometric parameters of nonwoven and composite materials

All strips of nonwoven samples of both compositions (Hemp_nw and Flax_nw) produced within the framework of the study (including those from which composite strips are made) with dimensions of 5 cm \times 30 cm were cut out using dies.

The mass of the nonwoven and samples of composite materials of both compositions (Hemp_nw, Flax_nw, Hemp_comp, and Flax_comp) is determined in accordance with the standard DIN EN 12127:1997 (the current version of the standard in Latvian LVS EN 12127:2001 [36]) – textile, fabric standard for determining the mass per unit area, using small sample sizes.

The thickness of the Hemp_nw nonwoven and Flax_nw nonwoven sample strips used for the tensile test and all composite samples was determined following the standard DIN EN ISO 9073-2:1995 (ISO 9073-2:1995 [37]). This standard regulates nonwoven material test methods, including thickness determination under specific pressure.

For the study of acoustic properties, the thickness of Hemp_nw nonwoven circle-shaped samples Ø 20 mm was measured using the *SDL Atlas J100* thickness measuring device intended for textile materials.

Testing of tensile strength of nonwoven and composite materials

The tensile strength test of nonwoven and samples of composite materials was performed in the laboratory of the Institute of Textile Technology (ITA) of the Aachen Rhine-Westphalia University of Technology, according to the standard DIN EN 29073-3:1992-08 (textile products – nonwovens test methods: determination of tensile strength and elongation [38]). The tensile test was performed with the Z2.5 machine. The machine operated at a force of 2.5 kN for testing all Hemp_nw nonwoven, Flax_nw nonwoven and Hemp_comp composite materials. In the testing of Flax_comp composite materials, the machine force was 20 kN. The samples were tested longitudinally in tension at a constant elongation.

The length of the nonwoven materials sample strip between the clamping clamps was 200 mm, the samples were tested in tension at a speed of 100 mm/min. The length of the composite sample between the clamping clamps was 100 mm, the samples were tested in tension at a speed of 50 mm/min.

Three-point bending test of composite materials

Composites of both compositions are tested in tree-point bending. The test was performed in the ITA laboratory in accordance with the standard DIN EN ISO 14125 (Latvian version of the standard LVS EN ISO 14125:2001 [39]). Composite samples with dimensions of 40 mm × 25 mm × thickness (depending on the composition) mm were used for the tests. In the tree-point bending test, the sample is placed longitudinally on two parallel needle supports with r = 5 mm. The loading force was applied from the top towards the middle of the sample, occupying a surface area of r = 5 mm. The test of the samples was started with an initial load of 0.1 MPa, the rate of test deformation was 1 mm per 1 min and an initial modulus of flexural elasticity of 1 mm per 1 min. The loading force induces tensile stresses on the convex surface of the test specimen and compressive stresses on the concave surface. In tree-point bending, the loading force induces shear stresses along the centreline of the material.

Sound absorption testing of nonwovens

Measurements of the sound absorption coefficient and the sound transmission loss coefficient were performed for Hemp_nw containing nonwoven material samples in a twochamber full impedance tube. Measurements were made according to standard LVS EN ISO 10534-2:2002 (Acoustics. Sound absorption coefficient and total impedance in pipes. Part 2: Transfer function method [40]). Standing wave equipment (impedance tube) Type 4206T from Brüel & Kjær company, which works together with PULSETM Acoustic Material Testing software-Type 7758, was used in the measurements.

Evaluation of the quality of nonwoven and composite materials

Visual evaluation, determination of geometric parameters, calculation of density and surface density and determination of tensile strength of Hemp_nw nonwoven, Flax_nw nonwoven, Hemp_comp and Flax_comp composite materials are considered standard material evaluation methods. The three-point bending test performed on composite materials is additionally characteristic of mechanical properties. The test of the acoustic properties of Hemp_nw nonwoven characterises the material's suitability for use in sound absorption, including use in car interior parts.

Methodology of data collection of life cycle inventory of nonwoven and composite materials

Methods, purpose of life cycle research, functional unit

The life cycle analysis (LCA) methodology, which is a standardised procedure for evaluating the environmental impact of proposed product systems, was used in this study [41]. The study was conducted in accordance with the standards ISO 14040:2006 [42] and 14044:2006. The LCA calculation model for the input and output systems of this study was created using the SimaPro 8.5.2 LCA software, which is combined with the Ecoinvent v3 database, which provides the necessary process data for the full calculation. The missing data in the database are combined from previous studies by other researchers, interviews given by growers and entered manually. The total environmental impact was assessed using the CML-IA-baseline method [43]. Global warming potential (GWP) for a 100-year period was estimated by the IPCC 2013 GWP 100a method [44].

The purpose of the life cycle study is to investigate the impact of the product production cycle on the environment and to evaluate the most environmentally friendly alternative scenarios for the cultivation of hemp and flax fibres. In the cultivation of hemp fibres, scenarios are developed that vary with annual N fertilization doses of 0 kg/ha, 30 kg/ha, 60 kg/ha, and 90 kg/ha (hereinafter, in the text, these fertilization scenarios will be labeled HN0, HN30, HN60, and HN90). In the cultivation of flax fibres, the scenarios of N fertilization with a dose

of 0 kg/ha, 20 kg/ha, 30 kg/ha, 35 kg/ha, and 40 kg/ha (hereinafter referred to as FN0, FN20, FN30, FN35, and FN40) were examined.

The identification of two different functional units allows a comparison of the environmental impact between the two composite materials developed in the study and a car parcel tray composite material on the market. Within the functional unit, all composites are equalised both in terms of tensile strength – assuming a tensile strength of 4.1 kN for all composites (corresponding to the strength of the Citröen C5 (2007) parcel tray on the market) and thickness of 4.5 mm (thickness of the parcel tray on the market).

Life cycle system boundaries and data quality

For the comparison of the LCA calculation of Hemp_comp and Flax_comp composite materials, an intermediate life cycle approach "cradle to gate" was applied, i.e. from the acquisition of raw materials to the output of the finished product from the production plant (Fig. 2.12). These research boundaries include the following production processes: cultivation of fibre hemp (in the case of material containing hemp fibre) and cultivation of fibre flax (in the case of material containing flax fibre), fibre processing, nonwoven material production, and composite material production. The use and end stage are not analysed within the scope of the study.



Fig. 2.12. The main stages of composite material production – from the extraction of the fibres forming it to the finished product.

Summary of Chapter 2

As an alternative in the production of textile materials used in the interior of the car, the polymers used so far such as PET, PP, PA, and PE are replaced by a blend of hemp (40 wt. %) and PLA (60 wt. %) fibres and parallelly developed flax (40 wt. %) and PLA (60 wt. %) fibre

blend. The hemp and flax fibres chosen for the reinforcement as well as the biodegradable PLA polymer chosen for the matrix are 100 % natural raw materials. Hemp and flax are resources that can be obtained annually, the production of both hemp and flax fibres during their growth is associated with the absorption of CO_2 from the environment.

Both the selected plant fibres and PLA polymer fibre are suitable for nonwoven material production by mechanical needle punching method. Mechanical needle punching has high-yield, but unlike other nonwoven material production methods, with a much smaller impact on the environment, as it does not involve the consumption of water resources.

In the Doctoral Thesis, the proportion of the selected fibre blend and the structure of nonwoven material was created in such a way that a composite was obtained from the nonwoven with the help of the hot-pressing method. Hot-pressing, as a composite material manufacturing method, has affected the structure of nonwoven material. Layers of pure PLA fibre are planned on the outer edges of the material so that the PLA polymer fibres melt under the influence of heat and cover the plant fibres, protecting them from the direct influence of heating surfaces. In the production of composites by pressing, it is important to correctly select the technological working settings of the equipment, such as pressure (3.75 bar), temperature (162 °C) and time (15 min).

Calculation of density and surface density and determination of tensile strength were performed for the finished nonwoven and composite materials samples, which are considered standard material evaluation methods. The three-point bending test performed on the composites additionally characterises the mechanical properties. Hemp_nw nonwoven acoustic properties testing characterises the suitability of the material for use in sound absorption, including in car interior parts.

The purpose of the life cycle study is to investigate the impact of the product production cycle on the environment and to evaluate the most environmentally friendly alternative scenarios for the cultivation of hemp and flax fibres. Two functional units were used for mutual comparison of the Hemp_comp and Flax_comp composite materials developed in the Doctoral Thesis and the polyamide and glass fibre composite (PA66/GF) of the car parcel tray on the market. In the case of the first functional unit, the same tensile strength is assumed for all composites -4.1 kN (corresponding to the strength of the parcel tray on the market). Within the second functional unit, all composites are compared at the same thickness of 4.5 mm (thickness of the parcel tray on the market).

3. EXPERIMENTAL PART

Comparative analysis of the properties of nonwovens

Comparative visual analysis of nonwovens

Due to the presence of PLA fibres, the nonwoven materials of both compositions have lighter than the natural colour of each plant fibre (see Figs. 3.1 and 3.2). The surfaces of the

samples show the holes of the needling needles left during the manufacture of nonwoven material, which are clearly visible on the surface of the material pierced by the needle plates of the needling machine. According to the indentations left by the needle, the direction in which the strips were cut can be determined for both nonwoven samples – parallel or perpendicular to the working direction of the machine.



Fig. 3.1. Strips of Hemp_nw_1 nonwovens samples.



Fig 3.2. Strips of Flax_nw_1 nonwovens samples.

Technical characteristics of nonwovens

According to the measurements made, the average weight of the Hemp_nw nonwoven sample strips (50 mm \times 300 mm) (between sample strips Hemp_nw_1, Hemp_nw_2, Hemp_nw_3, Hemp_nw_4) varies from 12.8–13.9 g or less than 9 %. The average weight of the Flax_nw nonwoven samples (between sample strips Flax_nw_1, Flax_nw_2, Flax_nw_3) varies from 17.5–18.1 g or within 3 %. The Hemp_nw nonwovens are thinner (7.0–8.8 mm) and the fibres are more tightly bound than the nonwovens containing flax fibres (13.6–14.9 mm).

Comparing the density of Hemp_nw nonwovens, it was found to be ~28 % denser (average density 108.3 kg/m³) than of Flax_nw nonwovens (average density 84.4 kg/m³). Comparing the surface density, we observe that Hemp_nw nonwovens (883 g/m²) have a 35 % lower surface density than the Flax_nw nonwovens (1187 g/m²). The graphs in Fig. 3.3 demonstrate the dependence of the nonwovens' surface density on the working direction in which the samples are cut. The diagrams clearly show that in the case of both materials, the samples that are located not only on the outer edge of the material but also in the direction of their production work is perpendicular to the direction of the machine and have significantly lower values.



Fig. 3.1. Dependence of nonwovens' surface density on the sample cutting direction: a) Hemp_nw, b) Flax_nw.

Tensile strength properties of nonwovens

The strength indicators of both nonwovens are affected by the working direction of the machine, in which the tested sample strips are oriented – the tensile strength of the material is higher in the working direction of the machine. For Hemp_nw nonwoven samples oriented parallel to the machine working direction, the material G_{max} varies from 0.05 MPa to 0.1 Mpa; when it is perpendicular to the machine working direction, G_{max} varies from 0.05 MPa to 0.08 MPa. Elongation caused by stretching or material ε_{Fmax} for samples oriented in the working direction of the machine varies between ~ 29–39 %, perpendicular to the working direction of the machine ε_{Fmax} varies ~ 36–46 %. For Flax_nw nonwoven samples, G_{max} in the working direction of the machine varies in from 0.03 MPa to 0.07 MPa, while perpendicular to the

working direction of the machine G_{max} varies from 0.04 MPa to 0.05 MPa. The elongation of Flax_nw nonwoven material parallel to the working direction of the machine $\varepsilon_{F\text{max}}$ varies between ~ 49–59 %, perpendicular to the working direction of the machine $\varepsilon_{F\text{max}}$ varies between ~ 40–47 %.

When comparing the average values of Hemp_nw nonwovens and Flax_nw nonwovens, a higher stress G_{max} average and a lower material elongation ε_{Fmax} average can be observed for the hemp fibre-containing nonwovens in the machine working direction parallel to the cut sample strips. Hemp_nw nonwoven G_{max} average value is 0.06 MPa or 28 % higher than of Flax_nw nonwoven. For the nonwoven composition containing hemp fibres, the material elongation $\varepsilon_{vid \ Fmax}$ is ~ 36 % higher, and it is 42 % lower than for the Flax_nw nonwoven.

Comparative analysis of the acoustic properties of nonwovens

The mechanical needling method provides air voids in the nonwoven material structure. These air inclusions have a positive effect on the material's ability to isolate sound. Hemp_nw nonwoven samples Hemp_nw_1, Hemp_nw_2, and Hemp_nw_4 were tested in the frequency band from 50–5000 Hz. As already mentioned above, noise absorption in the low frequencies up to 500 Hz, which is close to the sound radiation from the engine, intake and exhaust systems, is important in cars. Another common noise frequency band is between 500 Hz and 2000 Hz with a peak at 1000 Hz – tire-road noise. Nonwoven material samples Hemp_nw_1, Hemp_nw_2, and Hemp_nw_4 have low sound absorption coefficient values in almost the entire low frequency range (Fig. 3.4), (Table 3.1) and frequency range up to 800–1000 Hz, and they vary in a similar amplitude for all samples.



Fig. 3.2. Absorption coefficient values of Hemp_nw samples in the low frequency radiation range.

In the sound frequency range above 1000 Hz, the sound will be better absorbed by the nonwoven material of sample Hemp_nw_1. For all tested samples of nonwoven material in this range, α corresponds to the lower limit of Class E (Fig. 3.5), (Table 3.1). In the frequency range

up to 2000 Hz, sample Hemp_nw_1 perfoms better than the rest of both samples. Sample Hemp_nw_1 corresponds to Class E in the α frequency range above 1250 Hz, and to Class D at frequency 3150 Hz. The absorption capacity of samples Hemp_nw_2 and Hemp_nw_4 is slightly worse.

Nonwoven materials show a trend – as the sound frequency increases, the sound absorption coefficient increases (the sound absorption class increases).



Fig. 3.3. Absorption coefficient values of Hemp_nw samples in the medium frequency radiation range.

Table 3.1

Sample	Hemp_nw_1		Hemp_nw_2		Hemp_nw_4	
Frequency (Hz)	α	Sound transmission loss coefficient (dB)	α	Sound transmission loss coefficient (dB)	α	Sound transmission loss coefficient (dB)
50	0.07	1.20	0.07	1.90	0.07	0.79
63	0.09	1.20	0.09	2.00	0.05	0.76
80	0.11	1.30	0.09	2.10	0.06	0.84
100	0.08	1.40	0.07	2.20	0.06	0.96
125	0.09	1.40	0.06	2.30	0.07	1.10
160	0.08	1.50	0.07	2.40	0.06	1.15
200	0.08	1.50	0.06	2.50	0.06	1.18
250	0.08	1.50	0.06	2.50	0.06	1.25
315	0.08	1.50	0.06	2.50	0.05	1.31
400	0.09	1.60	0.07	2.60	0.07	1.43
500	0.09	1.70	0.07	2.60	0.07	1.43
630	0.10	1.70	0.07	2.70	0.07	1.51
800	0.11	1.80	0.08	2.80	0.08	1.57

Values of Absorption Coefficients and Sound Transmission Losses

Sample	Sample Hemp_nw_1		Hemp_nw_2		Hemp_nw_4	
Frequency	α	Sound transmission loss coefficient	α	Sound transmission loss coefficient	α	Sound transmission loss coefficient
(Hz)		(dB)		(dB)		(dB)
1000	0.13	1.70	0.09	2.80	0.09	1.58
1250	0.15	1.80	0.11	2.80	0.10	1.63
1600	0.16	2.00	0.15	2.90	0.13	1.75
2000	0.19	2.10	0.19	3.10	0.16	1.86
2500	0.24	2.20	0.25	3.20	0.19	1.97
3150	0.31	2.40	0.33	3.30	0.25	2.10
4000	0.40	2.50	0.45	3.50	0.33	2.25
5000	0.52	2.80	0.58	3.90	0.44	2.52

Absorption classes for a values

A 0.90; 0.95; 1.00

B 0.80; 0.85

C 0.60; 0.65; 0.70; 0.75

- D 0.30; 0.35; 0.40; 0.45; 0.50; 0.55
- E 0.15; 0.20; 0.25

Not classified 0.00; 0.05; 0.10

Not classified E

D

Table 3.1 continued

Comparative analysis of properties of composite materials

Comparative visual analysis of composite materials

The composite materials samples of both compositions (Figs. 3.6 and 3.7) became darker (compared to the same nonwoven material composition) after heat treatment (in the C press), having the colour of natural fibres. Under the influence of heat treatment, PLA polymer fibres form a transparent mass after melting.

Hemp comp and individual strips of Flax comp composite materials samples show partial leakage of melted PLA out of the material. The surface of Hemp composite's samples is rougher to the touch than of the Flax comp composite. The surface roughness can be explained by the technical fibres used in the production of Hemp comp composite materials, which are coarser in terms of fineness than the flax fibres used in the production of the composite of the other composition.

The light colour of Flax comp composite material's samples can be observed in different intensities of tones, which indicates uneven melting of PLA polymer fibres.



Fig. 3.4. Hemp_comp composite material sample strips: a) Hemp_comp_1; b) Hemp_comp_2; c) Hemp_comp_3; d) Hemp_comp_4; and e) Hemp_comp_5.



Fig. 3.5. Flax_comp composite material sample strips: a) Flax_comp_1; b) Flax_comp_2; and c) Flax_comp_3.

In Fig. 3.8, cross-sections of composite samples of both compositions are shown, showing white areas at different positions formed by not fully melted PLA. The not melted PLA in the cross-section of the Flax_comp composite has retained a more semi-crystalline structure, and this distinguishes it from the composition of the other composite.



Fig. 3.6. Cross-sections of composite materials: a) Hemp_comp composite material; b) Flax_comp composite material.

At 200 μ m optical microscope magnification, darker spots are observed on both hemp and flax fibres (Fig. 3.9), which could be assumed to be fibre defects. However, dark spots are not uncommon. The origin of the dark spots can also be explained by the biopolymer lignin in plant fibres, which is light yellow-brown in colour.



Fig. 3.7. Natural fibres embedded in composite materials: a) hemp fibre in Hemp_comp composite; b) flax fibre in Flax_comp composite.

Technical characteristics of composite materials

According to the measurements made, the strips of all Hemp_comp and Flax_comp composite samples kept the same width dimension after the hot-pressing process; it is the same as for NM strips – 50 mm. The length of the Hemp_comp composite material strips varies from 232 mm to 244 mm, for the Flax_comp composite material the length varies from 233 mm to 242 mm.

Hemp_comp composites are thinner (1.3–1.6 mm) than Flax_comp composites (1.7–1.9 mm).

Average density values for both composite materials are similar – Hemp_comp composite is ~1 % denser (average density 646.1 kg/m³) than Flax_comp NM (average density 637.6 kg/m³).

Comparing the average surface density values (Fig. 3.10), it is observed that for Hemp_comp composite (884 g/m²) it is almost 30 % lower than for Flax_comp composite (1148 g/m²). The surface density of the Hemp_comp composite in the machines in the working direction is about 5 % higher, and in the case of the Flax_comp composite, it is 12 % higher than of the samples cut perpendicular to the machine direction.



Fig. 3.8. Dependence of the surface density of the composite materials on the cutting direction of the initially made nonwoven material's sample: a) Hemp_comp; b) Flax_comp.

Compared to the average surface density results of nonwoven materials, the difference between the nonwoven and composite materials values for the Hemp_comp composite is less than 1 %. The biggest difference is between the Flax_comp composite materials – the nonwoven materials variation has an average surface density 3 % higher than the composite.

Tensile strength properties of composite materials

For both types of composite materials, the tensile strength was determined for sample strips cut parallel to the working direction of the machine. The tensile stress-strain curves of the Hemp_comp and Flax_comp composite strips (Figs. 3.11 and 3.12) can be imaginary divided into elastic limit, yield limit and maximum strength and ultimate tensile strength limit areas. In the region of elastic deformation, the average tensile strength-strain curves for both materials are similar in nature – at the initial stage, the curve of the relationship between tensile limit stress and deformation is a parabola, which turns into a straight line. G_{max} of each material is accepted as the yield strength of the composites of both compositions. For Hemp_comp composites, the tensile strength G_{max} ranges from 19.3 MPa to 34.0 MPa, with G_{max} averaging 27.1 MPa. The tensile material elongation ε_{Fmax} for the Hemp_comp composite varies between 2.2 % and 2.7 %, with an average of ε_{Fmax} of 2.45 %. For Flax_comp composites, the tensile strength G_{max} varies from 39.6–48.4 MPa, with G_{max} averaging 45.2 MPa. The tensile elongation of the material ε_{Fmax} varies from 3.6 % to 4.3 %, with an average of ε_{Fmax} of 3.91 %.



Fig. 3.9. Hemp_comp composite material in ultimate tensile strength to material collapse for strips Nos. 2 and 4 of samples: Hemp_comp_1, Hemp_comp_2, Hemp_comp_3, Hemp_comp_4 and Hemp_comp_5.



Fig. 3.10. Flax_comp composite material in ultimate tensile strength to material failure for strips Nos. 2 and 4 of samples: Flax comp 1, Flax comp 2 and Flax comp 3.

Traditionally used materials and methods in the production of passenger car textile parts

Looking at the passenger car models available on the market and comparing the parcel tray in them, it should be concluded that they are mainly available in two variations:

- 1) solid, one-piece (Fig. 3.13);
- 2) roll up principle (Fig. 3.14).





Fig. 3.11. Citröen C5 Aircross luggage compartment and parcel tray [45].

Fig. 3.12. BMW X6 luggage compartment and roll up parcel tray [46].

The outer layer of the parcel tray, like other interior parts made of textile materials, tends to be separated, and its composition may differ from the rest of the tray, as it is in direct contact with the user and is more exposed to various surface mechanical damages. Also, in the composition of the outer layer of the textile material, as well as in the composition of the structural part of the parcel tray itself, it is observed that PP is more often used in the USA, while PET is used in the rest of the world. The linear density of PP fibres used in the US market in the production of parcel trays varies from 15–18 denier, whose wear resistance requirements in the specification are higher, but in the Far Eastern countries and Japan, where the wear resistance requirements of textile materials used in automotive construction are lower, PET fibres with a linear density of 6 denier are used [36]. The thickness of the load-bearing layer varies by ~ 3 mm, and the thickness of the decorative layer ~ 1.5 mm, with the total surface density reaching ~ 1250 g/m² [6].

In order to further compare the parcel tray on the market with the two composites developed in the Doctoral Thesis, the size of the parcel tray has been chosen: 1000×500 mm. Thus, to withstand a force of 4,1 kN, the required thickness of the Hemp comp composite material is 3.0 mm (or 32.8 % less than the sample on the market), while the required thickness of the Flax comp composite material would be only 1.8 mm (or 59.6 % less than the sample on the market). According to the required amount of mass, the heaviest on the market is PA66 and 30 wt. % fibreglass parcel tray, Hemp composite material is 35.0 % lighter, the lightest would be Flax comp composite -61.4 % lighter than the heaviest sample on the market. Even though the much thinner Hemp comp and Flax comp material samples could provide equivalent strength to the panel on the market, there are still concerns about the rigidity of these two materials. For this reason, the composites have been compared by thickness, assuming a thickness of 4.5 mm (a value close to the thickness of the composite on the market) as a comparative value. At the same thickness -4.5 mm (Table 3.2), the ultimate strength of Hemp composite materials is 48.8 %, and the ultimate strength of Flax comp composite materials is 147.8 % higher than of PA66/GF. On the other hand, the amount of mass required to produce a 1000 mm \times 500 mm \times 4.5 mm panel varies within 3.2-4.5 % for all three composite materials.

Composite material	Boundary stress (MPa)	Mass (kg)			
Hemp_comp	27.1	1.45			
Flax_comp	45.2	1.43			
PA66/GF	18.2	1.50			

Composite Material Technical Parameters Depending on Panel Thickness 4.5 mm

It has just been shown that at the same material thickness, the ultimate strength of Flax_comp composite is significantly higher than of the other two composite materials. To Hemp_comp and PA66 and 30 wt.% glass fibre composites, the ultimate strength would be equal to the ultimate strength of Flax_comp composite (Table 3.3), the thickness of Hemp_comp composite should be increased by 66.5 % and PA66 and 30 wt.% glass fibre composite thickness. The thickness of the composites can be achieved by increasing the mass: Hemp_comp composite weight by 68.6 % and PA66 and 30 wt.% glass fibre composite is 159.35 % more than the mass of Flax comp composite.

Table 3.3

Composite material	Thickness (mm)	Mass (kg)
Hemp_comp	7.5	2.42
Flax_comp	4.5	1.43
PA66/GF	11.2	3.72

Technical Parameters of Composite Materials at a Tensile Force of 10.17 kN

In the Doctoral Thesis, both obtained composites are lighter in mass (the light mass saves the volume of raw material, while the use of the finished material in the construction of the car allows to reduce fuel consumption) and are made from renewable resources that are in line with the European Green Deal.

Tree-point bending properties of composite materials

A characteristic affecting the tree-point bending strength – the average cross-sectional area of the test sample for the Flax_comp composite is 33 % greater than for the Hemp_comp composite. The advantage of the bending resistance of the Flax_comp composite over the Hemp comp composite is 81 %.

The modulus of elasticity is 39.7 % higher for the Flax_comp composite. The relative error is similar for both compositions: Hemp_comp composite is 0.03 %, Flax_comp composite – 0.04 %.

The load and deflection curves of the 3-point bending tests are shown in Fig. 3.15. Comparing the bending strain, the composite materials of both compositions show an equivalent result of ~ 6 mm. The deflection of the Hemp comp composite was obtained at ~ 0.8

kN to 1.2 kN of high load. For Flax_comp composite ~ 6 mm deformation was obtained at 1.5 kN to 2.1 kN high load.



Fig. 3.13. Comparison of load and deflection curves of composite loading tests: a) Hemp_comp composite samples Hemp_comp_1, Hemp_comp_2, Hemp_comp_3, Hemp_comp_4 and Hemp_comp_5; b) Flax_comp composite samples Flax_comp_1, Flax_comp_2 and Flax_comp_3.

Indicative costs of manufacturing nonwoven and composite materials

To produce nonwoven material, the production line consists of a fibre bale opening device, a fibre low-pressure laying (and fibre mixing) device, a fibre coating layer formation and needle punching device. The final product of nonwoven materials can be obtained wound on a roll and its edges can be trimmed. To produce composite materials, the production line can be supplemented with a calender machine.

Within the framework of the study, the cost of nonwoven and composite materials was calculated, the input data of these calculations are summarised in Table 3.4.

Comparing the cost of nonwoven material within one composition, depending on whether it is produced in one or two shifts, is minimal. Needle-punched 26.0 mm thick (108.3 kg/m³) Hemp_nw nonwoven's approximate cost of production in one shift is 13.03 EUR/m², the cost of production in two shifts is 4 % cheaper – 12.51 EUR/m². For comparison, the approximate initial cost of needle-punched 35.0 mm thick (84.4 kg/m³) Flax_nw nonwoven when produced in one shift is 15.53 EUR/m², when produced in two shifts, the cost is ~5 % cheaper at 14.84 EUR/m². It should be noted that almost 2 % less material is used per 1 m² of nonwoven material to produce the composition containing flax fibres.

Physical	l-mecha	anical	and	Tec	hno	logical	Par	amet	ers o	of He	emp_	_nw,	Flax_	nw	N	onwo	oven	and
			Hen	np_c	com	o, Flax	_co	mp C	om	posite	e Ma	ateria	als					

Designation	Parameter of composition of hemp fibre	Parameter of composition of flax fibre		
Density of finished nonwoven material (kg/m ³)	108.3	84.4		
Density of the finished composite material (kg/m ³)	646.1	637.6		
Thickness of finished nonwoven material (mm)	26	35		
Thickness of finished composite material (mm)	4.5	4.5		
Surface density of the finished nonwoven material (g/m ²)	2797	2834		
Surface density of the finished composite material (g/m ²)	2800	2930		
The working width of the production line (m)	2.6	2.6		
Production line yield limit (kg per 1 h)	400	400		
Production line product output rate (m per 1 min)	0.9	0.9		
Product output speed of the production line when working in 1 shift (K m per 1 year)	104.3	99.6		
Product output speed of the production line when working in 2 shifts (K m per 1 year)	208.7	199.3		
Production line product output speed when working in 1 shift (K m ² per 1 year)	244.2	233.2		
Production line product output speed when working in 2 shifts (K m ² per 1 year)	488.3	466.3		
Line capacity (kW)	117.5	117.5		
Line electricity consumption, kW/h (makes up 75 % of the equipment's capacity)	88.13	88.13		
Depreciation period of equipment (years)	10	10		
Bank (financial institution ALTUM) rate (%)	1.9	1.9		
Co-financing of the financial institution ALTUM for the purchase of equipment (%)	80	80		
The number of direct labour hours for equipment service personnel in one shift if they work 8 hours a day, assuming that there are 253 working days in 2022 (h per 1 year)	2024	2024		
The useful working time of the equipment during one shift, excluding idle time of 30 min. (h per 1 year)	7.5	7.5		
The useful working time of the equipment during one shift, excluding idle time of 30 min. (h per 1 year)	1897.5	1897.5		
Losses of plant fibres during processing (%)	20	20		
Losses of PLA fibres during processing (%)	5	5		

Compared to the cost of composites per 1 m^2 , the cost of Hemp_comp composite (12.66 EUR/m²) produced in two shifts is almost 18 % lower than that of Flax_comp composite (15.18 EUR/m²) produced in two shifts change.

Results of analysis of life cycle inventory data of nonwoven and composite materials

Environmental impact of hemp and flax cultivation

The CML graph (Fig. 3.16) shows the sources of emissions to GSP from the cultivation of hemp (fertilization scenarios HN0 and HN90) and flax (fertilization scenarios FN0 and FN40). In the cultivation of hemp under the HN0 fertilization scenario, the main sources of emissions of harmful substances are diesel fuel and the use of agricultural machinery that consumes it. For the HN90 fertilization scenario, the emission source mentioned above is supplemented by the emission from ammonia caused using mineral fertilizer. In both cases of hemp cultivation, the above-mentioned emissions are significantly exceeded by the large accumulation of CO_2 which is -96 % of the total impacts in both fertilization scenarios.



Fig. 3.14. CML2 Baseline results from fertilization scenarios HN0, HN90, FN0, and FN40 for global warming (GWP100a) impact category.

In the case of flax cultivation, the main emission sources under the FN0 fertilizer scenario are diesel fuel and the agricultural machinery that consumes it, as well as the use of pesticides and herbicides. In the case of the FN0 fertilization scenario, the CO₂ accumulation is almost

-66 % of the total environmental impacts of this scenario. As the fertilizer rate increases (in fertilization scenario FN40), the total amount of emission per unit of flax harvest decreases

significantly. The emission of diesel fuel, which causes the greatest impact, decreases to 11 %, while the impact of emissions from the use of agricultural machinery decreases to 1.5 %. In the FN40 scenario, an increase in CO_2 accumulation can be observed up to -83 %, which in the case of flax reflects the efficiency of fertilization.

The increase in green mass yield per unit area of both hemp and flax proves the usefulness of N fertilization. The economic benefit of the harvest against the environmental impact of emissions from fertilizer and field operations has to be evaluated.

Environmental impact of composite materials manufacturing

The impact on the environment from the point of view of composite production is considered when producing 1 ton of nonwoven material. Emissions within both material compositions for flax are 11–12 % higher than the emissions observed in NM. Comparing the environmental impact of the two composite materials Hemp_comp HN90 and Flax_comp FN40 in the environmental categories considered by CML, it is proportional to what is observed in the nonwoven material production of these two compositions. The Hemp_comp HN0 composite has a lower environmental impact from the manufacturing process than the Flax_comp FN0 composite, except for the GSP impact. The production processes of both composite materials account for the impact resulting from the nonwoven material production process (mostly all impacts) and the electricity consumption used to make the composite (accounts for less than 1 % for the Hemp_comp HN90 composite and slightly more than 1 % for the Flax_comp FN40 composite).

In the case of the Hemp_comp and Flax_comp composite materials developed in the Doctoral Thesis, and in the case of the PA66/GF composite materials available on the market, greater impacts on the environment (Fig. 3.17) can be observed for the considered composite variations of the second functional unit (composite thickness 4.5 mm). As for the composite materials of the first functional unit, only the thickness of the PA66/GF variation composite is 4.5 mm, it is smaller for the other two composite materials, so the usable amount of fibres and the impact of emissions related to their acquisition on the environment are also smaller. In order to reach the selected strength threshold, each composite requires a different amount of raw materials. The difference between the smallest and largest required amount of fibres is 61 %. At GSP, the lowest impact is observed for the Flax_comp 4.1 kN (1.8 mm) (0.7 kg CO₂ eq) composite, which is 2.4 times less than the Hemp_comp 4.1 kN (3.0 mm) composite, but the largest impact is caused by the PA66/GF composite with 13.7 kg CO₂ eq (which is almost 20 times more than the observed lowest impact for the Flax_comp composite). When studying abiotic depletion, a similar effect is observed for all three composites, and it varies from 7.08E-06-7 to 78E-06 kg Sb eq.



Fig. 3.15. Environmental impact of Hemp_comp HN90, Flax_comp FN40 and PA 66/GF composite materials at both functional units. Method: CML-IA baseline V3.04 / EU25/.

The shape retention provided by the Hemp_comp composite thickness of 3.0 mm is evaluated against the smaller effects of the relatively thinner Flax_comp composite of 1.8 mm on the various categories of the environment (e.g. global warming potential, ozone layer depletion, acidification, toxicity to humans, terrestrial, freshwater and seawater ecotoxicity).

Summary of Chapter 3

Hemp-PLA and Flax-PLA materials are obtained close to the colour of the plant fibre of the respective composition. The nonwoven material, because of the PLA fibre in outer sides, is in lighter colour. The composites are darker because the PLA fibres have melted and become transparent due to hot-pressing.

The nonwoven and composite materials of both fibre blend variations have a lower surface density than the conventionally accepted parcel tray decorative and load-bearing layers combined (~1250 g/m²). In comparison, Hemp_comp composite materials have a surface density 29 % lower than the conventional parcel tray, for nonwoven material it is 883 g/m² and for the composite it is 884 g/m². For the Flax_comp composite material, the surface density is 5-8 % lower, or for nonwoven material it is 1187 g/m², for the composite it is 1148 g/m².

50–2000 Hz is the frequency isolation region that best characterises the acoustic performance of the engineered Hemp_nw nonwoven, fully encompassing the dominant car noise frequency range of 1000 Hz and engine noise below 500 Hz.

In comparison with the parcel tray on the market, which is made of polyamide 66 and 30 wt. % glass fibre, the established limit stresses are ~ 1.5 times higher for Hemp_comp composite (27.1 MPa), and ~ 2.5 times higher (45.2 MPa) for Flax_comp composite. The Flax_comp composite also shows higher results in 3-point bending strength, where the average ultimate bending strength is 1.0 MPa and is 1.9 times higher than for the Hemp_comp composite (0.5 MPa).

In the case of both compositions, the cost of materials is lower for production in two shifts, where the cost of 1 m² of Hemp_nw nonwoven (12.51 EUR/m²) is almost 17 % lower than of Flax_nw nonwoven (14.84 EUR/m²). The cost of 1 m² Hemp_comp composite (12.66 EUR/m²) is almost 18 % lower than that of Flax_comp composite (15.18 EUR/m²).

The application of N fertilizer demonstrates an increase in green mass yield per unit area of both hemp and flax. In the case of the scenario of maximum fertilization of flax, the total amount of emissions per unit of flax yield is significantly reduced.

Between the two examined functional units, lower environmental impacts can be observed for composite variations at the first functional unit. In this case, the thickness of the composite depends on the ultimate tensile stress of 4.1 kN. Both Hemp_comp and Flax_comp composite materials reach the tested limit stress for the market-based cover panel at significantly less material thickness than 4.5 mm.

CONCLUSIONS

In the Doctoral Thesis, nonwoven materials have been studied with the aim of creating a competitive composite material from them based on environmentally friendly starting materials, which would be used in the automotive industry. For this purpose, light, sufficiently durable materials with good acoustic properties are needed. Current EU regulations require the introduction of fully recyclable and environmentally friendly materials in the automotive industry. Hemp and flax fibres suitable for cultivation in Latvian conditions, as well as biopolymer polylactide fibres, have been selected for solving the tasks.

Conclusions have been made by planning, manufacturing, researching, analysing nonwoven materials and composite materials made from them compared to existing composite materials on the market.

- The tensile strength of NM and the elongation of the material is affected by the tightness of the fibres in the material structure. The tighter the fibres interlock in the material structure, the higher the tensile strength and the lower the elongation of the material. This is confirmed by the results of the tensile strength of the NM in which Hemp_nw nonwoven is thinner and lighter by weight than Flax_nw nonwoven, but the strength of the first composition is higher (6_{max vid} is 0.06 MPa) and the elongation of the material is less (ε_{vid Fmax} is ~ 36 %).
- At the same NM thickness and varying surface density, a higher sound absorption factor or higher sound absorption capacity will be for material with a higher surface density value. The absorption Class E of the Hemp_nw nonwoven material developed in the Thesis is suitable for carpets. Classes D and C are suitable for most gypsum inner wall systems (sufficient for most large-use spaces). Class D is appropriate for room height bulkhead systems with absorption elements.
- For composite materials, the tensile strength is affected by the thickness of the material. The thicker the nonwoven material used in the production of the composite, the higher the tensile strength of the resulting composite. This is confirmed by the results of the tensile strength of the composites, in which the Flax_comp composites are thicker in thickness and have higher tensile strength (6_{max vid} ir 45.2 MPa).
- In the hot-pressing treatment of a blend of plant and polymer fibres, a compromise must be found in the choice of technological settings so that the plant fibres do not burn and the polymer fibres do not melt. According to the literature [66], [99] mentioned and tested, the most suitable working temperature is 165 °C.
- At various composite thickness: 1.3–1.6 mm (Hemp_comp) and 1.7–1.9 mm (Flax_comp), the tensile strength of the Hemp_comp composite is approximately 1.48 times higher than that of the marketed and experimentally tested parcel tray (4.5 mm PA66/GF), while the tensile strain elongation of the material is almost equal to 2.45 %. The tensile strength of the Flax_comp composite is 2.48 times higher and the tensile strain is ~1.5 times higher than that of the tested parcel tray. The results of the obtained mechanical properties confirm that the use of plant fibre

proportions of 40 wt.% in the production of composite materials is an advantage compared to materials made on the basis of petroleum resources.

- Both NM and composite unit manufacturing costs are lower when manufacturing in two shifts compared to production in one shift.
- From an environmental sustainability point of view, the creation and use (including in the production of car interior parts) of compositions of blends of both fibres is useful for reducing carbon footprints.

The results of the Doctoral Thesis allow to confirm the Theses put forward for defence.

- 1. The research has created fibre blends from renewable resources: a blend of hemp and PLA fibres and a blend of flax and PLA fibres. The structure of the multi-layered material created from fibre blends allows for the production of two different materials in terms of consistency and purpose of use: first, nonwoven acoustic materials are produced using the mechanical needling method, and then composite materials are produced based on them using the thermopressing method.
- 2. Lignocellulose component mass proportion 40 mass. % in the developed fibre blends of both compositions (Hemp-PLA and Flax-PLA) is sufficient to integrate NM and composite materials made from it into the passenger car interior structure. The higher impedance results of Hemp_nw nonwoven material achieved in the frequency range of 63–1000 Hz and the better performance of the material to isolate sound in the frequency range of 1250–5000 Hz confirm the material's positive acoustic properties. On the other hand, the tensile strength of the Hemp_comp composite (thickness 1.3–1.6 mm) is 1.48 times greater, and that of the Flax_comp composite (thickness 1.7–1.9 mm) is 2.48 times greater than of the sample on the market, proving the competitiveness of both composite materials.
- 3. The creation of both types of fibre blends and their use in the production of respective composites is valid for reducing the carbon footprint compared to the traditionally used petroleum-based composites. So, for example, for the production of a panel of the same size (by area and thickness), the Flax_comp composite produces a carbon footprint of 1.2 kg CO₂ per 1 kg of composite and is 87 % less than 1 kg of PA66/GF composite. On the other hand, the CO₂ footprint created by Hemp_comp composite is 81 % smaller than that of 1 kg PA66/GF composite.

Proposals for further research

- The results of the tensile test indicate the suitability of both developed composites (Hemp_comp and Flax_comp) for use in the car interior; however, there are concerns that plate-shaped parts may bend. It is possible to provide for anchorage ribs during the design process to reinforce the stability of the shape of parcel tray or other slab-shaped product. Anchorage ribs can be made from the same or other materials. Thus, even with a slight increase in mass, the product would still be much lighter than all plastic analogues, but its mechanical strength would undoubtedly increase.
- The acoustic properties of Hemp_nw nonwoven material in the medium frequency range can be improved by laminating the external surfaces of the material, for example with ready-made polymer coatings from the company Tex Tech [47] (designed to improve certain properties, including noise suppression), and by combining the material in several layers.
- The production of PLA fibres is energy-intensive, so the CO₂ footprint of making composites of Flax_comp and Hemp_comp can increase, either by an environmentally friendly process of obtaining PLA fibre fibres, or by replacing PLA with less energy-intensive biopolymer fibres.
- Hemp_nw, Flax_nw, Hemp_comp and Flax_comp materials may be integrated not only in the construction of parts of the car compartment but also in building interior finishing materials such as ceiling plates, wall panels, and separation bulkheads. By stacking composite materials in multi-layers, the increasing thickness of the materials and the non-penetrating holes formed with a certain regularity improve the sound absorption layer ability of the materials and can also shape the surface design.
- The restrictions on people-to-people contact due to the Covid-19 pandemic have highlighted issues such as the need to separate one workplace from other in open planning offices. Residents who were forced to move their workplace from the office to a home with a limited area (including distance learning pupils, students) have also encountered such a problem. In both cases, the separation of workplaces from the surrounding environment can be solved with light partition structures. In the office, in this case, the main task is to keep people at a distance, in households, the worker should prevent surrounding sound irritations.
- When integrating Hemp_comp and Flax_comp composite materials in interior parts of a car and as building finishing materials, it is necessary to consider the possibility of adding pigments to PLA polymer fibres. By adding a pigment to the PLA polymer, it would be possible to obtain a composite in different colours during the hot-pressing process.

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