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GREEN-LOOP

Sustainable manufacture systems towards novel bio-based materials

WP6 – Upscale production and demonstration

D6.2 – Bio-rubber production and use at TRL 6

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Executive Summary

In this report, the application possibilities of the GREEN LOOP bio-based rubber panels are considered. These possibilities are based, on one hand, on the possible intended use of the products as studied in previous work, particularly the analysis provided in the deliverable D3.1. On the other hand, the possibilities are based on properties of the product scaled up to TRL6.

It is important to distinguish between the properties of material and the properties of a formed product. While the material has been developed and tested in previous work, this report focuses on characteristics inherent to a near-final product scale. Nonetheless, it should be noted that a number of properties at the stage of TRL 6 are still material properties. Since the production process adds an additional phase - forming the product - it was necessary to consider carefully whether characteristics obtained at the material level remain valid at the product level.

Section 3 provides a brief description of the production process. This report does not aim to present all details, which are available in other reports such as those of WP3. However, it does describe the production of panels from the material at larger scale. It also presents key performance indicators of the GREEN LOOP bio-based panels for use in the construction industry. The initial idea, described in task 6.2, has been expanded to address key issues arising from the construction industry's general approach: assessing a product with real-life installation conditions in mind.

The assessment structure follows the pattern of characterization of construction products, grouped in the so-called essential requirements for construction works. Based on recent legislation in the EU, the Basic requirements for construction works (BWR) are:

1. Structural integrity of construction works
2. Fire safety of construction works
3. Protection against adverse hygiene and health impacts related to construction works
4. Safety and accessibility of construction works
5. Resistance to the passage of sound and acoustic properties of construction works
6. Energy efficiency and thermal performance of construction works
7. Emissions into the outdoor environment of construction works
8. Sustainable use of natural resources of construction works

Not all requirements are reflected in the products' characteristics. In the case of the GREEN LOOP bio-based rubber panels, the analysis focuses on fire safety (BWR 2) and acoustic (vibrational) properties (BWR 5). The reason for that is that the product does not contribute to structural safety (BWR 1) nor its use represents inherent danger in use (BWR 4). Other basic works requirements are much less significant for the product (BWR 3, BWR 6, BWR 7 and BWR 8). Nonetheless these were still addressed in the analysis, however the focus has been given to fire related properties and vibrational properties.

The results presented in the report show comprehensive performance characteristics of the GREEN LOOP bio-based panels for construction use.

Additional considerations about the intended use of the product arose during development, namely the possibility to use the boards or the material formed in a different shape in systems for seismic isolation of structures. The flexible approach to analysis, not strictly tied to a single end-use, made it possible to explore such alternative applications. However, seismic isolation systems are complex, and the GREEN LOOP boards would require further adjustment of properties and a shift in focus; for example, reaction-to-fire performance would be less critical, while mechanical performance (static and dynamic) would become much more important.

Finally, Section 4 provides a condensed version of the characteristics and values. The purpose of this section is to provide information that could be used at higher TRLs in the process of putting the product on the market.

Overall, the product represents a viable material and form for certain construction applications. The weaknesses are explored, and suggestions are given on the improvement of the product to broaden its application range as far as possible. Naturally, testing at higher TRLs will have to be amended, based on the performance claim of the product. Nonetheless the product at **TRL 6 offers a solid basis for limited immediate use and a good starting point for extended use, provided that improvements in critical areas, such as raising the reaction-to-fire rating beyond the current class E (according to EN 13501-1), are achieved.**

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Abbreviations

BR	Butadiene rubber
CPR	Construction product directive (305/2011)
CO_{2, eq.}	CO ₂ equivalent
DMP	Data Management Plan
EAD	European Assessment Documents
EC	European Commission
EU	European Union
EoL	End of Life
EOTA	European Organization for Technical Assessments
GWP₁₀₀	Global Warming Potential, an indicator, as defined in EN 15804
hEN	harmonized European standard
HMW	High Molecular Weight
ICT	Information and Communication Technology
IIR	Butyl rubber
LCA	Life Cycle Assessment
MCI	Material Circularity Indicator
MFR	Materials for recycling, an indicator, as defined in EN 15804
MLV	Manufacturer Limiting Value
SBR	Styrene butadiene rubber
TRL	Technology Readiness Level
VOCs	Volatile Organic Compounds
WP	Work Package

1 Introduction

The purpose of this document is to provide information on performance of the developed GREEN-LOOP bio rubber products, using methods, foreseen in corresponding documents for the assessment of products for putting the product on the market.

The products have been tested for their most relevant characteristics, as foreseen in the description of work for the task T 6.2. Additionally, the results for WP3 were considered for inclusion in product assessment in cases where test result is independent of the sample's physical size, provided the methods are applicable to smaller-scale samples.

The results are summarized in the table of results.

The final part of product evaluation is organizing information on requirements and limit values for the individual product property into a technical assessment form, corresponding to the procedures as set out in the new EU legislation in force (CPR, Regulation on construction products, 2024/3110).

2 Bio-rubber based production

2.1 General description of the production

The bio-rubber material composition was derived and refined during WP3. The composition was optimised for acoustic and fire retardancy whilst achieving mechanical properties equivalent to competitor products. The rubber formulation consisted of devulcanised rubber (from ground tyre waste), natural rubber and kraft lignin powder (0-20 wt%). Additional curatives were added to promote the cross-linking reaction. This was laid out in full in Deliverable 3.2. No changes were made to the formulation during WP6. The materials were sourced from the same suppliers as in WP3.

2.2 Lignin production

Lignin is a complex organic polymer found in the cell walls of plants, particularly in wood and bark, where it provides stiffness and resistance to decay. It is formed as a byproduct of plant metabolism, particularly during the biosynthesis of secondary cell walls. The production of lignin is essential for the structural integrity of plants and the transportation of water through the vascular tissue [1]. In industry, lignin is often a waste product of paper and bioethanol production. The researchers are investigating its use in bioplastics, adhesives and as a renewable source of aromatic compounds[2].

Recent research has focused on the use of lignin as a flame retardant and bio-based additive. The main role of lignin in product formulation is to improve the fire behaviour of the final product. Among the different types of lignin, kraft lignin is a promising candidate as it is the most commonly produced form of technical lignin, which is a byproduct of the kraft pulping process. Its high thermal stability, strong char-forming ability and aromatic structure with numerous reactive hydroxyl groups make it a potentially renewable material for the development of flame retardant lignin-rubber composites. These properties not only improve fire resistance, but also support the integration of lignin into sustainable, high-performance materials.

2.2.1 Sourcing

Lignin is primarily obtained from plant biomass, especially from woody plants such as pine, spruce and eucalyptus. The most common industrial source is kraft lignin, a by-product of the kraft pulping process used in papermaking, where lignin is separated from cellulose with sodium hydroxide and sodium sulphide. Kraft lignin accounts for most of the lignin produced worldwide, although much of it is still burned for energy and not used for value-added applications. Other sources include lignin from sulphite pulp and from agricultural residues such as corn stover and wheat straw [3].

2.2.2 Production process

The isolation of lignin in the NIC facilities is carried out according to the process flow diagram shown in Figure 1 (*left*) using a typical protocol. Specifically, the high-pressure batch reactor (Parr; Figure 1(*right*)) is filled with dry biomass (hardwood sawdust) and the required solutions: 17 % sodium hydroxide (NaOH) and 5 % sodium sulphide (Na₂S). The amount of reagents required is calculated according to the amount of dry biomass (100 g). Thus, 17 g sodium hydroxide (NaOH) and 5 g sodium sulphide (Na₂S) were dissolved in 500L. The reaction mixture is heated to 175 °C in 45 minutes and further heated for 3 hours. The autogenous pressure rises to 6-7 bar. At the end of the experiment, the reaction mixture is cooled to ambient temperature within two hours. The reaction mixture is then filtered using vacuum filtration to remove the cellulose-rich (wood) residue. To initiate lignin precipitation, the filtrate (lignin-rich stream) is acidified to pH 2 with 75 mL of 15 % sulfuric acid (H₂SO₄). The precipitated lignin is filtered, suspended in the acidic solution (5 % sulphuric acid) and filtered again. The lignin extraction process was scaled up and optimized to produce up to 130 g/batch. Several batches were carried out to achieve the required amount of 15 kg for further material incorporation into the bio-rubber matrix.

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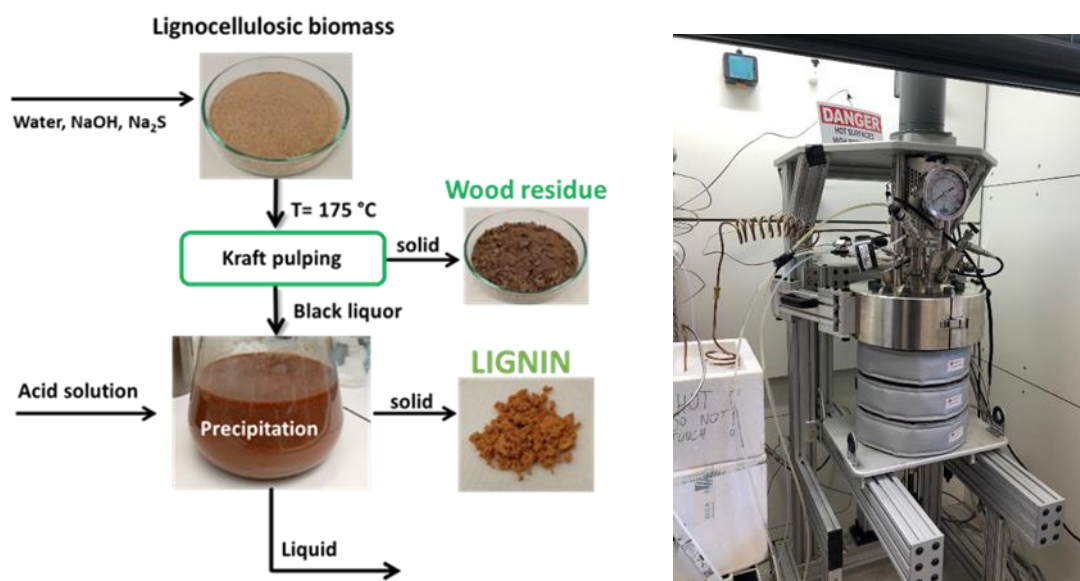


Figure 1: Kraft lignin extraction process flow diagram (left), a high-pressure batch reactor 10L (right).

2.2.3 Testing

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Degree of delignification and the purity of lignin was determined following the standard procedures TAPPI T222 om-02 and NREL/TP-510-42618[4],[5].

Quantitative ³¹P NMR experiments were carried out precisely following the protocol reported elsewhere [6]. The measurements were conducted in CDCl₃/pyridine 1:1.6 mixture at 25°C, and N-hydroxy-5-norbornene-2,3-dicarboxylic acid imide (NHND) was used as an internal standard. Prior to the analysis, lignin samples were derivatized using 2-chloro-4,4,5,5-tetramethyl-1,2,3-dioxaphospholane (TMDP). Three parallel ³¹P NMR measurements were performed for each sample, and the averages are reported in this work. The maximum standard deviation of presented results was 2×10⁻² mmol/g, while the maximum standard error was 1×10⁻² mmol/g.

Size-Exclusion Chromatography (SEC). Prior to the analyses, the lignin samples were acetylated with pyridine/acetic anhydride according to the procedure reported elsewhere [7]. SEC was performed on a size-exclusion chromatographic system (Ultimate 3000, ThermoFisher Scientific, Massachusetts, US) equipped with a UV detector set at 280 nm and a PLgel 5µm MIXED D 7.5×300 mm column with THF as an eluent at a flow rate of 1 cm³/min. Calibration was made with polystyrene standards (Polymer Standards Service; PSS) with molecular weights in the range

from 127 kDa to 672 Da. The chromatographic data were processed with the PSS WinGPC Unity software.

Glass transition temperature (T_g) of lignin was measured using Toledo DSC 2 (Mettler Toledo, Ohio, USA). The analysis was carried out within a temperature range of 25 °C to 200 °C, with a heating rate of 10 °C/min and a nitrogen flow rate of 20 cm³/min.

2.2.4 Results

Key parameters and characteristics of Kraft lignin used for bio-rubber panels are summarized in the Table 1.

Table 1: Key parameters and characteristics of Kraft lignin.

Wood residue (yield_1)	46.7 %
Delignification degree	95.9 %
Yield of Lignin (yield_2)	8.4 %
Purity of Lignin	75.1 %
Aliphatic OH group content	0.07 mmol/g
Phenolic OH group content	1.92 mmol/g
Carboxylic OH group content	0.28 mmol/g
Total OH group content	1.92 mmol/g
Average molecular weight	3500 Da
Glass transition temperature (T_g) approx.	145 °C

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2.3 Rubber compounding

In WP3, the material batches were scaled up from 60 grams to 4 kilograms per batch, generating sufficient material for the production of small test panels used in the lab-scale testing in Task 3.6. A further scale-up was necessary in WP6 for two purposes: A) to advance beyond lab-scale production and demonstrate the production of the rubber blend on pilot-scale equipment, and B) to generate enough material for large-scale acoustic and reaction-to-fire testing at UBRIS and ZAG respectively (as detailed in Section 3).

The formulation used in the scale up remained consistent with the recipe outlined in Deliverable 3.2. Batch sizes of 16 kilograms were successfully achieved for the



GREEN-LOOP bio-rubber formulation. This was possible by transitioning to a larger Banbury-style internal mixer with an increased mixing capacity. All components were combined in a one-shot mixing process. Following mixing, the material was passed through a two-roll mill to form a roll approximately 300 mm wide and 10 mm thick. In total, seven 16 kg batches were produced, yielding over 100 kg of material.

2.4 Production of panels

After compounding, the material was pressed into panels by compression moulding. The compression moulding was done in a 1100 tonne hydraulic press manufactured by Pinette PEI [8]. Figure 2 shows the press used at the NCC to manufacture the parts. This is the same press as was used in WP3 but loaded with tooling with a larger cavity. The dimensions of the WP6 parts were 500 x 500 x 10mm.



Figure 2: (Left) 1100 tonne hydraulic press, (right) material loaded into tool prior to pressing.

The process was similar to the one described in WP3, however, a change was made to a hot demoulding process rather than cold demoulding (cooling the panels

whilst still under pressure and then demoulding). This enabled a significant reduction in the cycle time so that up to two panels an hour could be produced. This was a significantly greater rate of production compared to WP3 and also reduced the energy consumption per part as the tool no required heating and cooling between each component. A maximum rate of 10 panels per day was achieved for an eight-hour shift. This could be further optimised in a production environment rather than an R&D environment.

In total, 40 panels were produced, equivalent to 10m² of rubber panelling. 4m² of panelling was sent to UBRIS for acoustic testing and 4m² was sent to ZAG for reaction to fire testing. The panels are shown in Figure 3.



Figure 3: Rubber panels produced with dimensions of 500 x 500 x 10 mm.

Non-destructive testing (NDT) was carried out on a subset of the panels to look for the presence of any defects in the bulk of the panels. For this, ultrasound scanning was used. The equipment used was as follows:

- Technique: Phased Array Pulse-Echo Semi-auto Contact
- Model: OlympusOmniscan MC2 (OMNI2-103870)
- Instruction number: 3.21.4.04
- Probe: 2.25MHz 64el Array NW1
- Settings:
 - Main Gain -25dB
 - Probe Frequency -2.25MHz
 - Transmitter voltage -40v
 - Pre-amp -none
 - Scan pitch -1mm

Overall, the panels that were tested showed a consistent attenuation which indicates a consistent consolidation across the panel (Figure 4). Some small defects were identified, the blue circles in Figure 4, which are likely to be small voids caused by trapped air. These defects are small, and few and far between so are unlikely to impact the performance of the panels.

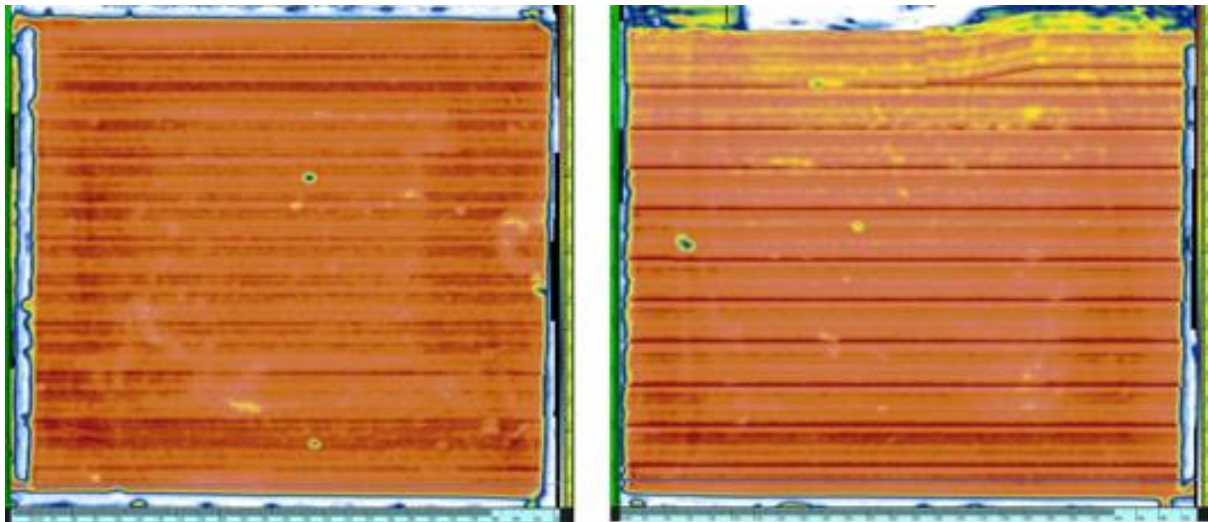


Figure 4: Ultrasound C-scan of rubber panels containing 20% lignin.

2.5 Production of 4-layered samples

In addition to “monolithic” panels, thinner samples were also prepared during the research to explore the possibility of stacking them into a thicker board (three or four layers) while coating the surfaces of individual layers to enhance the properties of the final product. This approach is a variation of the main concept and was not explored to its full extent.

Samples for testing were 4-layer samples with varying position of coatings. The schematics of samples is shown in Figure 5. Polyacrylic acid (PAA, molecular weight 250,000 g/mol, 35% in water, density 1.15 g/mL), bare sepiolite nanorods ($\text{Si}_{12}\text{O}_{30}\text{Mg}_8(\text{OH})_4(\text{OH}_2)_4 \cdot 8\text{H}_2\text{O}$) with 20-100 nm diameter, 0.2-1 μm zinc oxide, stearic acid, 2,2'-Dibenzothiazyl Disulphide (MBTS) and sulphur were obtained from Merck. Devulcanized rubber was supplied by Tyromer Ltd., natural rubber was supplied by Corrie MacColl Europe B.V and lignin was supplied by NIC. All chemicals were used as received, without any further processing or modification. Semi-cured rubber panels were created to enhance interlayer bonding between rubber sheets and tailor the final structure of the rubber part after coating and

vulcanization. The improved bonding results from the increased surface roughness of the semi-cured panels, which promotes better surface coverage and interaction between the coatings and the rubber. The coating formulation used in this study derives from prior research demonstrating improved mechanical properties of polyurethane foams using Layer-by-Layer (LbL) dip coating. The present work proposes a coating formulation based on PAA, lignin, and sepiolite (20 wt.% lignin, 1 wt.% sepiolite, 1 wt.% PAA, 78 wt.% deionised water). These fillers were selected for their potential to enhance the mechanical and thermal properties of the final product. At the same time, PAA serves both as a mechanical reinforcing agent and a binder for the fillers. The mixture was homogenized using an IKA T25 digital Ultra Turrax homogenizer at 12000 rpm to ensure proper dispersion of the components. Following homogenization, the solution underwent a degassing stage to minimize air bubbles, ensuring a more uniform coating on the rubber surface. The degassed solution was cast onto rubber sheets, and the samples were placed in an oven at 80 °C until the coating was dried. The samples were then processed using hot pressing to form multi-layered panels (Figure 5).

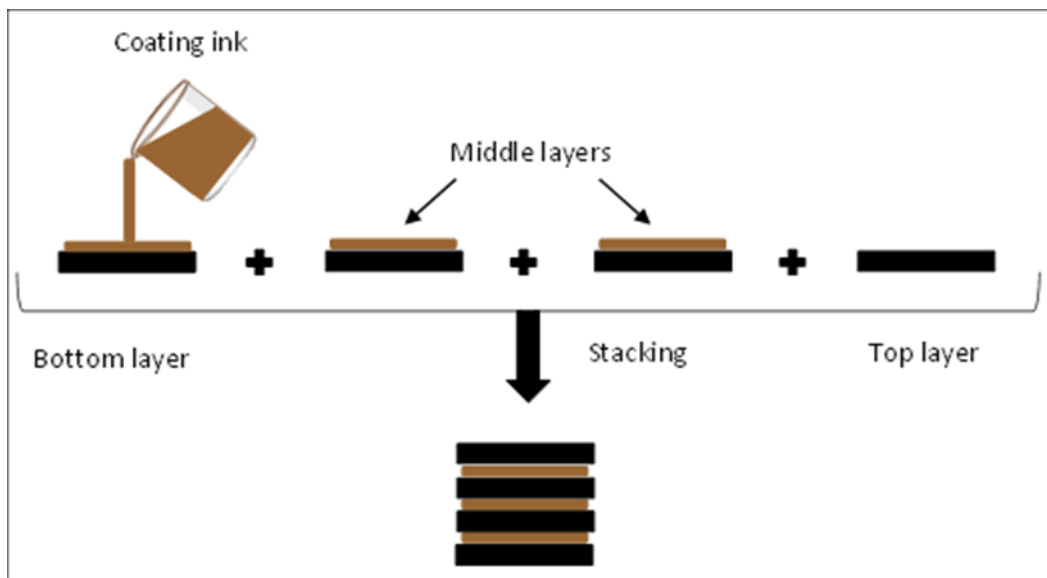


Figure 5: Schematic of production process of the multilayer GREEN LOOP bio-based rubber panels.

The coated rubber layers were stacked on each other, and placed in a 100×100 mm aluminium mould, providing a final thickness of 10 mm. For hot pressing, a JRD Bipel Laboratory Compression Press equipment with a platen size of 400×400 mm and a maximum pressing force of 40 tonnes was used. Three types of samples were

produced to study the effect of the number of coated layers on the properties of the final product, unlayered, 3-layer, and 4-layer samples. The 4-layer samples were used for the fire testing with two different coating designs. The A samples were coated on both sides, resulting in 8 coating layers and for the B samples the top and bottom layers had one coating layer on the inner surface while the middle layers were coated on both sides resulting in 6 coating layers for the fire test. After coating, the layers were stacked in the mould and pressed at 160 °C for 8 minutes under a 10-tonne load. At the end of the curing cycle, the parts were allowed to cool down to room temperature and demoulded. The excess rubber was trimmed using a blade.



3 Testing

3.1 Test plan

In general, there is a distinction between material testing and product testing. The former is intended to characterise material, while the latter is intended to characterise the product. However, in the case where the final product is a single shaped material, the distinction is not so clear.

Because material testing also included methods applied to samples representative of the product, there is some overlap in the testing. Nonetheless, due to the potential influence of the scale-up process on final product performance - arising from factors such as uniformity across the thickness, surface behaviour, and the dependence of certain characteristics on thickness - tests were also repeated on samples of the pilot product for comparison with the material-level results.

The list of tests was also adapted to the final intended end-use. Since the products are designed for internal applications, external fire performance testing was removed, and fire performance testing for flooring applications was added, as the product is intended for both wall and flooring use.

Based on the test plan, developed in WP3 and given in the deliverable D3.2, the following testing is relevant for the bio-rubber product, considering its end-use intended application.

Table 2: list of relevant characteristics

Chemical properties	Method	Test conditions
Reaction to fire - class	Class (A1-E)	As per EN 13501-1
Reaction to fire for floorings	Class (A1-E) _{fl}	As per EN 13501-1
Smoke production rate	EN 13823 or ISO 5660-1	m ² /s
Toxic gases in smoke	ISO 5660-1 and ASTM E1678)	50 kW/m ² , 21% oxygen
Content of dangerous substances	Analysis of input materials	-
Emission of dangerous substances	Selection to be done, list EN 14041	As appropriate for the method
Environmental indicators, GWP	EN 15804:2012+A2:2019	(calculation)

Physical properties	Method	Test conditions
Thermal conductivity	EN 12667	ISO 8302 or ISO 8301 apparatus, min. 100 x 100 mm
Thickness	ASTM D3767	Portable ultrasonics or laser equipment
Electrical resistance	EN 14041:2018, Table 13	As per method, selected
Sound absorption coefficient	ISO-10534-2	Impedance tube
Impact sound insulation	ISO 140-8	Impactor (tapping) machine for standard
Mechanical properties	Method	Test conditions
Bond strength (shear)	ASTM D1002	Lap joint rig
Compressive strength	ASTM D395, ASTM D1414	Servo-hydraulic testing machine
Hardness	ISO 48 ISO 7619-1	Indenter
Tensile resistance	ISO 37:2017	servo-hydraulic testing machine

The intention of testing at TRL 6 is demonstration; therefore, some methods were adapted or replaced with equivalent ones corresponding to the same characterization. The testing was extensive and included efforts to identify potential improvements. However, certain mechanical properties not relevant to the intended applications were assessed as "deemed to satisfy without further testing."

3.1.1 Samples

There were two sets of samples tested, as given in Table 3.

Table 3: list of samples.

Designation	Lignin content (%)	Composition (layers, coating)	Description
0%	0	No coatings	Single layer samples
10%	10%	No coatings	Single layer samples with lignin in bulk
20%	20%	No coatings	Single layer samples with lignin in bulk
1% A	1% in coating	4 layers	4 layers, stacking order A
1% B	1% in coating	4 layers	4 layers, stacking order B
3% A	3% in coating	4 layers	4 layers, stacking order A
3% B	3 %in coating	4 layers	4 layers, stacking order B
5% A	5% in coating	4 layers	4 layers, stacking order A
5% B	5% in coating	4 layers	4 layers, stacking order B

Samples were pressed in a single layer or pressed from 4 layers, including coatings on surface of individual layers.

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Single layer samples were produced in general thickness of 10mm. In the bulk of the material, lignin was added in the amounts 0% (no lignin), 10% and 20%, referring to the mass of the material. The details are given in sections 2.3 and 2.4. Multilayered samples were prepared based on thin sheets without lignin. These sheets are described as follows:

3.2 Fire related properties

When addressing fire, two major scenarios related to buildings are considered, corresponding to the stages of fire growth: the ignition and growth phase up to flashover, and the post-flashover phase.

The first, pre-flashover scenario, describes the phase in which the fire is limited to the room of origin. The scenario mainly depends on reaction to fire of the exposed surfaces and limited thickness of underlying layers to incident heat flux. Heat flux originates from the already ignited objects or the source itself. The scenario depends on properties of the exposed materials and construction products, i.e. it

depends on reaction to fire of these products, such as rubber boards. Reaction to fire is therefore relevant for GREEN-LOOP bio rubber sheets.

The second scenario, the post-flashover phase of fire is about preventing of spread of fire to neighbouring parts of building (so called fire sectors) through prevention of fire penetration through structures, such as walls, doors, ceilings etc. The dynamics of fire spread depends on fire resistance of individual structures and their connections to each other. Except in rare cases, such as monolithic concrete wall, it is not a property of one material or construction product, but rather a property of a composed system. As such it is not relevant at single product level, such as a bio rubber sheet.

To assess reaction to fire of construction products and their behaviour in general, several individual characteristics are assessed. This principle has been employed in characterisation of GREEN-LOOP bio rubber boards.

The behaviour of the material has been assessed through a set of tests:

- Behaviour at the fire attack, directly using small ignition source. The corresponding test is the so called "small flame attack", standardized in EN ISO 11925-2[9],
- Behaviour at corner fire attack, responding to the 30kW fire body, placed in corner, lined with the tested material (worst case), the so called "single burning item test", standardized as EN 13823 [10],
- Behaviour at combined attack from above, i.e. ignited by pilot flame and at the same time receiving external heat flux of density, decreasing with distance, the so called "flooring fire test", standardized in EN ISO 9239-1 [11],
- Behaviour at the attack with incident heat flux of various densities on a material exposed surface of 100 mm by 100 mm size (representative for indication of fire behaviour), the so called "cone calorimeter test", standardized in ISO 5660-1 [12],
- Release of smoke and its composition (gaseous fraction) to assess toxic potential of the smoke, the so called "fire smoke toxicity", as described in ISO 19702 [13].

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Additionally, we have been looking at possible improvements of surface properties with respect to fire, i.e. approaches to delay or cancel ignition to provide guidelines for further improvements of the reaction to fire properties of the GREEN-LOOP bio-based rubber products.

The tests have been done on samples with variable lignin content (see Table 3). However, the tests did not confirm any significant effect of sample layering; only the overall lignin content showed a minor influence on the reaction-to-fire results. In the following sections, individual tests and results are described. Finally, all results are summarized in chapter 6.

3.2.1 Small flame attack

The small flame attack is part of reaction to fire testing methods with the purpose of simulating the ability of material to withstand initial ignition from small sources. In this report, all relevant data is given. Part of testing was done within WP3 in as part of final material characterization. In WP6, comparative measurements have not shown any significant difference in respect to fire behaviour.

3.2.1.1 Method description

The tests performed were done according to EN ISO 11925-2. The deviation from the test standard was a slightly shorter test sample, without an effect on the test result. The method represents the reaction of the material to impact of a small fire impingement to surface and edge of material, with a source representing the flame of a lighter.

Small pilot flame is placed next to the surface or lower edge of the vertically positioned specimen for 15 or 30 seconds for classification of E, or possible B to D classification requirements, respectively. During the exposure to the pilot flame and 5 or 30 seconds after removal of the pilot flame, depending on required classification, the height of the flame from burning specimen is observed. If the height of the flame is not higher than 150 mm, the tested product passed requirements of EN 13501-1 for specific classification; 15 seconds of exposure plus 5 seconds additional observation for class E and 30 seconds of exposure plus 30 seconds additional observation for class B to D with additional required results of testing with EN 13823 (SBI) method for class B to D.

Testing is done in a dedicated chamber with a holder for tested sample and propane burner to control unwanted environmental influences, such as draught across the sample.

3.2.1.2 Testing protocol

The testing protocol was according to EN ISO 11925-2. The sample was installed in a sample holder. The source flame was ignited and calibrated, adjusting gas flow. Propane gas (>99% purity) was used. The extraction of the gases from the chamber was done using flow-controlled extraction fan in such a way that the conditions, requested in the standard, were met.

Once the flame height has been calibrated, the source was moved to the test specimen, either at its bottom edge for the so called "edge attack" or to the surface impinging 40 mm above the bottom edge, the so called "surface attack".

The exposure lasted for 15s in the case where class E according to EN 13501-1 was targeted or 30s in the case where class D or above according to EN 13501-1 was targeted.



Figure 6: test specimen and pilot flame, calibrated before the test.



Figure 7: test specimen during the test with surface exposed to the pilot flame.

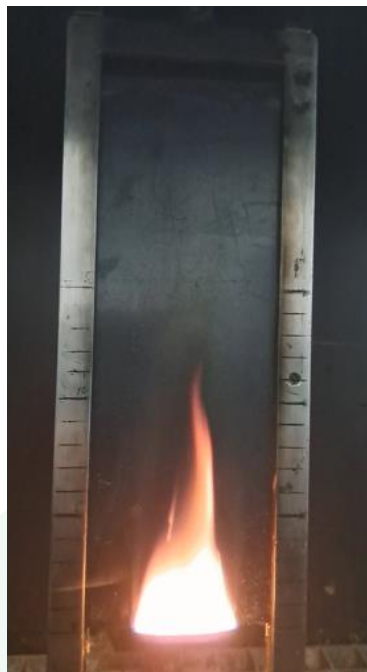


Figure 8: test specimen after removal of the pilot flame, exposed to the lower edge.

3.2.1.3 Results

Testing on finalized material of specimens for potential class determination:

The tests were done in the context of final confirmation of the material formulation. The samples contained 10% and 20% of lignin. Thickness of samples was 3 mm. The testing (exposure) time was 15 seconds (for classification E) and 30 seconds (for possible classification D or better).

Table 4: Results of testing with small flame attack. "YES" in flame occurrence means test fail.

			Did ignition occur?	time /s	Did flame reach 150 mm?	time /s	max height of flame / mm	time /s	Did flaming stop?	time /s	Did filter paper ignite?	Potential classification (EN 13501-1)
10% of lignin	30 s application	surface	yes	7	No		60	30	yes	30	no	Does not support class D or better
		edge	yes	1	Yes	28	170	30	no		no	
	15 s application	surface	yes	8	No		20	15	yes	15	no	E
		edge	yes	1	No		80	20	no		no	
20% of lignin	30 s application	surface	yes	9	No		110	60	no		no	Does not support class D or better
		edge	yes	1	Yes	34	170	34	no		no	
	15 s application	surface	yes	10	No		20	15	yes	15	no	E
		edge	yes	1	No		50	20	no		no	

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Testing on finalized products

Tests on finalized products were done additionally, on formed product, i.e. 13 mm sheets. Based on material confirmation tests it was possible to test final product with 30 s exposure, as the results can be used for both class E and higher classes. The samples with 0%, 10% and 20% lignin were tested.

Thickness of the samples was 13mm. Testing time: 30 seconds (for possible classification D or better)

Table 5: Results of testing with small flame attack

			Did ignition occur?	time / s	Did flame reach 150 mm?	time / s	max height of flame / mm	time / s	Did flaming stop?	time / s	Did filter paper ignite?	Potential classification (EN 13501-1)
0% of lignin	30 s application	surface	no	-	no	-	-	-	-	-	no	E
		edge	yes	2	no	-	100	60	no		no	
	30 s application	surface	no	-	no	-	-	-	-	-	no	
		edge	yes	2	no	-	90	60	no		no	
10% of lignin	30 s application	surface	no	-	no	-	-	-	-	-	no	E
		edge	yes	2	no	-	90	60	no		no	
	30 s application	surface	no	-	no	-	-	-	-	-	no	
		edge	yes	2	no	-	90	60	no		no	
20% of lignin	30 s application	surface	no	-	no	-	-	-	-	-	no	E
		edge	yes	2	no	-	100	60	no		no	
	30 s application	surface	yes	25	no	-	40	30	yes	30	no	
		edge	yes	2	no	-	100	60	no		no	
	30 s application	surface	no	-	no	-	-	-	-	-	no	
		edge	yes	1	no	-	110	50	no		no	

3.2.1.4 Summary of test results:

The comparison of the results obtained on finalized material formulation and shaped products in larger sheets has not revealed any significant difference with respect to reaction to fire, as tested by the protocol according to ISO 11925-2 (small flame attack). This finding makes it possible to use test results on finalized material and shaped product for the same evaluation, with the exception of tests that must be done on larger, composed samples (SBI and reaction to fire of flooring).

This type of tests does not foresee any statistical treatment of the results. Instead, all tests must be passed. For the applications with 15s attack (class E), the product must be at least 3mm thick. For applications with 30s attack (potential class D and higher), the product must be at least 10mm thick. Content of lignin does not influence the potential reaction to fire classes E/D.

The summary of the results is as follows:

Table 6: Summary of the results of testing with small flame attack

Thickness more than 3mm			
% of lignin	Pass all criteria?		
	15s application - edge	15s application - surface	15s application overall
0	Yes	Yes	Yes
10	Yes	Yes	Yes
20	Yes	Yes	Yes

Thickness more than 10mm			
% of lignin	Pass all criteria?		
	30s application - edge	30s application - surface	30s application overall
0	Yes	Yes	Yes
10	Yes	Yes	Yes
20	Yes	Yes	Yes

Grouping all results of testing with small flame impingement show that material with addition of lignin reveals the material can be classified E also for thicknesses as small as 3 mm.

3.2.1.5 Conclusion

Reaction-to-fire performance, as determined by the small flame test, complies with class E. This result is expected, as rubber, the base material, requires appropriate fire-retardant treatments to achieve higher classes. For the intended use of GREEN-LOOP bio-based rubber without direct environmental exposure, class E may be sufficient. However, for applications with exposed material, additional coatings or other protective measures may be required. See 3.2.6.

3.2.2 Single burning item test

The single burning item (SBI) test method is a part of reaction to fire testing methods with the purpose of simulating the ability of material to withstand initial

ignition from medium-sized fire source, such as a trash bin full of paper in the corner of the room.

The deviation from the test standard was a slightly shorter long wing of the test sample, without an effect on the test result.

3.2.2.1 Method description

Test specimen is representing a wall corner with a 30 kW propane burner in the corner. Test specimen is 1,5 m high and has short and long wing 0,5 m and 1 m wide, respectively. The burner in the corner is lit for 21 minutes. During the test, heat release is observed based on oxygen and CO₂ concentration and temperature of exhaust gases. Based on the total heat release in the first 600s (THR_{600s}) and fire growth rate (FIGRA), classification is determined (namely class B, C or D). Another classification is determined from measurements of exhaust gases transparency (class s1, s2 or s3) and by observation of falling flaming droplets or particles (class d0, d1 or d2).

Test specimen was prepared on two plasterboards. On each plasterboard, three boards from tested rubber were mechanically fixed so they represent a surface of approximately 0,5 m width and 1,5 m height on each wing. For the SBI test, the material with 20% added lignin was chosen.

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3.2.2.2 Testing equipment

SBI test was done on standardised equipment according to EN 13823. It consists of a chamber with controlled access of fresh air, corner-shaped sample support, hood and measuring system with sampling of gases: O₂, CO₂ and CO. The system also comprises optical density of combustion gases measuring system as well as control for maintaining set airflow through the device.

3.2.2.3 Testing protocol

The testing protocol used was compliant to EN 13823. In the first step, calibration of the burners is done. In the second step, primary 30 kW burner is ignited, carefully considering the transition time between the ignition and detection of the combustion via oxygen analyser.

During testing, the automated system records all parameters necessary to derive heat release related quantities.





Figure 9: Test specimen before start of the SBI test.



Figure 10: Test specimen at the beginning of the test after ignition of the burner, but before tested material ignited.



Figure 11: Burning test specimen after 10 minutes of testing time, before the test need to be stopped from safety reasons.

3.2.2.4 Results

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The summary of results is shown in Figure 12 and Figure 13.

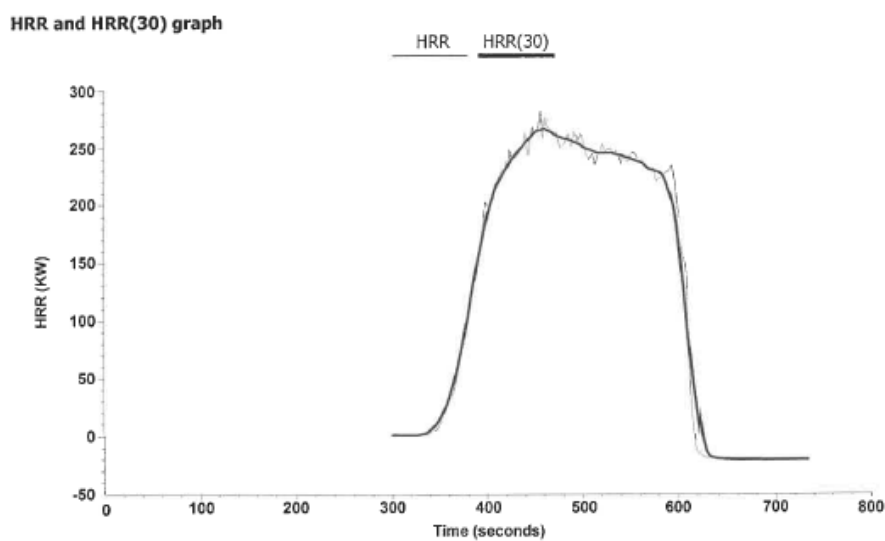


Figure 12: measured heat release rate (HRR), and averaged heat release rate (HRR(30)) in the experiment.



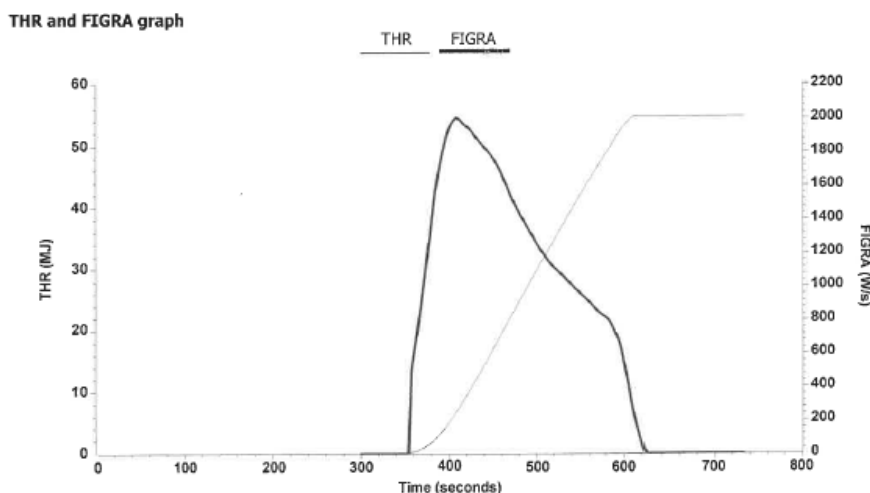


Figure 13: measured total heat release (THR), and fire growth rate index (FIGRA) in the experiment.

Shortly after burner ignition, the specimen ignited. In approximately 10 minutes of testing, which is 5 minutes after primary burner ignition, the heat release and the temperature inside the exhaust duct were too high and from safety reasons the test was terminated early.

Class D was not achieved.

3.2.2.5 Conclusion

The reaction to fire class E prevents use of exposed material as lining (same as e.g. wood). Additional surface treatment is therefore needed for the material to be exposed. This treatment may be in the form of coating or covering with a layer of non-combustible material.

3.2.3 Reaction to fire of flooring

Reaction to fire test for floorings simulates conditions of developed fire, where heat flux from the fire already significantly affects the flooring.

3.2.3.1 Method description

Test method for floorings includes radiant panel heat source and pilot flame igniter. Radiant panel is positioned so that the incident heat flux on the test specimen decreases from one specimen edge to the other along one-meter

specimen length. At the beginning of the test, a pilot flame linear igniter is positioned along the specimen's edge with higher incident heat flux for 10 minutes before the pilot flame is removed. Flame spread is observed during the 30 minutes test duration – the further the flame spreads, lower classification is achieved. The test result represents a critical heat flux value at the position of the flame spread boundary. Together with flame spread, smoke production rate is measured.

3.2.3.2 Testing protocol

The test was performed according to EN ISO 9239-1 testing standard. The sample was installed in a sample holder. Test specimen was around 5 cm shorter than given in standard, but without an effect on the test results.

Test sample was placed on wooden substrate made of oriented strand board (OSB), representing combustible substrates in end use.



Figure 14: Product was placed on the wooden substrate (OSB).



Figure 15: Test apparatus with test specimen before exposure to heat flux.



Figure 16: Ignition of the rubber after pilot flame exposure at one end of the specimen.



Figure 17: Propagation of fire front during the test.

3.2.3.3 Results

D_{fl} class for floorings was not achieved, so based on small flame test the product can be classified E_{fl} when used as a flooring. Substrate did not ignite and did not contribute to the fire.

3.2.3.4 Conclusion

The GREEN-LOOP bio-based rubber product at TRL 6 meets the requirements for class E_{fl}. Although this is not exclusive per se, additional efforts should be put into surface treatment to significantly delay or prevent ignition of the surface. In developing a solution, care should also be given to functional properties, such as flexibility of the coating.

3.2.4 Cone calorimeter– small scale reaction to fire test

Cone calorimeter tests are material tests, done according to ISO 5660-1. These tests have been done in final formulation of the material and on samples cut from the finalized products. Since material tests are not influenced by product geometry, tests on the finalized formulation are relevant for characterizing the final formed products, provided it is demonstrated that the forming process does not affect reaction-to-fire performance.

The samples tested are generally 100mm x 100mm with actual product thickness (nominally 10mm).

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Figure 18: samples for ISO 5660-1 test prior the testing.

The tests were done on samples with 0%, 1%, 3%, 5% lignin and 10% and 20% lignin. Samples with 0%, 10% and 20% lignin were done on samples, cut from finalized products. Samples with 1% 3% and 5% of lignin were done on multilayer formulation of the product. As in small flame attack, these nuances do not affect reaction to fire properties significantly. Hence behaviour is roughly the same, regardless of lignin quantity up to 20%.

The thickness of the samples was roughly 10mm. The size of the test specimen was 100mm x 100mm. One test specimen was tested per configuration: impinging heat flux, content of lignin.

3.2.4.1 Method description

The testing with cone calorimeter is part of both assessment of material and indicative testing of product, measuring reaction of the material to the exposure of a constant heat flux, incident to the surface of the sample.

The essence of the ISO 5660-1 method is exposing one surface of the sample to stable impinging heat flux density. It is realized using a cone heater, which is essentially a resistance-heater coil, with a bottom diameter approximately 200mm, shaped in a cone so that a quasi-closed hemisphere is realized above the sample. This ensures the view factor between the heater and the sample is constant and high. The cone shaped heater top is opened to allow gases and flame rising through to the exhaust duct.

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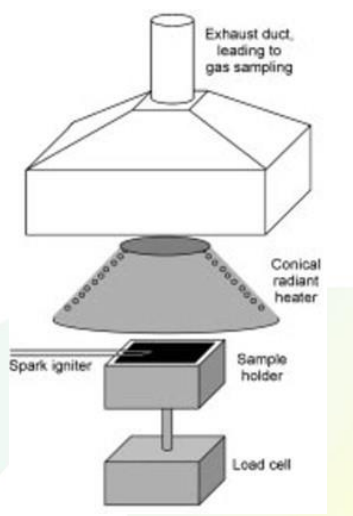


Figure 19: schematics of cone calorimeter heater [14].

The combustion gases are collected in a hood and led in the exhaust duct. At sampling point combustion gases are sampled to measure concentration of oxygen, carbon dioxide and carbon monoxide in the gases, using dedicated paramagnetic (oxygen) or NIR (CO₂/CO) detector.

Test samples of 100 mm × 100 mm dimensions were exposed to heat flux of 10 kW/m² to 25 kW/m². Released flammable gases are ignited with spark igniter. Time of ignition of the test material is observed and heat release is measured based on measured oxygen consumption, CO₂ concentration and temperature of exhaust gases. Furthermore, transparency of exhaust gases and mass loss of the specimen is measured. If needed, exhaust gases can be led to FTIR analyser for toxic gas analyses.

3.2.4.2 Testing protocol

Testing was done on samples with 0%, 10% and 20% lignin. Each sample was exposed to two heat flux densities: 10 kW/m² and 25 kW/m². The test commenced when the heat flux shield on the apparatus is opened, causing heat flux to instantly rise from 0 kW/m² to target value.

Time to ignition was recorded.

The test was done for 600 s or more. The criteria for test termination is also based on HRR, which typically comprises two peaks. The first peak represents ignition period of increased heat release, and the second peak represents heat reaching the bottom of the sample and intensified combustible gases release. The time between the two peaks therefore depends on the thickness of tested specimen.

3.2.4.3 Results

The results of the testing of calorimetric properties: heat release rate (HRR) and fire growth rate index (FIGRA) are given in the following tables and charts.



Figure 20: samples for ISO 5660-1 test during the testing.



Figure 21: samples for ISO 5660-1 test after the testing.

3.2.4.3.1 Different heat fluxes

GREEN LOOP bio-based rubber with 0 % lignin, 10 % lignin and 20 % lignin was tested with incident heat fluxes 15 kW/m² and 25 kW/m².

Recycled rubber with different lignin addition was compared for two incident heat fluxes: 15 kW/m² and 25 kW/m².

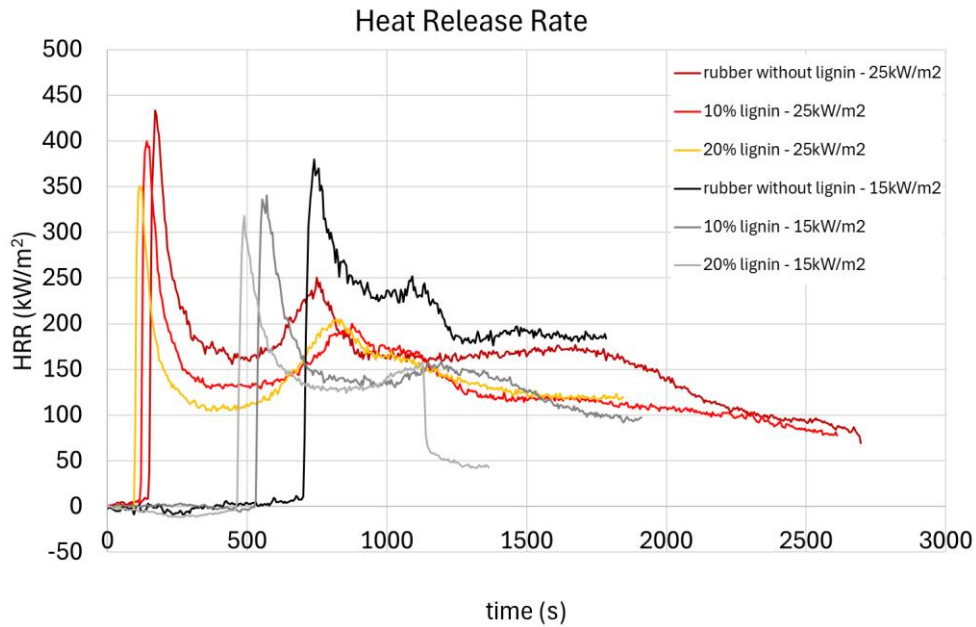


Figure 22: Heat release rate of two samples with 15kW/m² and 25kW/m² heat flux.

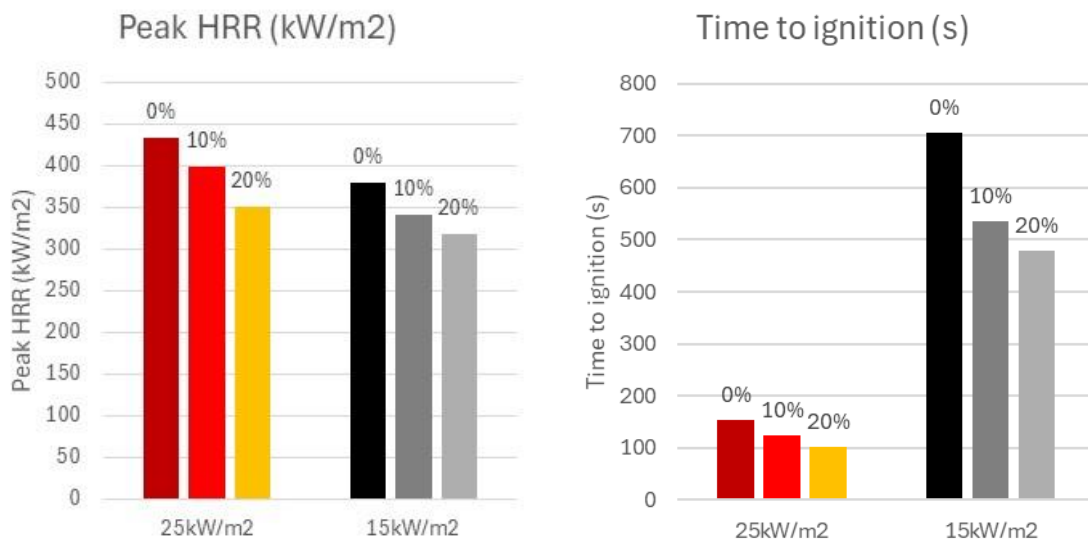


Figure 23: Comparison of results of the heat release rate of three samples with 15kW/m² and 25kW/m² heat flux.

The conclusion derived from the tests is that addition of the lignin does not improve reaction to fire properties but rather worsens it slightly. This would indicate that the added lignin ignites faster than the rubber material. This is supported by measurements of lignin powder.

3.2.4.3.2 Multi layered product

In the course of the material development and variations, a study of the influence of the internal sample composition was done. 4-layered product made of coated recycled rubber of different lignin addition was tested with the impinging heat flux 25 kW/m² and compared along with rubber without lignin, 10 % lignin and 20 % lignin. With respect to multilayered coated samples two different types of products were prepared:

- Product A: all four layers were coated on both sides
- Product B: the two middle layers were coated on both sides; the outer layers were coated on inner layer only.

The results are shown in Figure 24, Figure 25, Figure 26 and Table 7. Also, in this case general trend of shortening the ignition time with increased lignin content is seen.

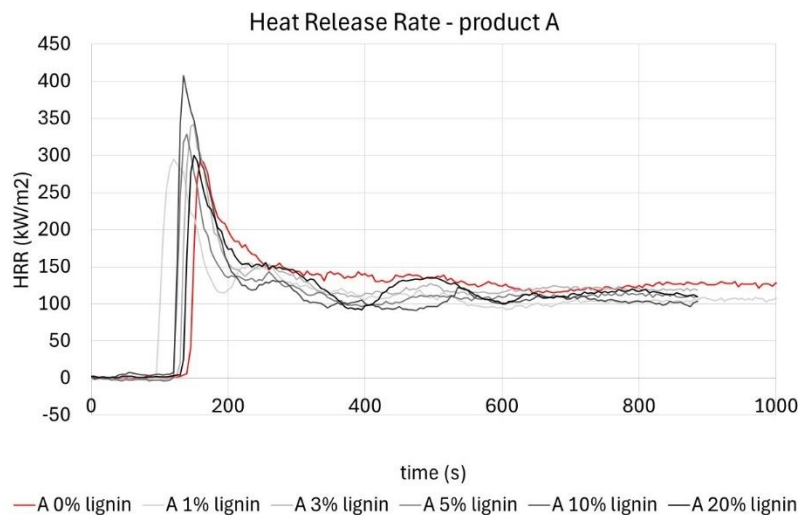


Figure 24: Heat Release Rate (HRR) for product A – comparison between products with different lignin addition [14]

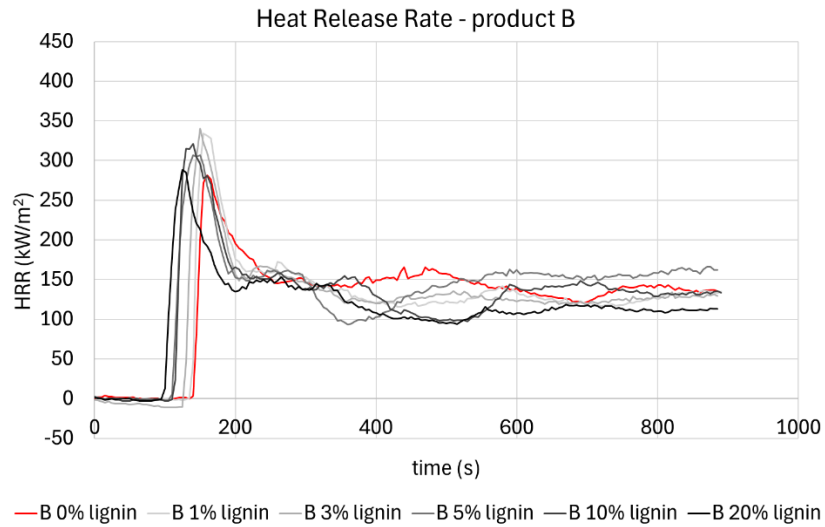


Figure 25: Heat Release Rate (HRR) for product B – comparison between products with different lignin addition

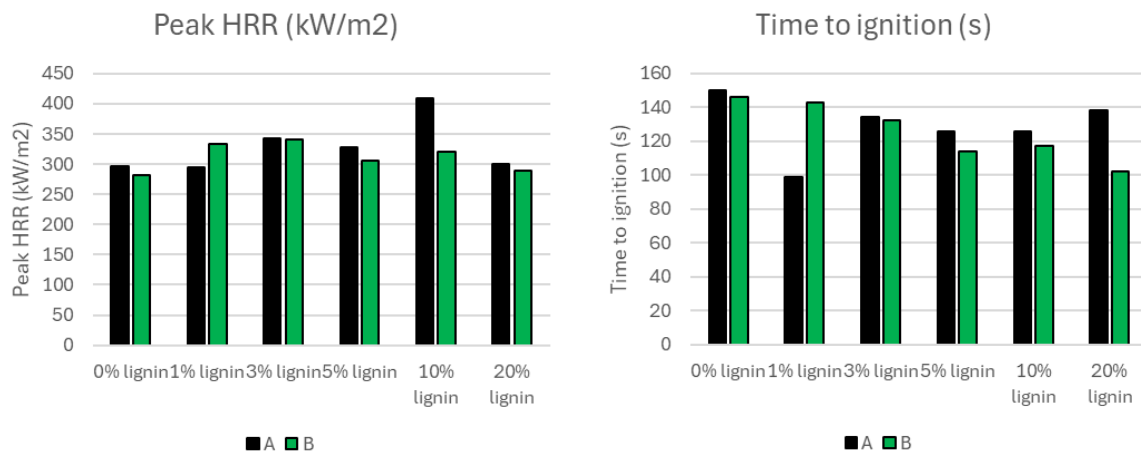


Figure 26: Comparison of peak HRR (heat release rate) and time to ignition between product A and product B with different lignin addition.

Table 7: summary of test results (HRR and time to ignition) for two methods of stacking individual layers in dependence on lignin content.

Lignin content (%)	Peak HRR (kW/m ²)		Time to ignition at 25 kW/m ² (s)	
	A	B	A	B
0	296	281	150	146
1	295	334	99	143
3	342	340	134	132
5	329	306	126	114
10	408	321	126	117
20	300	288	138	102

3.2.4.4 Conclusion

There are minor differences found among the samples, depending on lignin content. The differences are found in time to ignition and the dynamics of heat release rate (peak, steady state value, total heat released in certain time). However, the differences are not significant, which leads to the conclusion the lignin in the sample does not significantly alter behaviour of the GREEN-LOOP bio-based rubber products with respect to reaction to fire. It seems the presence of lignin slightly worsens the performance. The most likely explanation to this phenomenon is that while lignin clearly exhibits intumescent properties, it still burns at the temperature or incident heat flux intensity as it is found at the surface of the sample. Lignin in the core of the material does not provide significant cooling or oxygen access restriction without heat release.

The differences in stacking order of the layers does not seem to have influence on reaction to fire parameters.

3.2.5 Toxic gases and smoke

3.2.5.1 Method description

The method used was based on the combination of ISO 5660-1, coupled with FTIR and analysis, compliant to ISO 19701. There were no deviations from the test methods.

The essence of the test method is generation of combustion gases, as described in 3.2.4.1. In addition, at the sampling point an additional gas stream is sampled and led via heated pipeline (180°C), to avoid any danger of condensation, to the FTIR with a heated test cell (180°C). The FTIR samples spectra approximately 1/3s. The spectra are analysed using dedicated software, where time dependent concentration of gases is resolved. The software analyses for 22 various gases, detectable by FTIR technique and normally found in combustion gases. This analysis is targeted, but not limited to acutely toxic gases, i.e. gases that cause an immediate adverse reaction.

In the combustion gases, constituents not detectable by FTIR may be present, such as dioxins (PCDD), furans (PCDF) or polycyclic aromatic hydrocarbons (PAHs). Test to determine these compounds are very extensive, and it only makes sense to test the material on a fully developed product, if the composition (e.g. presence of chlorine or bromine) can indicate the possibility of PCDD/F or PAH creation.

3.2.5.2 Testing protocol

The testing of toxicity is done in parallel to the calorimetric testing. All details on samples apply as described in 3.2.4. Sampling of the combustion gases for toxicity analysis was done fully in parallel with calorimetric measurements.

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3.2.5.3 Results

All 22 compounds, detected by FTIR have been analysed for their toxicity. However, a selection of those, present in the combustion gases in a non-marginal quantity: SO₂, ammonia and CO (in open air cone calorimeter) is presented in following figures: Figure 27, Figure 28 and Figure 29. In Figure 30, a summary of the results is shown as percentage of the allowed value per different compounds. Only sulphur dioxide is present to a notable level, however it remains below 50% of limit values as applied in ship building industry. These limit values are considered more demanding than those that may apply for buildings. However, due to lack of legislative framework on composition of combustion gases across the EU the limiting values for construction sector are not (yet) set.



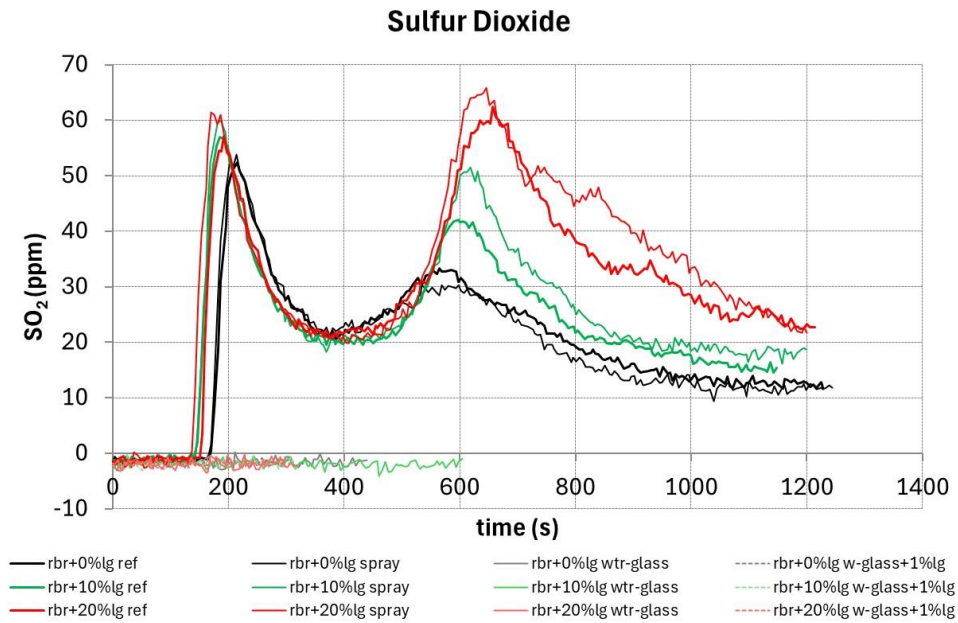


Figure 27: time dependant concentration of SO2 for all tested samples.

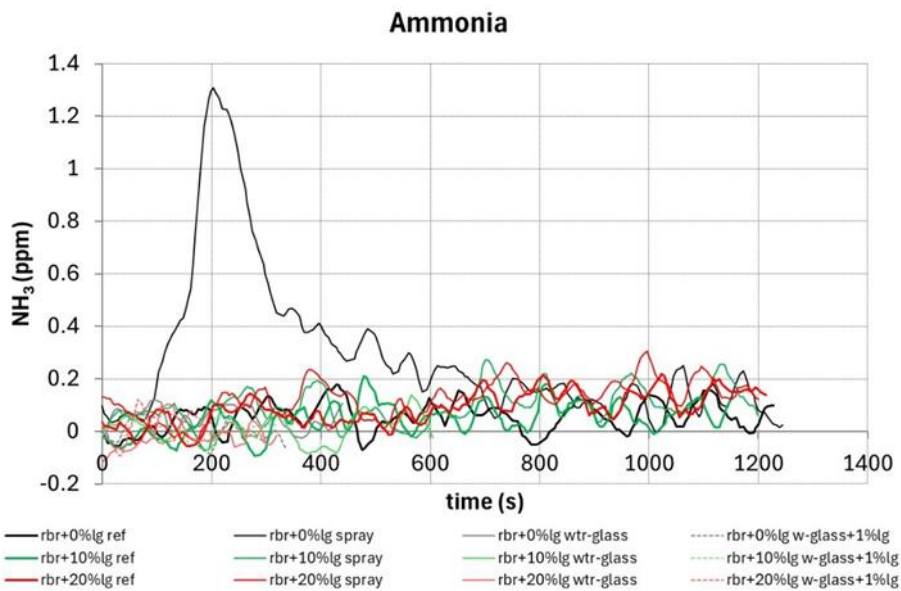


Figure 28: time dependant concentration of ammonia for all tested samples

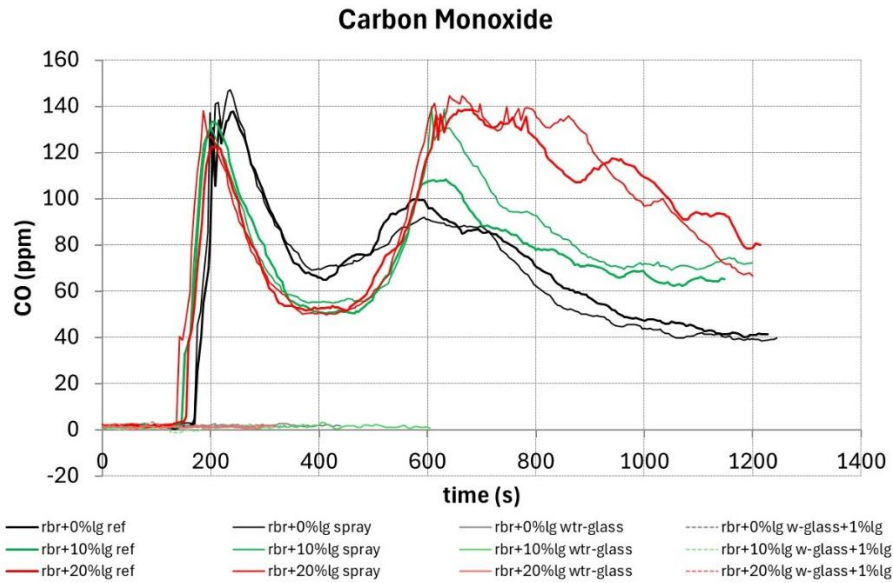


Figure 29: time dependant concentration of carbon monoxide (measure of oxygen access) for all tested samples.

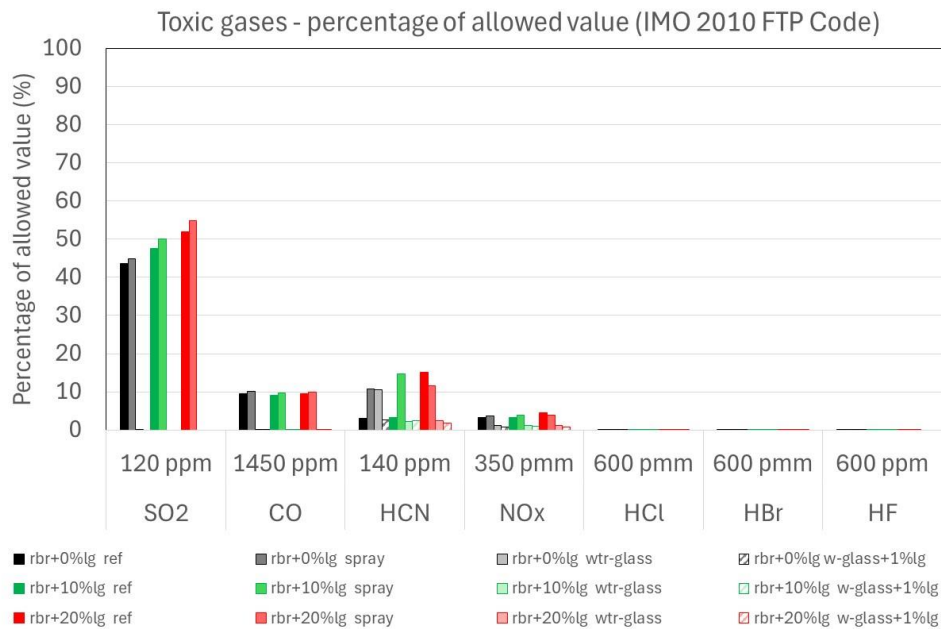


Figure 30: normalized toxicity potential (relative to limit values) as measured in individual sample configuration for compounds with known (acute) toxic potency.

3.2.5.4 Conclusion

In conclusion, the combustion gases do not have a content of toxic gases to the extent that would raise concern about the toxicity. Focus should be given to SO₂, originating most likely from remains of vulcanisation, however values found are safely below the allowed limits as given in the IMO 2010 FTP code.

3.2.6 Additional improvements

In order to explore options to improve reaction to fire of the GREEN LOOP bio-based rubber application of surface treatment with fire retarding agents was considered. Several options were explored:

- addition of commercial fire retardant intended to improve reaction to fire of textiles,
- addition of water glass (sodium silicate water solution) on the surface (approximately 1 kg/m²),
- addition of water glass with admixed lignin (1% in weight) to improve flexibility and adhesion of the samples.
- addition of water glass with admixed lignin (0.5% in weight) to improve flexibility and adhesion of the samples (in one case only).

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The coatings have been applied to the base GREEN-LOOP bio-rubber test sample with 0% lignin, 10% lignin and 20% lignin. All 3 bases were tested without coating as a reference and with listed coatings applied. In total 13 combinations have been measured. The matrix is seen in the results Table 8 and in Figure 32. Both thermal properties (i.e. heat release rate and time-to-ignition) and toxicity of combustion gases were assessed.

3.2.6.1 Method description

The method is in detail described above. ISO 5660-1 was followed. Because of intumescence of surface materials wire restrainers were applied, however the intumescence was very intensive and the restrains did not prevent surface of the material to rise towards the heat source. By that the impinging heat flux density was higher than calibrated. However, the results are on the safe side, as higher impinging heat flux density accelerates combustion.

3.2.6.2 Testing protocol

In total 13 different variants have been tested. The coatings were applied



The measurements were done in sets, based on percentage of lignin in the rubber formula. For each basis a series of tests was done:

1. Reference (without any coating)
2. Reference + coating 1 (commercial coating)
3. Reference + coating 2 (water glass)
4. Reference + coating 3 (water glass + 1% wt. lignin)
5. Reference + coating 4 (water glass + 0.5% wt. lignin) – only with 0% lignin rubber

3.2.6.3 Results

The results of individual measurements are shown in

Figure 31 and Figure 32. Again, the general trend of slight worsening of reaction to fire properties by adding lignin into rubber is confirmed.

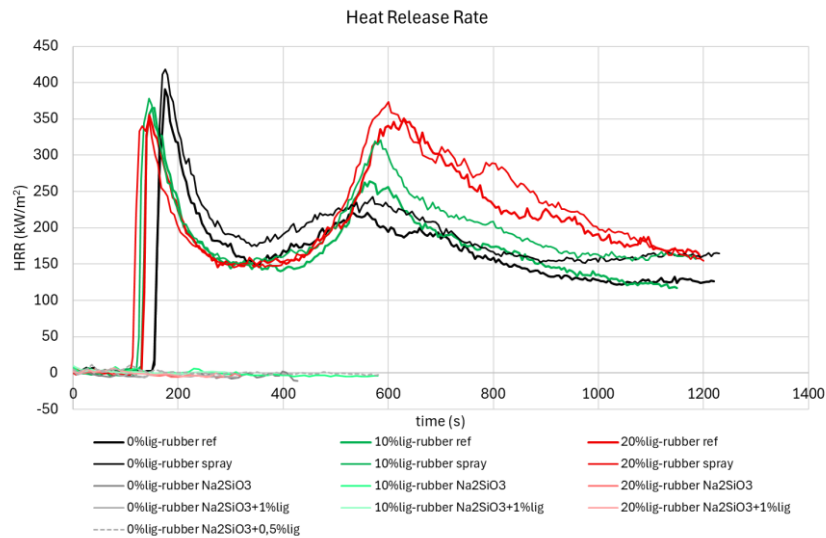


Figure 31: Heat release rate with applied fire retarding coatings.

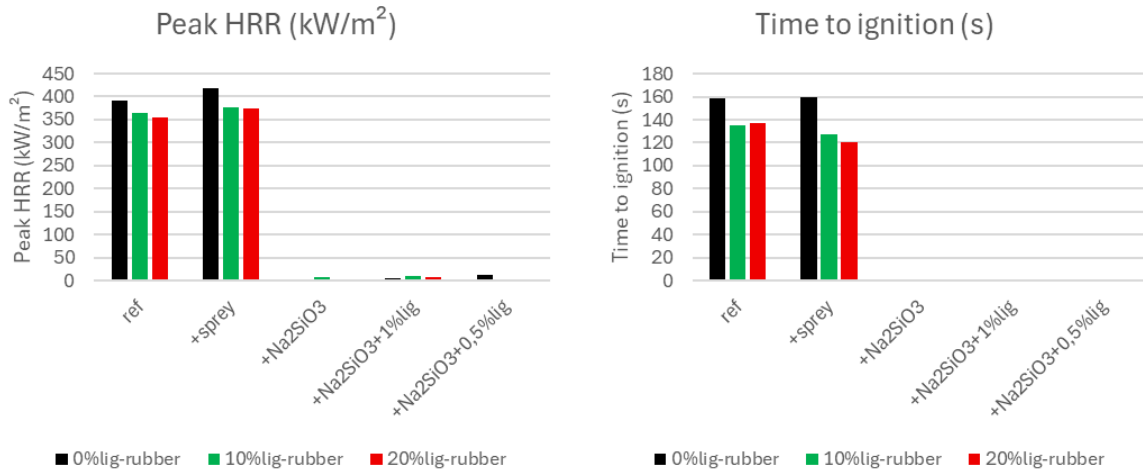


Figure 32: comparison of peak heat release rate (HRR) and time to ignition for various fire-retarding coatings applied to base GREEN LOOP bio-based rubber.

Table 8: Results with fire retarding coatings applied.

Time to ignition (s)			
Basis	0% lignin	10% lignin	20% lignin
Coating			
None	159	135	137
Commercial	160	127	120
Water glass	No ignition	No ignition	No ignition
Water glass + 1% lignin	No ignition	No ignition	No ignition
Water glass + 0.5% lignin	No ignition		
Peak HRR (kW/m ²)			
Basis	0% lignin	10% lignin	20% lignin
Coating			
None	391	365	355
Commercial	418	378	373
Water glass	2*	8*	3*
Water glass + 1% lignin	6*	10*	6*
Water glass + 0.5% lignin	12*		

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* smouldering, no ignition

3.2.6.4 Conclusion

Conclusion: the application of well-known fire retarding material (38% water glass solution) on the surface of the product prevents the ignition of the samples under the tested conditions. The effect is not diminished with the addition of 1% lignin into the water glass solution which is admixed to improve the adhesion properties of the coating to the GREEN-LOOP bio-based rubber.

3.3 Safety related properties

3.3.1 Content of dangerous substances

Content of dangerous substances is formally determined by the composition of ingredients. It does not account for possible reactions between ingredients.

3.3.1.1 Results

The list of ingredients of the GREEN LOOP bio-based rubber is the following

Table 9: constituents of GREEN LOOP bio-based boards.

No	Constituent	Code
1	Devulcanised rubber (Styrene butadiene rubber, Polybutadiene rubber, carbon black)	CAS no: 9003-55-8 CAS no: 9003-17-2 CAS no: 1333-86-4
2	Lignin powder	CAS no: 8068-05-1
3	Natural Rubber SMR CV60	CAS no: 308075-06-1
4	Low Lead Zinc Oxide	CAS no: 1314-13-1
5	Stearic Acid	CAS no: 57-11-4
6	MBTS-75 (accelerator)	CAS no: 120-78-5
7	300 Mesh Sulphur	CAS no: 7704-34-9

3.3.2 Emission of dangerous substances

Sampling and analysing VOCs from rubber panels is challenging due to their volatility, low concentrations and complex matrix. Experimental examinations using chamber and cell methods are expensive and time consuming, and small-scale laboratory methods are faster, cheaper but more influenced by sample preparation and environmental parameters. As has been shown on other materials, there is significant correlation between small scale and chamber methods [15], [16], which makes them an effective preliminary test, suitable for analysis of novel materials such as our bio-rubber composite. The product has been characterized with respect to emission of dangerous substances in leachate for potential contact with water and to indicate possible results of full VOC in air determination test campaign.

3.3.2.1 Method description

The testing was done on a sample of GREEN-LOOP bio-based rubber. The sample is immersed into solvent solution and left at the laboratory conditions for sufficient time to leach and extract individual compounds into the solvent. The solvent containing the sample is then tested using gas chromatography coupled to mass spectrometry (GC-MS) analytical technique. Retention times for individual components are measured in the GC part. The GC separated compounds are led to the mass spectrometer to assign the molar mass of individual compounds and base do that derive qualitative composition of the sample. The quantification is done using dedicated software tools.

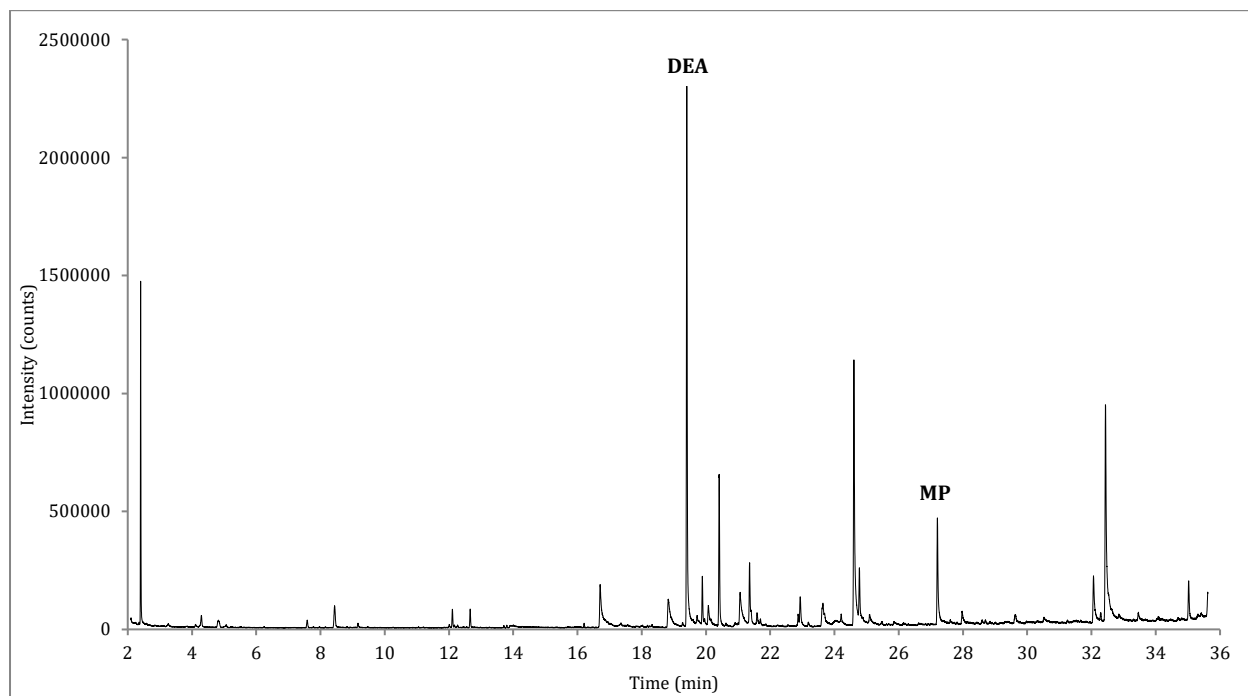
3.3.2.2 Testing protocol

The GREEN LOOP bio-based rubber sample was cut to approximately 1 mm thick particles. Approximately 3 g of the particles was weighed into 50 ml PE centrifuge tube with PP screw cap and around 10 g of methanol (SupraSolv® Methanol for gas chromatography MS, Merck, Darmstadt) was added. The exact mass of the rubber particles was recorded. Extraction was conducted at room temperature for around 50 days. For qualitative analysis this was used in further procedure. For a quantitative analysis a known amount of internal standard (isobutanol – iBuOH), VOC stopper (diethyl adipate – DEA) and SVOC stopper (methyl palmitate – MP) was added to the centrifuge tube.

Gas chromatography–mass spectrometry analysis was performed on a 7890B gas chromatograph (Agilent, Santa Clara, CA, USA) coupled with a quadrupole mass detector (5977B). The GC-MS conditions were as follows: column, DB-5 MS Ultra Inert (Agilent, Santa Clara, CA, USA); injected volume, 1 µL; inlet temperature, 250 °C; carrier gas, He; and split ratio, 1:20. Temperature program: Initial temperature, 35 °C; hold time, 5 min; ramp rate, 8 °C/min; final temperature, 280 °C, hold time, 5 min. The components were identified based on the mass spectra in comparison with probability-based matching (Agilent, Santa Clara, CA, USA). Quantitative analysis was performed based on the peak area of each component compared to the peak area of the internal standard (iBuOH). Both were done using Agilent Masshunter software. A relative response factor 1 against internal standard iBuOH was used in calculations for all detected compounds.

3.3.2.3 Results

The GC-MS chromatogram of the extract is presented in Figure 33. Identified and quantified compounds are listed in the Table 10.



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Figure 33: chromatogram of the sample. The data for the compounds are given in Table 10.

Individual peaks were identified based on retention time and MS identification. Compounds with identification quality $\geq 70\%$ and $> 0.001\%$ mass fraction are presented. VOC and SVOC content results are given in Table 11.

Table 10: VOC and SVOC compounds with identification quality $\geq 70\%$ and $> 0.001\%$ mass fraction.

Retention time (min)	Compound	Mass fraction (%)	Quality (%)
VOC			
8.44	o-xylene	0.015	95
9.17	2-hydroxy-2-cyclopenten-1-one	0.002	76
12.11	1,1-dimethoxy-cyclohexane	0.008	80
12.66	limonene	0.008	94
16.21	dodecane	0.002	70
16.71	benzothiazole	0.050	94
16.95	1,2-benzisothiazole	0.002	86
18.83	2,6-dimethoxy phenol	0.044	97
SVOC			
20.08	Dicyclohexanamine	0.016	90
20.41	1,2-dihydro-2,2,4-trimethyl-quinoline	0.073	94
21.07	apocynin	0.049	97
21.36	3,4-dimethoxymandelic acid, methyl ester	0.034	72
21.59	(+)-6-chloro-4-methyl-2,3-dihydro-4H-1-benzopyran	0.010	83
22.87	4-(1,1,3,3-tetramethylbutyl) phenol-	0.006	90
22.94	2-(methylmercapto)benzothiazole	0.022	96
24.03	2(3H)-benzothiazolone	0.004	76
24.61	3,5-dimethoxy-4-hydroxyacetophenone	0.170	97
24.78	2,3-dimethyl-4-biphenylamine	0.037	72

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Table 11: total VOC and SVOC content in the leached compounds.

VOC content (%)	SVOC content (%)
0.166	0.545

The limits of VOC compounds may be laid down in various legislative acts that are not necessarily harmonized across the EU with respect to limit values of individual compounds. In general, however, none of the listed compounds are classified as highly toxic or extremely hazardous in typical laboratory or industrial contexts. However, several are moderately toxic and can cause irritation, skin or eye damage, or respiratory issues if not handled properly.

In summary:

- Benzothiazole and 1,2-Benzisothiazole have moderate toxicity and potential irritant effects.
- o-Xylene is more toxic compared to many of the other compounds and can affect the central nervous system and organs with significant exposure.
- Dodecane and limonene generally pose low toxicity risks but can cause irritation.

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These compounds should be considered in more detail once the final entry to the market is done. Additionally, a VOC chamber test should be carried out once the product is fully ready for market, as the legislation usually requires concentration values measured in chamber-based standard tests.

3.4 Environmental characteristics

3.4.1 Determination of the Global Warming Potential (GWP)

Measuring Global Warming Potential (GWP₁₀₀) involves assessing the greenhouse gas (GHG) emissions associated with the product's life cycle, expressed in CO₂ equivalent (CO₂eq). To obtain a complete GWP analysis, various factors must be taken into account, such as the type of rubber (synthetic or natural), additives used (lignin), and the energy consumption of each stage of the process. This calculation was carried out using a Life Cycle Assessment (LCA), with the help of the latest versions of SIMAPRO software and the ecoinvent database. The steps to follow to carry out the LCA are:

1 - Definition of objective and scope: the purpose of the study, the boundaries of the system (which processes will be included), and the functional unit (e.g., 1 kg of rubber panel material) are established.

2 - Life cycle inventory (LCI): All input flows (raw materials, energy) and output flows (emissions, waste) associated with each stage of the product's life cycle are collected and quantified.

3 - Life cycle impact assessment (LCIA): The inventory data is translated into environmental impacts, such as Global Warming Potential (GWP), acidification, eutrophication, among others.

4 - Interpretation: The results are analysed, critical points are identified, data quality is assessed, and improvements are proposed. This phase also ensures that the conclusions are consistent with the defined objectives.

The complete environmental characteristics of each of the scenarios evaluated for this value chain are found in *WP2: Sustainability and Circularity by design* and respective reports, *D2.2 Energy, exergy, LCA, LCC and sLCA* as the intermediate preliminary evaluation, which has been updated in D2.3.

3.4.1.1 Method description

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CML-IA baseline V3.10 / EU25 is the method used to obtain the GWP values for each of the scenarios evaluated in this value chain. This methodology is based on a mid-point impact characterisation approach, which means that it translates inventory flows into intermediate potential environmental impacts, such as Global Warming Potential (GWP), acidification, eutrophication, ozone depletion, among others. It is adapted to the European context (25 EU countries) and provides consistent and widely accepted characterisation factors for comparability between studies. In SimaPro, this method is commonly used to assess environmental impacts in LCA studies following a structured approach compatible with the ISO standards mentioned above.

3.4.1.2 Testing protocol

Details of the calculations are reported in *D2.2/D2.3. Energy, exergy, LCA, LCC and sLCA*. As the work carried out in this deliverable focuses on the construction sector, the analysis explained here follows the CML methodology mentioned in the previous sections, while the analyses included in deliverables D2.2 and D2.3 follow the Environmental Footprint methodology, which has been developed by the



European Commission within the framework of the Product Environmental Footprint (PEF) initiative. It provides a mid-point approach but is specifically designed to harmonise the environmental assessment of products and organisations in the European context. When working in a European context or with products that must comply with EU regulations, the EF method is usually more appropriate.

3.4.1.3 Results

The Global Warming Potential (GWP) has been calculated for all the scenarios considered in the Life Cycle Sustainability Assessment (LCSA): Current synthetic rubber, Bio-Rubber Panel 1 (BRP1) with 20% of lignin, and BRP2 with 0% of lignin. It is important to highlight that the same lignin process has been included as an additive in the current panel manufacturing process, due to it is a benchmark that has a higher TRL than the two bio-based scenarios.

GWP1 = 63.9 kgCO₂eq / kg of current panel material produced.

GWP2 = 65.1 kgCO₂eq / kg of BRP1 panel material produced.

GWP3 = 5.28 kgCO₂eq / kg of BRP2 panel material produced.

With the results calculated above using SIMAPRO and ecoinvent, it is possible to observe the drastic reduction in GWP when lignin is no longer used as an additive in the development of the material for the panels.

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3.5 Thermal properties

3.5.1 Thermal conductivity

Although the bio rubber under development was primarily intended for sound and vibration insulation of buildings thermal properties namely its thermal conductivity could also be important and was, therefore, determined in a range of temperature of 25°C to 120°C. As part of this evaluation thermal diffusivity and specific heat were also determined. The rubber compositions tested are presented in Table 12.



Table 12: Bio rubber compositions

Compounded bio-rubber	Components			
	Additives	Natural rubber	Thickness (mm)	Purpose
GL-P-022A	0 wt% Lignin	20 PHR	3	Final characterization testing
GL-P-017B	10 wt% Lignin	20 PHR	3	
GL-P-018A	20 wt% Lignin	20 PHR	3	

3.5.1.1 Method description

The measurements were made with the well established flash method using NETZSCH 457 MicroFlash equipment, whereby the front surface of a specimen is heated by a short pulse laser and the temperature increase on the rear surface measured by an infrared detector. All measurements were performed in an inert gas atmosphere. The test equipment is presented in Figure 34.

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Figure 34: Netzsch LFA 457 thermal test equipment

3.5.1.2 Testing protocol

Measurements were performed on disc shaped specimens with 12.6 mm diameter and 2.5 mm thickness. The specimens were coated with a thin layer of graphite, about 5 µm thick to eliminate possible problems related with transparency, reflections and emission absorption in certain wavelengths. Vacuum is initially made in the test chamber followed by a purge with nitrogen. The measuring equipment was validated against a reference sample of Pyroceram 9606.

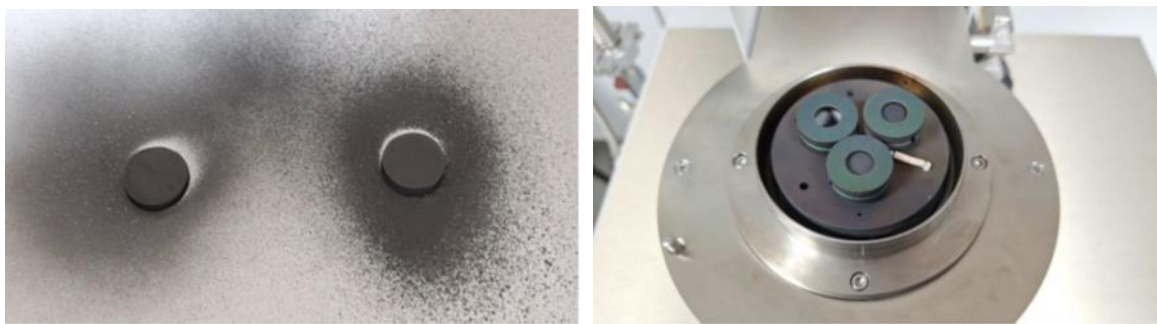


Figure 35: Graphite coating of the specimens (right); and specimens mounted in the testing machine (right)

3.5.1.3 Results

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The thermal properties of the bio rubber are presented in Figures 36 to 38. All materials showed a peak more noticeable for some properties than others between roughly 50 and 70 °C, repetitions of measurements at these temperatures confirm this behaviour but reasons for this behaviour are not obvious at this stage since it does not seem to be directly connected with the lignin content. Although lignin content slightly decreased the thermal conductivity and thermal properties of the rubber the behaviour of all materials was fairly similar.

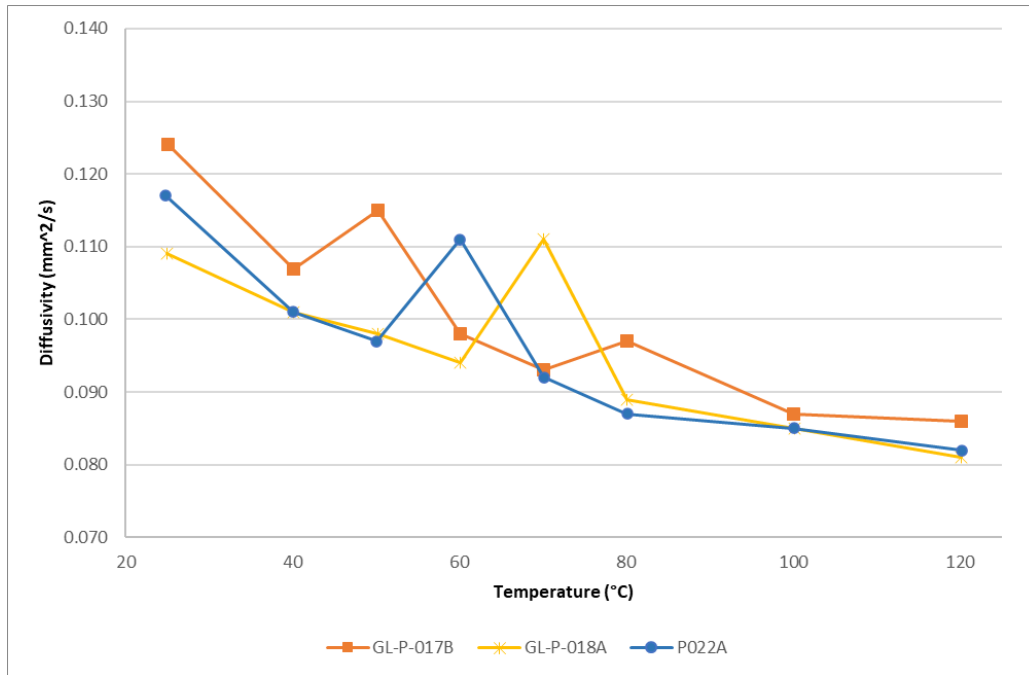


Figure 36: Thermal difusivity of the bio rubber

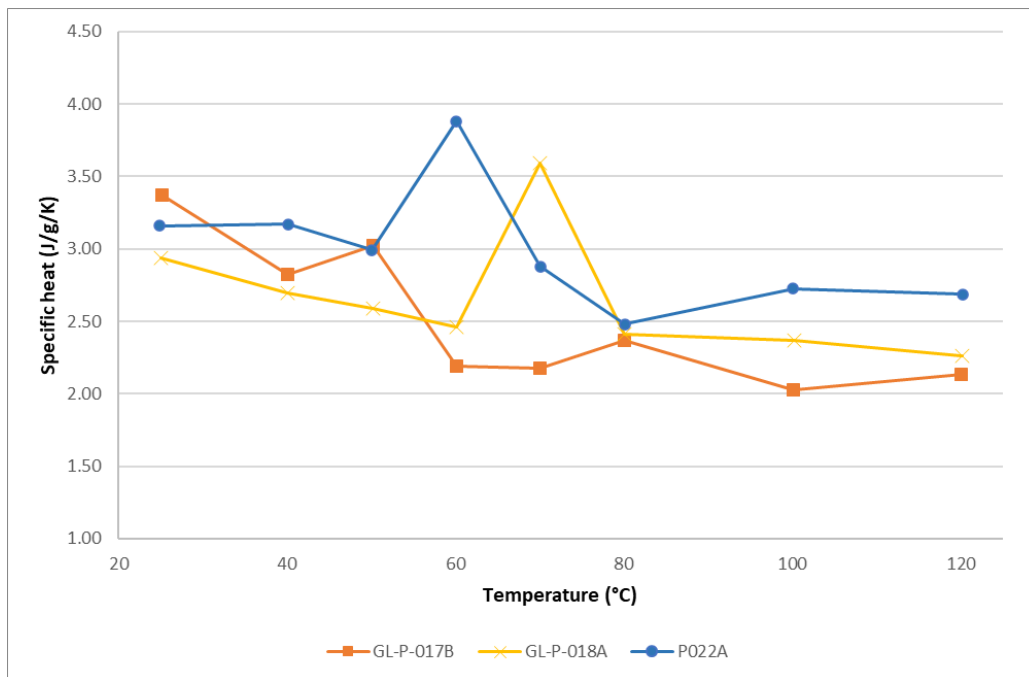


Figure 37: Specific heat of the bio rubber

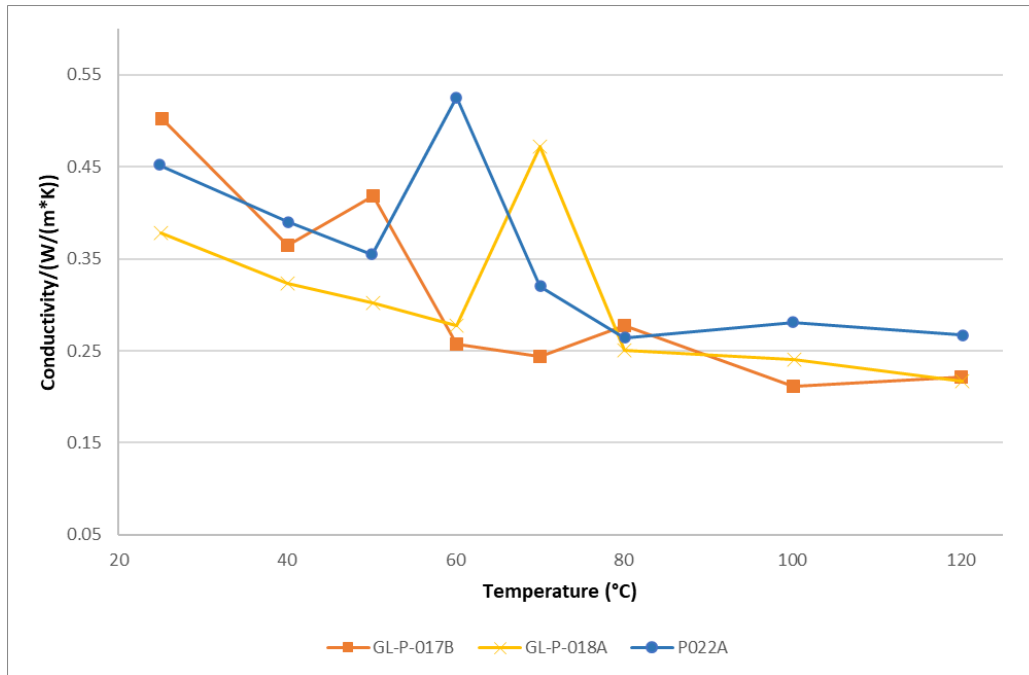


Figure 38: Thermal conductivity of the bio rubber

3.6 Electrical and acoustical (vibrational) properties

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Electrical properties are generally determined by measuring of electrical resistance of the flooring material. However, this only applies to materials that are exposed to electrostatic charging.

3.6.1 Electrical resistance

3.6.1.1 Method description

The electrical resistance measurements were performed following a simple internal procedure used to evaluate the resistance of electrical insulators using a direct measurement with a mega ohmmeter. The bio rubber evaluated are presented in Table 13.

Table 13: Bio rubber materials tested for electrical resistance

Compounded bio-rubber	Components			
	Additives	Natural rubber	Thickness (mm)	Purpose
GL-P-022A	0wt% Lignin	20 PHR	3	Final characterization testing
GL-P-017B	10wt% Lignin	20 PHR	3	
GL-P-018A	20wt% Lignin	20 PHR	3	
GL-P-019A	0wt% Lignin	20 PHR	13	
GL-P-021A	20wt% Lignin	20 PHR	13	

3.6.1.2 Testing protocol

Specimens with approximately 120x55 mm were cut from bio rubber panels Figure 19. Metallic electrodes were applied to them and the electrical resistance evaluated through thickness using the mega ohmmeter.

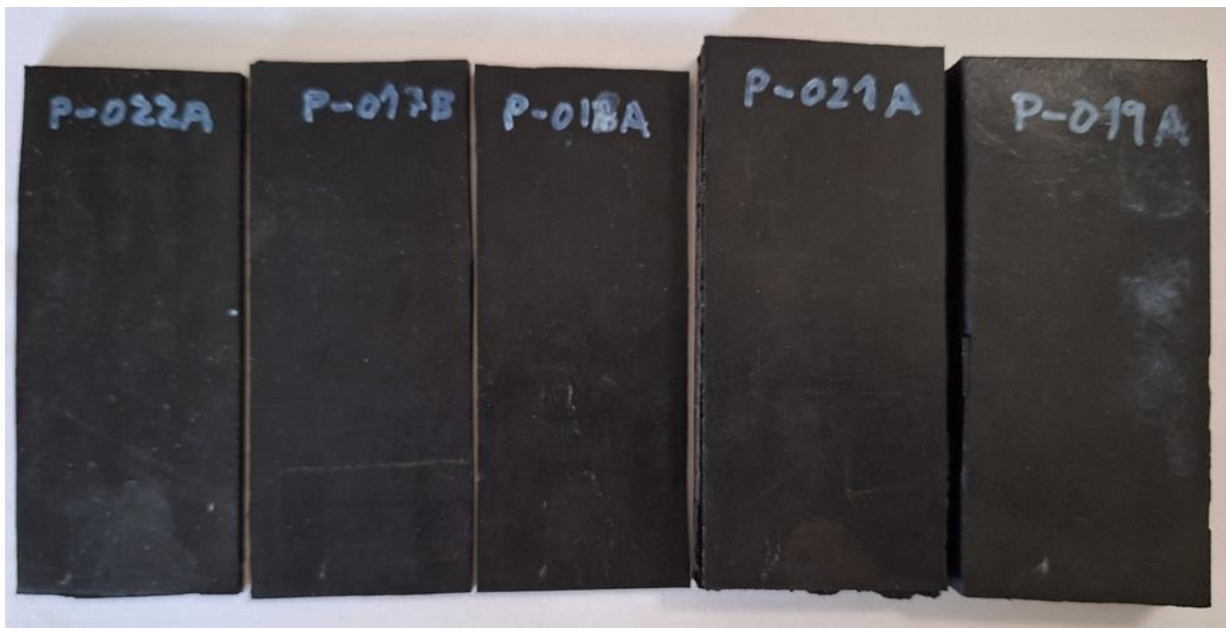


Figure 39: Bio rubber specimens used for electrical resistance measurements

3.6.1.3 Results

The electrical resistance values obtained are presented in Figure 40. All materials exhibited an electrical resistance in the GΩ range as required. The resistance consistently decreased with increasing Lignin content and quite noticeably for the 20% lignin material. Surprisingly the resistance also showed a sharp decrease with increasing thickness of the material.

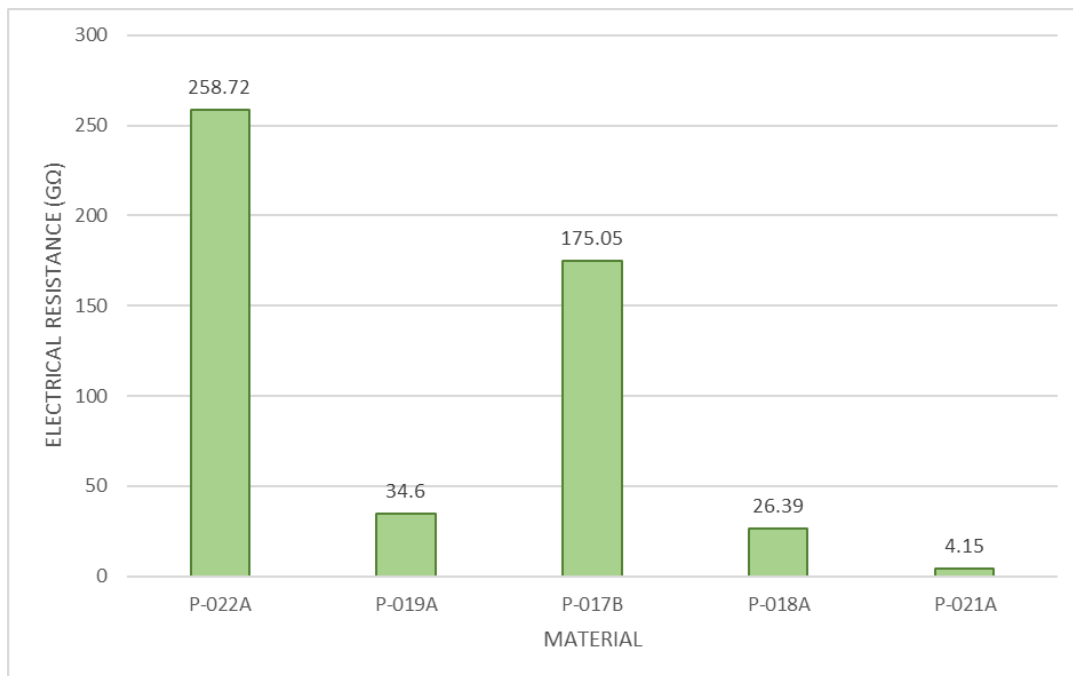


Figure 40: Electrical resistance values determined for the different bio rubber material

3.6.2 Vibrational behaviour

3.6.2.1 Method description

Large-scale panels were tested on a custom and reconfigurable platform designed for GREEN-LOOP to analyse the response under hammer, pulse and sine sweep (electrodynamics shaker) and walking tests. The platform and the testing procedures were designed following recommendations and guidelines presented in the 2006 EUR 21972 EN report [17]. The ISO 2631-2 standard was followed to manage the signal processing and obtain the root mean square (RMS) responses for the different load cases.

The reconfigurable testing platform is shown in Figure 41 and Figure 42. The same platform can be adjusted to perform ground-based tests (impact hammer and walking tests), as well as elevated tests for pulse and sine-sweep excitations. The length of the platform can be adjusted from 3 m to 5 m by varying the spacing between movable supports. The variable length allows to provide a flexible and stiffer platform to explore the performance of the rubber panels between the frequency range 0.5 Hz-100 Hz.

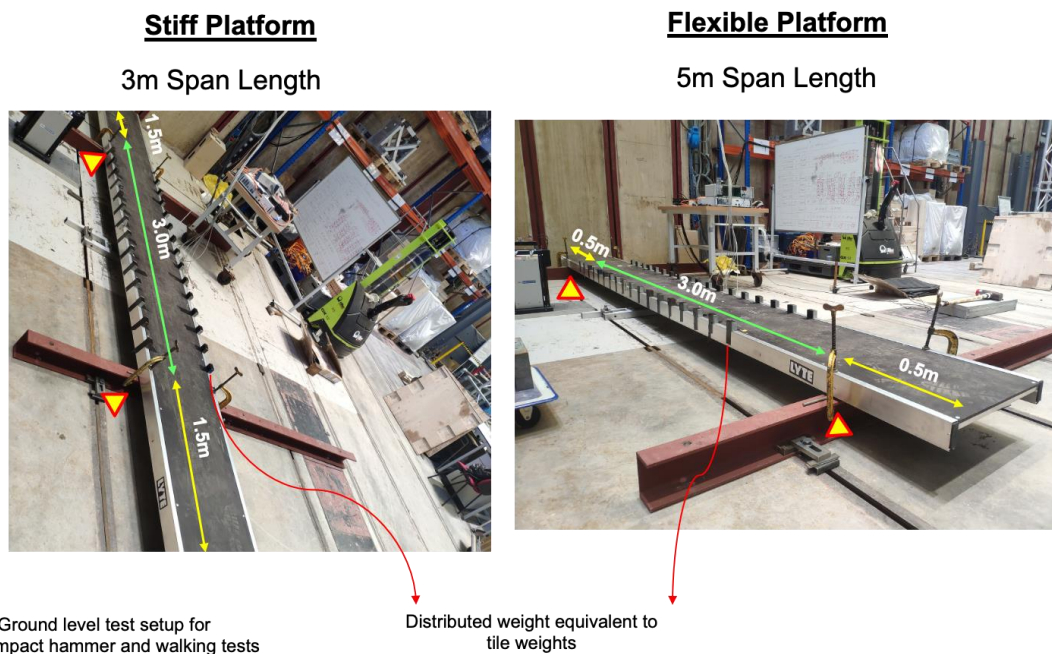
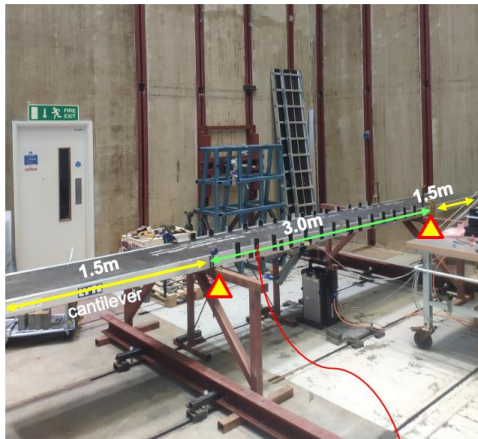


Figure 41. Ground level test setup for the large-scale vibration measurements

Stiff Platform

3m Span Length



*Elevated test setup for pulse and sine-sweep tests

Distributed weight equivalent to tile weights

Flexible Platform

5m Span Length



Figure 42. Elevated test setup for the large-scale vibration measurements

A large sledgehammer with a CB Piezotronics – 086D50 force transducer was used to apply input excitation during testing. The hammer has a 5.5 kg head and a force transducer with a sensitivity of 0.23 mV/N ($\pm 15\%$), allowing accurate measurement of impact forces up to $\pm 22,240$ N peak. Electrodynamics excitation was applied using an APS Dynamics Model 420 electrodynamic shaker, capable of delivering up to 900 N peak sine force over a frequency range of 0.1–200 Hz. It has a long-stroke capacity of 150 mm and a peak sine velocity of 1 m/s. The forces applied by the actuators were measured using 700-series precision 2.5 kN ‘S’-type beam load cells, designed for tension/compression and bi-directional testing. These load cells have a non-linearity of $<0.05\%$ of full-scale output and a hysteresis of $<0.05\%$ of full-scale output. Each load cell includes an in-line conditioning module, providing a ± 10 V output. The load cells are five-point calibrated with traceability to national standards and feature M12 \times 1.75 threaded connections at both ends. They were mounted beneath the platform and connected to the test panels via M12 bolts. An M12-to-M6 adapter was used to connect the bottom of each load cell to the stinger bar attached to the actuator. The thin stinger bar decouples cross-axis force input while being rated for loads up to 900 N. The BK Precision 4052 was used as the

signal generator in the tests. It is a dual-channel function/arbitrary waveform generator capable of producing a wide range of signal types. For these tests, it was used to generate sine sweep signals in the range of 1–32 Hz and single-pulse signals.

3.6.2.2 Testing protocol

Figure 43 provides a schematic of the platform setups for the tests and a table listing the number and types of tests performed. A total of 42 tests was carried out.

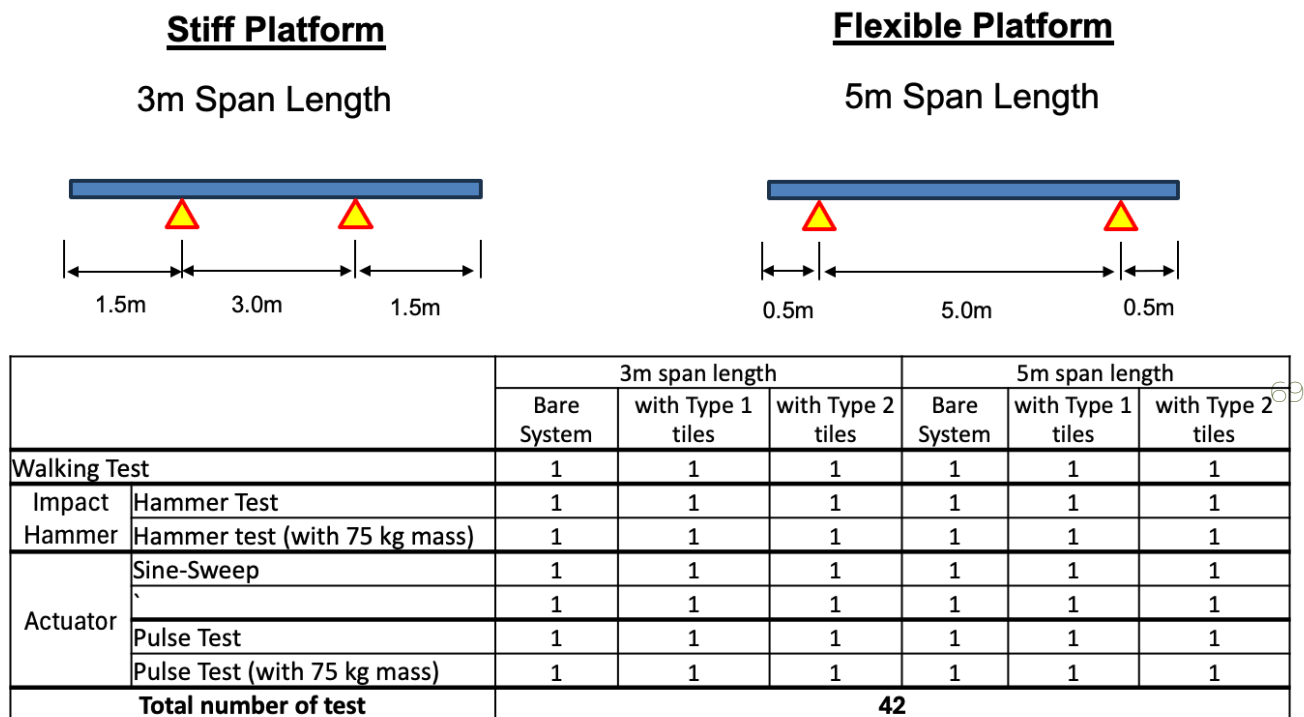
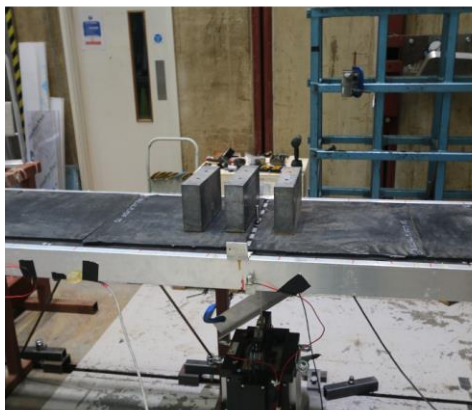


Figure 43. Schematics of the stiff (3m) and flexible (5m) platforms for large-scale vibration tests and a table summarising the measurements performed.

Hammer tests were applied to the bottom of the platform/top of the panels. Data filtered using a 3rd-order band-pass filter with a cutoff frequency of 0.5 Hz-20Hz. A single pulse applied with an actuator and free vibration of the platform were recorded. Data filtered using a 3rd-order band-pass filter with a cutoff frequency of 0.5 Hz-20Hz. Sine-sweep tests were carried out using the electrodynamic shaper at constant force and sweep frequency between 1Hz to 32 Hz. Hammer and pulse tests were also carried out when concentrated masses totalling 75 kg were placed

on the platforms (Figure 44). The weights were simulating the effect of an adult statically placed over the bare platform and on the rubber panels. Walking tests were carried out by male randomized walkers of 75kg average weight (Figure 44). Peak accelerations and peak displacements of the platforms were recorded during each walking cycle (Figure 45). It must be emphasized that the walking tests were not performed following a EDI trial practice in terms of ethics and GDPR data, only as demonstration of technical performance for the platform and rubber panels.

Stiff Platform (with Tiles)
3m Span Length + 75kg weight



Flexible Platform (with Tiles)
5m Span Length + 75kg



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Platform with Tiles + 75 kg Weight

Figure 44. Stiff and flexible platform with rubber tiles and concentrated masses of 75 kg

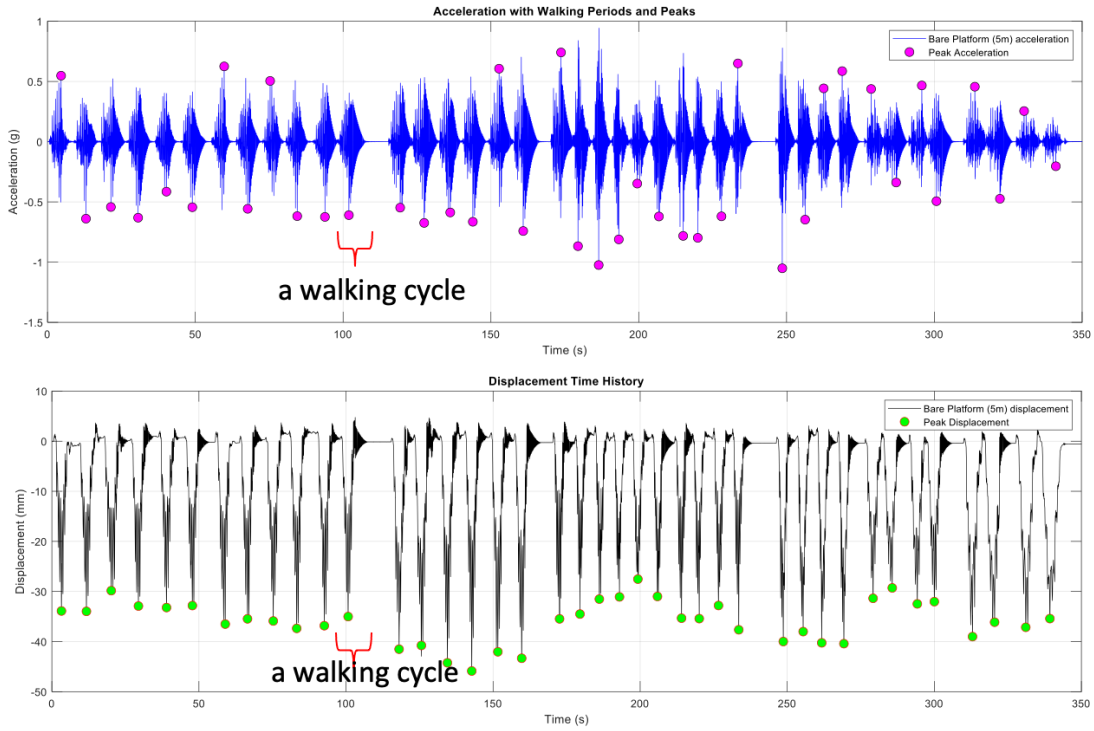
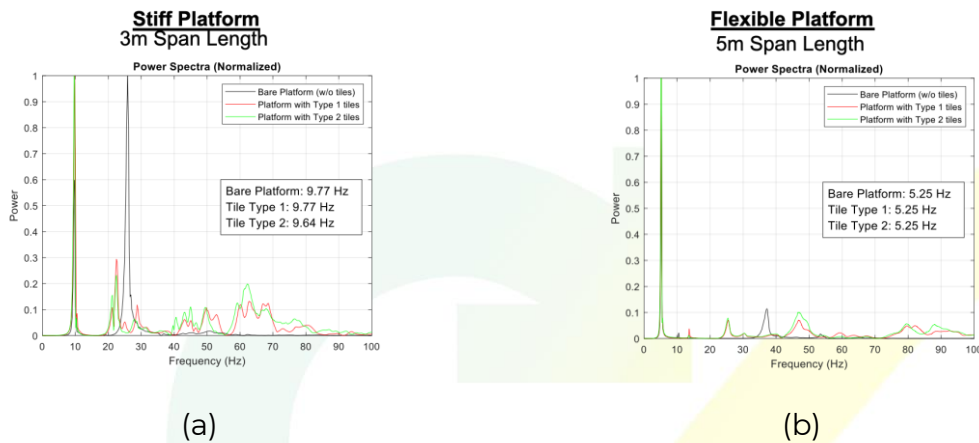
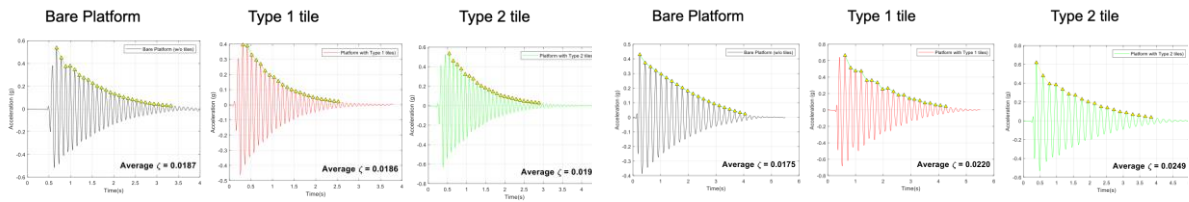


Figure 45. Time histories of walking tests accelerations and displacements

3.6.2.3 Results

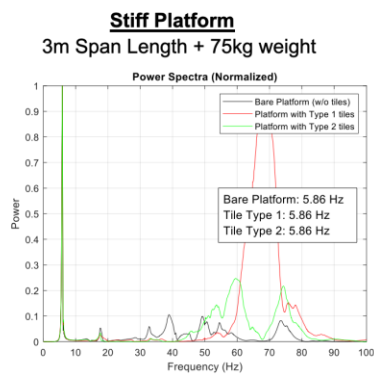
The frequency response functions and summary of results for the impact hammer tests with and without 75kg concentrated masses are shown in Figure 46 and Table 14.



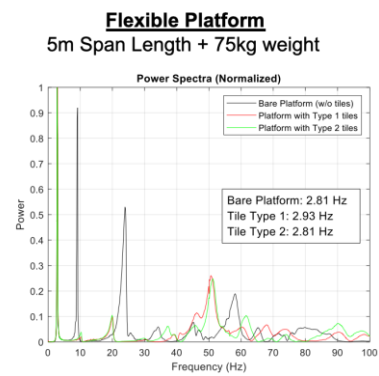


(c)

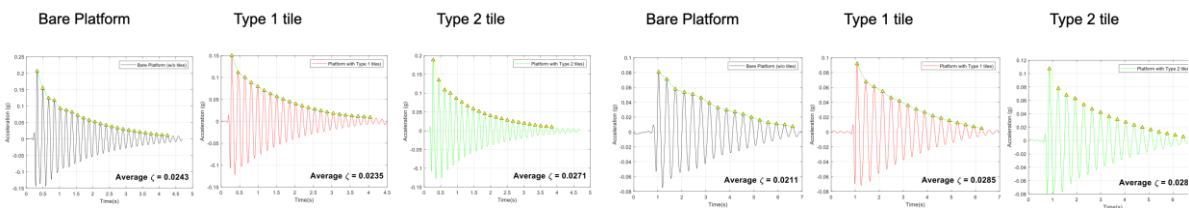
(d)



(e)



(f)



(g)

(h)

Figure 46. Dynamic response of the stiff (a,c) and flexible (b,d) platforms with and without the rubber panels. Similar responses of the systems with 75kg weight are shown in (e, g) and (f,h). Type 1 tile corresponds to rubber panels with 0wt% of lignin; Type 2 tile is the rubber panel with 20wt% lignin.

Table 14: Summary of the hammer tests results

	3m Span Length		5m Span Length	
	Freq. (Hz)	Damping (%)	Freq. (Hz)	Damping (%)
Bare System	9.77	1.87	5.25	1.75
Type 1 Tile	9.77	1.86	5.25	2.20
Type 2 Tile	9.64	1.98	5.25	2.49

↓ Increase in damping

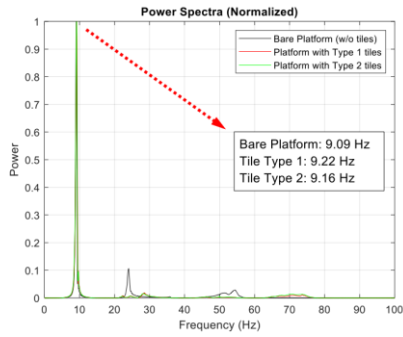
	3m Span Length		5m Span Length	
	Freq. (Hz)	Damping (%)	Freq. (Hz)	Damping (%)
Bare System	5.86	2.43	2.81	2.11
Type 1 Tile	5.86	2.35	2.81	2.85
Type 2 Tile	5.86	2.71	2.81	2.82

↓ Increase in damping

The results show the capability of the platform to tune the frequency range of the system, either by increasing the distance between support spans, or applying the concentrated masses. The presence of the rubber panels does not modify the natural frequencies of the platforms, either in stiff or flexible configurations. There is a notable increase of damping with the rubber panels at 20%wt lignin, although similar levels of damping are shown by the two types of rubber panels when the added weight of 75 kg is considered.

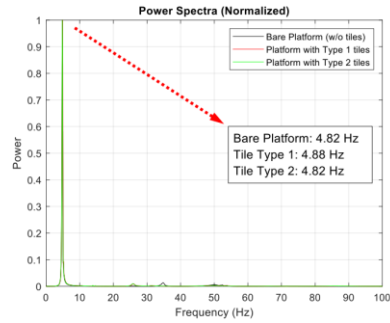
The large-scale pulse tests results with related free vibration analysis are shown in Figure 47 and Table 15.

Stiff Platform 3m Span Length

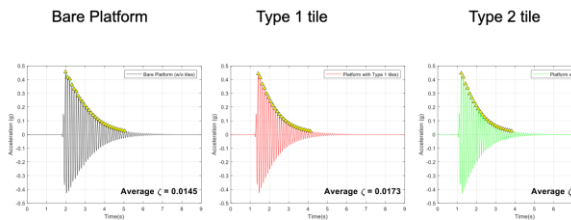


(a)

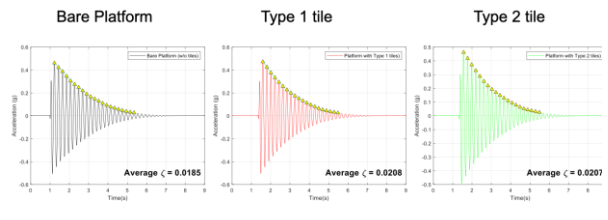
Flexible Platform 5m Span Length



(b)

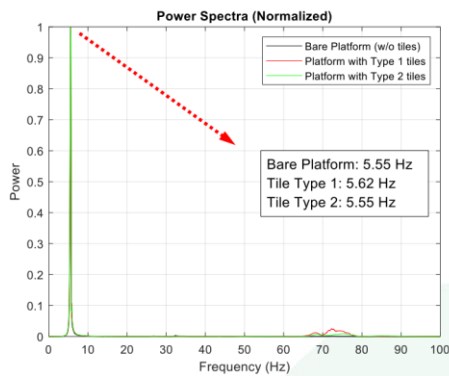


(c)



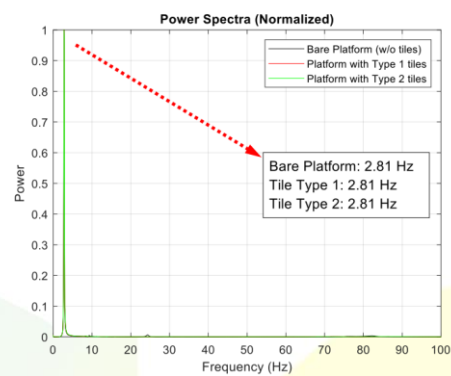
(d)

Stiff Platform 3m Span Length + 75kg weight



(e)

Flexible Platform 5m Span Length + 75kg weight



(f)

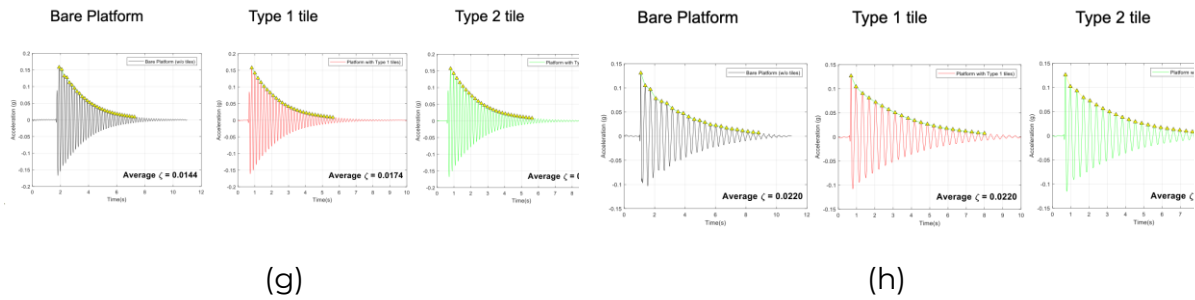


Figure 47. Power response functions and time histories of the response of the platform in stiff, flexible and with added mass configurations.

Table 15: Summary of results for the pulse tests

	3m Span Length		5m Span Length	
	Freq. (Hz)	Damping (%)	Freq. (Hz)	Damping (%)
Bare system	9.09	1.45	4.88	1.85
Type 1 Tile	9.22	1.73	4.88	2.08
Type 2 Tile	9.16	1.78	4.82	2.07

↓ Increase in damping

	3m Span Length (with 75kg)		5m Span Length (with 75kg)	
	Freq. (Hz)	Damping (%)	Freq. (Hz)	Damping (%)
Bare system	5.65	1.44	2.81	2.2
Type 1 Tile	5.62	1.74	2.81	2.2
Type 2 Tile	5.55	1.72	2.81	2.23

↓ Increase in damping

The pulse tests provide similar results to the hammer ones. The presence of the rubber tiles increases the damping of the system, although does not modify the modal response. A very slight increase of damping levels is observed with the 20 wt% lignin rubber panel, only when the 75kg added mass is present.

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The results of the sine-sweep tests at constant force between 1Hz-32Hz are shown in Figure 48 and Table 16.

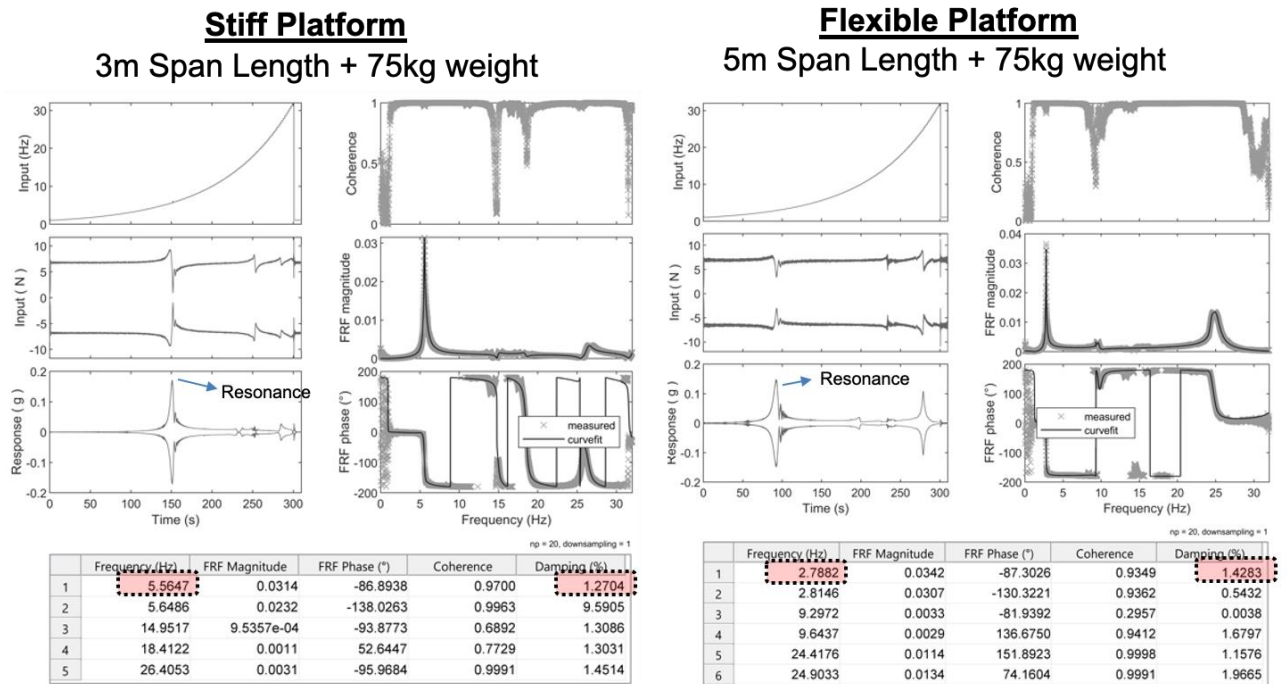


Figure 48. Results of the sine-sweep tests on the stiff and flexible platform

Table 16: Summary of sine-sweep test results.

	3m Span Length		5m Span Length	
	Freq. (Hz)	Damping (%)	Freq. (Hz)	Damping (%)
Bare system	9.45	2.33	5.05	1.66
Type 1 Tile	9.56	2.49	5.09	2.10
Type 2 Tile	9.55	2,89	5.02	2.10

↓ Increase in damping

	3m Span Length (with 75kg)		5m Span Length (with 75kg)	
	Freq. (Hz)	Damping (%)	Freq. (Hz)	Damping (%)
Bare system	5.56	1.27	2.78	1.43
Type 1 Tile	5.57	1.85	2.70	2.27
Type 2 Tile	5.55	1.86	2.71	2.59

↓ **Increase in damping**

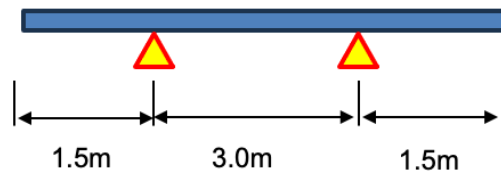
A notable result from the sine-sweep tests is increase of damping provided by the 20wt% lignin rubber panel, when the 75 kg added mass is placed on the flexible platform (81% compared to the bare system and 14% when benchmarked against the 0wt% lignin rubber panel).

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Table 17 and Table 18 provide a summary of the large-scale walking tests carried out during the experimental campaign. The signals have been filtered using a Butterworth bandpass filter between 0.5Hz-20 Hz.

Table 17: Summary of walking test results for the stiff platform.

Stiff Platform (3m Span Length)



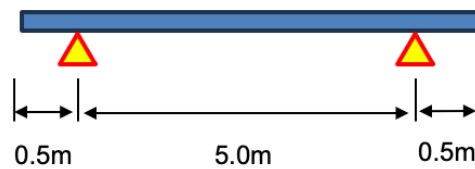
3m Span Length - Accelerations				
	Number of Walking Cycles	Peak Acceleration (g)	Std Dev. (g)	Attenuation (%)
Bare System	54	0.170	0.050	-
Type 1 Tile	61	0.167	0.045	2%
Type 2 Tile	60	0.165	0.053	3%

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3m Span Length - Displacements				
	Number of Walking Cycles	Peak Displacement (mm)	Std Dev. (mm)	Attenuation (%)
Bare System	54	7.035	1.085	-
Type 1 Tile	61	6.988	1.085	1%
Type 2 Tile	60	6.366	1.000	10%

Table 18: Summary of the large-scale waling tests for the flexible platform.

Flexible Platform (5m Span Length)



5m Span Length - Accelerations				
	Number of Walking Cycles	Peak Acceleration (g)	Std Dev. (g)	Attenuation (%)
Bare System	40	0.595	0.176	-
Type 1 Tile	40	0.478	0.148	20%
Type 2 Tile	40	0.440	0.148	26%

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5m Span Length - Displacements				
	Number of Walking Cycles	Peak Displacement (mm)	Std Dev. (mm)	Attenuation (%)
Bare System	40	35.884	4.257	-
Type 1 Tile	40	33.219	4.207	7%
Type 2 Tile	40	32.569	4.471	9%

Walking tests carried out over the stiff platform configuration do not show a strong contribution provided by the rubber panels, except for a 10% attenuation provided by the 20wt% lignin panel in terms of maximum displacements of the platform. Results are however more significant for the flexible platform. In this case, the rubber panels provide a 20% and 26% of peak acceleration attenuation compared to the baseline configuration, and up to 9% of peak displacement attenuation. The 20wt% lignin panel provides the largest contribution to the attenuation phenomena.

3.7 Mechanical properties

Although not intended for a structural use the bio rubber must possess some mechanical strength to endure handling and assembly stresses as well as some stresses and wear resistance inherent to the service conditions. Mechanical properties were determined to evaluate the fitness for service of the bio rubber and comparison with current materials used for the same function. Table 6 presents the different formulations of bio rubber tested.

Table 19: Bio rubber materials tested for the mechanical strength characterization

Compounded bio-rubber	Components			Purpose
	Additives	Natural rubber	Thickness (mm)	
GL-P-022A	0wt% Lignin	20 PHR	3	Final characterization testing
GL-P-017B	10wt% Lignin	20 PHR	3	
GL-P-018A	20wt% Lignin	20 PHR	3	
GL-P-019A	0wt% Lignin	20 PHR	13	
GL-P-020D	10wt% Lignin	20 PHR	10 & 13	
GL-P-021A	20wt% Lignin	20 PHR	13	

3.7.1 Bond strength

Bond strength is not a material property but could be relevant to attach the rubber plates to their support structure in service. Some limited tests were therefore performed in bio rubber with 10 and 20% lignin.

3.7.1.1 Method description

Test were performed according to ISO 813 whereby a strip of rubber is attached with an adhesive to a rigid substrate and the load required to pull it out at an 90° angle is recorded. The tests followed the standard procedure without major deviations. All tests were performed in a standard test jig in an Instron electromechanical test machine under displacement control and using three mm thick specimens.

3.7.1.2 Testing protocol

The specimens were glued to a rigid steel substrate using 3M 847 nitrile adhesive specific for rubber materials, Figure 49.



Figure 49: Specimens of bio rubber used for the adhesion test

3.7.1.3 Results

The tests were performed in an Instron machine with a displacement rate of 50 mm/min, Figure 49. The test results are presented in Figure 50 and Figure 51. Fractures showed a mix of adhesive and cohesive failure at quite low loads even when most failure was cohesive, Figure 52 and Figure 53.

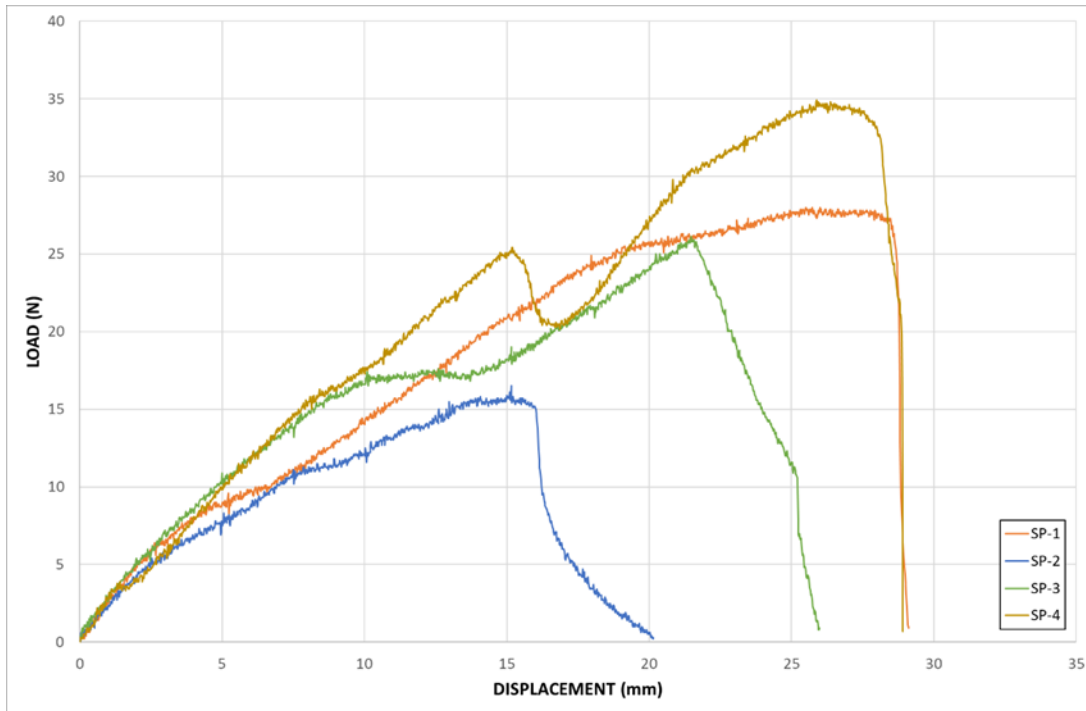


Figure 50: Bond strength test results for GL-P-018B bio rubber.

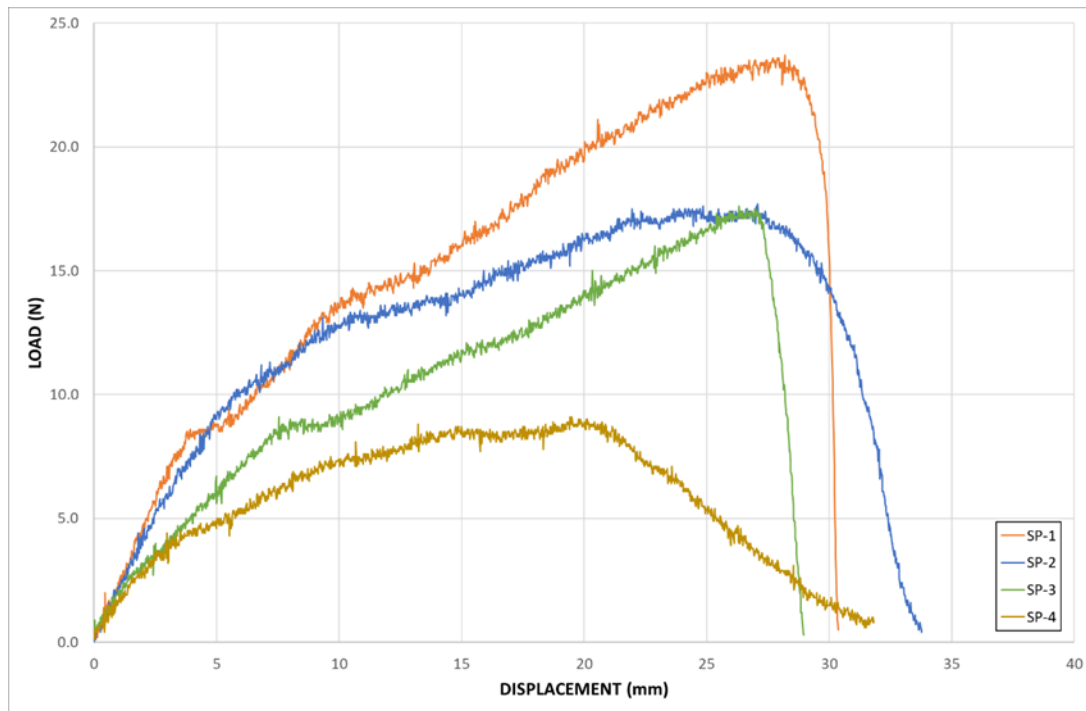


Figure 51: Bond strength test results for GL-P-017A bio rubber.



Figure 52: Failure modes of GL-P-018B bio rubber after adhesion test.



Figure 53: Failure modes of GL-P-017A bio rubber after adhesion test.

3.7.2 Compressive strength

Since the bio rubber could be used in flooring application compression tests were also performed within WP3. Further details can be seen in Deliverable 3.5.



3.7.2.1 Method description

The tests were performed according to ASTM D575 standard without any major deviations. An Instron electromechanical testing machine under displacement control was used for all test

3.7.2.2 Testing protocol

The tests were performed on a special test jig to keep proper alignment of load. Compression was achieved between two hard steel plates up to a total strain of about 30 % and different materials compared at 25% strain.



Figure 54: Compression test setup.

3.7.2.3 Results

Table 17 summarises the results of the compression tests which show a slight increase of compression strength with increasing Lignin content

Table 20: Summary of compression properties of bio rubber.

MATERIAL	STRESS @ 25%	MAX		MODULUS
	(Mpa)	Stress (Mpa)	Strain (%)	(Mpa)
P-019A 0%Lignin	0.347	0.411	27.33	1.157
P-020D 10%Lignin	0.395	0.522	28.64	1.333
P-021A 20%Lignin	0.495	0.652	27.88	1.547

3.7.3 Hardness

Hardness is an important property for wear resistance, thus, also relevant for flooring application and was therefore determined as a function of the lignin content. This characterisation was performed in WP3, and further details can be seen in Deliverable 3.5

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3.7.3.1 Method description

The tests were performed according to ISO 48 standard shore A without major deviations from the standard procedure.

3.7.3.2 Testing protocol

A portable equipment was used for all measurements usually performed on the shoulders of tensile specimens.

3.7.3.3 Results

Table 21 summarises the hardness of the bio rubber tested which show a slight increase of hardness with lignin content.

Table 21: Shore hardness of bio rubber material.

	GL-P-022A 0wt% lignin	GL-P-017B 10wt% lignin	GL-P-018 20wt% lignin
HARDNESS SHORE A 24.7°C RH65%	57	60	66
	58	61	67
	57	59	67
	57	61	66
	58	61	65
	57	61	65
	57	60	67
	58	60	65
	56	60	67
	57	59	66
Average	57.2	60.2	66.1

3.7.4 Tensile resistance

Tensile resistance is an important property for functionality of the product. This characterisation was performed in WP3, and further details can be seen in Deliverable 3.5

3.7.4.1 Method description

Test were performed in an Instron electromechanical test machine under displacement control and according to ASTM D412 standard.

3.7.4.2 Testing protocol

Specimens with dumb bell shape were cut from rubber plates using a special tool. The specimens were removed in two orthogonal directions to check eventual variation of properties, Figure 55. Strain was measured by a side contact long travel mechanical extensometer.



Figure 55: Left to right, bio rubber pad after some specimens extracted, specimen cutting tool and test specimens ready.

3.7.4.3 Results

Table 19 summarises the tensile properties of all bio rubber materials. Unlike compression properties lignin did not seem to have a clear effect upon the tensile

Table 22: Tensile properties summary for all bio rubber materials

TENSILE TEST DATA		STRAIN @ MAX STRESS	MAX STRESS	MODULUS
		(%)		(MPa)
P-017B	AVG	265.1	6.162	1.98
	STD	26	1.358	0.317
P-018A	AVG	213.1	4.643	2.34
	STD	7.3	0.373	0.089
P-022A	AVG	239.7	7.322	1.949
	STD	14	0.845	0.211

4 Technical assessment

In this section summary of testing in terms of methods and results is presented in format, conforming to the principles of European Technical Assessment for construction products, falling in the scope of the Construction product regulation (CPR, 2024/3110). This section as such does not represent a legal assessment. It does, however, give information, helpful in legal procedures.

4.1 Structure and purpose

In this section condensed information on testing is provided, organized by the basic requirements for the construction works, as per 2024/3110 Construction Products Regulation (CPR).

4.2 Proposed clauses and sections

4.2.1 Product description

The product is flat, bio-based rubber board of general thickness 10mm-15mm. It is intended to be used as flooring or wall lining in buildings.

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4.2.2 Essential characteristics

4.2.2.1 BWR1 Structural integrity of construction works

Basic requirement for construction works BWR 1 is not relevant for the product, because the product does not contribute to the structural stability of the construction work (building).

4.2.2.2 Fire safety of construction works

Requirement

Reaction to fire of the product shall be assessed following methods for assessing compliance to the respective class according to EN 13501-1

Assessment and expression of the results

The reaction to fire shall be expressed as class according to EN 13501-1

The achieved class ins class E.



4.2.2.3 Protection against adverse hygiene and health impacts related to construction works

Requirement

The products shall not cause any adverse effects on human health

Assessment and expression of the results

The potential for adverse effects should be checked by analysing the constituents of the product (input materials), emissions during production and emissions of harmful substances during the lifetime.

Emissions were tested using a proxy test, release of substances into solvent solution and analysing the content for potential emissions to air.

No critical substances were found.

4.2.2.4 Safety and accessibility of construction works

Requirement

Various aspects of safety in use shall be explored, including safety of entrapment, physical harm, chemical harm and electrical harm.

Assessment and expression of the results

The product does not represent safety concerns. With respect to electrical safety and possible electrostatic charge build electrical resistance of the material has been measured.

The product exhibits electrical resistance of 4 GW and higher (see the detailed results).

4.2.2.5 Resistance to the passage of sound and acoustic properties of construction works

Requirement

Protection against structural sound transmission shall be assessed.

Assessment and expression of the results

Vibrational tests for damping of vibrations have been employed as a measure, connected to reduction of structural sound transmission.

The damping factor is given in the report.

4.2.2.6 Energy efficiency and thermal performance of construction works

Requirement

Energy efficiency and thermal performance is of secondary importance. Nonetheless it shall be known.

Assessment and expression of the results

Thermal conductivity shall be measured using an appropriate method (guarded hot plate, laser flash, hot wire etc.).

The measured and extrapolated thermal conductivity is between 0.40W/mK and 0.55 W/mK at mean temperature 10°C.

4.2.2.7 Emissions into the outdoor environment of construction works

Requirement

The products shall not cause any adverse effects on the environment and consequently human health

Assessment and expression of the results

The material shall be tested for leaching when in contact with water.

Emissions to water were tested using a proxy test, release of substances into solvent solution and analysing the content for potential emissions to water.

No critical substances were found.

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4.2.2.8 Sustainable use of natural resources of construction works

Requirements

Contribution of the product to climate change shall be assessed

Assessment and expression of the results

The assessment shall be done based on the life-cycle analysis and expressed through global warming potential (GWP)

The assessed GWP is given in the technical section of the report.

4.2.2.9 General aspects of functionality and durability

Requirements

Additional aspects of functionality and durability, that do not contribute directly to the basic requirements for the construction works shall be considered

Assessment and expression of the results

These requirements refer to mechanical properties:

- Bond strength
- Compressive strength
- Tensile strength
- Hardness

Individual values and results are given in the respective clauses of the technical part of the report.

4.2.3 Proposed AVS system

The proposed level of assessment and verification system (AVS) is

	Characteristic	AVS
1	All characteristics except those, listed in line 2	3
2	Contribution to climate change	3+

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- 3 for all characteristics, except the contribution to climate change.
- 3+ for contribution to climate change

In **AVS 3** the tasks for the manufacturer are:

- further assessment of the performance of the product on the basis of testing (including sampling of the items to be taken as representative of the type), type calculation, tabulated values or documentation describing that product;
- factory production control;

- the drawing up of technical documentation containing proof of the correct application of this Regulation with regard to the assessment of performance;
- the drawing up of technical documentation containing proof of conformity with the applicable product requirements under this Regulation.

In **AVS 3+** tasks for the manufacturer are:

- the assessment of the performance of the product on the basis of data collection for input values, assumptions and modelling;
- factory production control.

Note: the AVS recommendation is based on standard practice and depending on effects on BWR 1 and BWR 2, as found in EU regulations CPR 305/2011 and CPR 2024/3110. For details of tasks for the manufacturer please see the cited sources. There may be more parties involved in the AVS (i.e. manufacturer and certification body; tasks for the certification body are not given here).

5 Conclusions

The GREEN LOOP bio-based rubber panels were developed based on a previously established material formulation, as described in the deliverables of WP3. The focus of WP6, however, was on upscaling production to a level suitable for practical use. Potential changes in behaviour from material-level tests to product-scale tests were investigated for fire-related and vibration-related properties, as these require testing on larger samples. Other properties are not considered likely to change with scale-up, which is supported by the nature of the tests conducted on small-size samples.

In order to present the complete picture of material behaviour, some results, obtained within the material optimization and in principle already covered in WP3 reports are given within this report, in particular to support holistic technical assessment as described in section 4.

It is worth noting that, during the course of the project, the testing program was significantly expanded compared to the range specified in the Description of Work. This was made possible by the consortium's excellent composition, combining specialist partners with partners who had access to a wide range of testing methods beyond reaction-to-fire and vibrational characteristics.

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The summary findings on the product evaluation are as follows:

- Addition of lignin of 0%, 10% and 20% of mass has not influenced fire characteristics significantly. In all configurations the achieved reaction to fire class is class E, which allows for the use of material in practice if the material is properly protected against thermal radiation of large thermal power. However, **lignin can play an important role in adhesion properties**, were one to integrate a protective layer to the product, as indicated in the body of the report. Therefore, possible further development should be based on substrate with **20% lignin content**.
- Vibrational properties of the produced board are found to increase vibration damping of the structures through addition of the GREEN-LOOP bio-based rubber tiles. The damping was more significant when the panels were placed over flexible platforms, rather than on stiff supports. Similar as with fire, addition of lignin in variable percentage (0%, 10% and 20%) does not significantly change the vibrational properties but it has some limited effect in terms of improved characteristics with more lignin in the material.



Walking tests reveal that rubber panels with 20% lignin slash peak acceleration by 26% compared to the bare platform, while also **reducing peak deformation**. This highlights **lignin's effectiveness in enhancing rubber panels** for flexible structures under walking loads.

- Other tests and calculations: mechanical tests, acute toxicity tests, release of dangerous substances, global warming potential calculation and thermal conductivity tests have shown that characteristics of the **GREEN-LOOP bio-based boards are comparable to commercially** available products.

Final conclusions can be summarized in a statement that the **GREEN LOOP bio-based boards represent a viable basis for application in construction sector**, especially if additional fire retarding coating (e.g. based on sodium silicate solution) is applied to the exposed surface, be it in factory or on site, where such coating is part of the kit to be used in construction.

References and Resources

- [1] Q. Liu, L. Luo, and L. Zheng, “Lignins: Biosynthesis and Biological Functions in Plants,” *International Journal of Molecular Sciences* 2018, Vol. 19, Page 335, vol. 19, no. 2, p. 335, Jan. 2018, doi: 10.3390/IJMS19020335.
- [2] V. Georgs, H. Piili, J. Gustafsson, and C. Xu, “A critical review on lignin structure, chemistry, and modification towards utilisation in additive manufacturing of lignin-based composites,” *Ind Crops Prod*, vol. 233, p. 121416, Oct. 2025, doi: 10.1016/J.INDCROP.2025.121416.
- [3] J. A. Poveda-Giraldo, J. C. Solarte-Toro, and C. A. Cardona Alzate, “The potential use of lignin as a platform product in biorefineries: A review,” *Renewable and Sustainable Energy Reviews*, vol. 138, p. 110688, Mar. 2021, doi: 10.1016/J.RSER.2020.110688.
- [4] Technical Association of the Pulp & Paper Industry, “Acid-insoluble lignin in wood and pulp, Test Method T 222 om-15,” 2021, Technical Association of the Pulp & Paper Industry.
- [5] J. Sluiter, A. Sluiter, C. Scarlata, D. Templeton, D. Crocker, and B. Hames, “Determination of Structural Carbohydrates and Lignin in Biomass: Laboratory Analytical Procedure (LAP) (Revised August 2012),” 2012.
- [6] X. Meng et al., “Determination of hydroxyl groups in biorefinery resources via quantitative ³¹P NMR spectroscopy,” *Nat Protoc*, vol. 14, no. 9, pp. 2627–2647, Sep. 2019, doi: 10.1038/S41596-019-0191-1.
- [7] E. Jasiukaityte-Grojzdek, M. Kunaver, and C. Crestini, “Lignin structural changes during liquefaction in acidified ethylene glycol,” *Journal of Wood Chemistry and Technology*, vol. 32, no. 4, pp. 342–360, Oct. 2012, doi: 10.1080/02773813.2012.698690.
- [8] “Pinette PE,” <https://pinetteemidecau.eu/en/>.

- [9] International Organization for Standardization, "ISO 11925-2:2020 Reaction to fire tests — Ignitability of products subjected to direct impingement of flame — Part 2: Single-flame source test."
- [10] International Organization for Standardization, "EN 13823:2020: Reaction to fire tests for building products - Building products excluding floorings exposed to the thermal attack by a single burning item."
- [11] International Organization for Standardization, "ISO 9239-1:2025 Reaction to fire tests for floorings Part 1: Determination of the burning behaviour using a radiant heat source."
- [12] International Organization for Standardization, "ISO 5660-1:2015 Reaction-to-fire tests - Heat release, smoke production and mass loss rate - Part 1: Heat release rate (cone calorimeter method) and smoke production rate (dynamic measurement)," 2015.
- [13] International Organization for Standardization, "ISO 19702:2015 - Toxicity testing of fire effluents - Guidance for analysis of gases and vapours in fire effluents using FTIR gas analysis," 2015.
- [14] A. R. Horrocks and D. Price, Eds., *Advances in Fire Retardant Materials*, 1st ed. Woodhead Publishing, 2008.
- [15] H. Perera, L. Lebanov, E. Sanz Rodriguez, A. Taoum, B. Paull, and E. Sivret, "Analytical approaches for sampling and assessing volatile organic compounds emitted from engineered wood products," *Build Environ*, vol. 271, p. 112578, Mar. 2025, doi: 10.1016/J.BUILDENV.2025.112578.
- [16] M. M. Minnich, B. A. Schumacher, and J. H. Zimmerman, "Comparison of Soil VOCs measured by soil gas, heated headspace, and methanol extraction techniques," *Soil Sediment Contam*, vol. 6, no. 2, pp. 187–203, 1997, doi: 10.1080/15320389709383556.
- [17] E. C. D.-G. for R. and Innovation, C. Butz, G. Sedlacek, and C. Heinemeyer, *Generalisation of criteria for floor vibrations for industrial, office, residential and public building and gymnastic halls*. Publications Office, 2006.