GREEN-LOOP

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GREEN-LOOP

Sustainable manufacture systems towards novel bio-based materials

WP3 – Bio-rubber material production

D3.1 – Report with bio-rubber material specification

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Consortium	17 organizations. 15 EU Member States + 2 non-EU state

GREEN-LOOP Consortium Partners

	Partner	Acronym	Country
1	IDENER RESEARCH & DEVELOPMENT	IDE	ES
2	NATIONAL INSTITUTE OF CHEMISTRY	NIC	SI
3	SLOVENIAN NATIONAL BUILDING AND CIVIL E. I.	ZAG	SI
4	FRAUNHOFER GESELLSCHAFT ZUR FOERDERUNG DER ANGEWANDTEN FORSCHUNG E.V	FHF	DE
5	LABRENTA SRL	LBRT	IT
6	MIXCYCLING SRL	MYX	IT
7	NERO SU BIANCO	NSB	IT
8	GERACE MARIA CRISTINA - TERRE DI ZOE’	TDZ	IT
9	IRIS TECHNOLOGY SOLUTIONS, SOCIEDAD LIMITADA	IRIS	ES
10	GLOWNY INSTYTUT GORNICTWA	GIG	PL
11	AACHEN UNIVERISTY: PROCESS CONTROL ENGINEERING / AACHEN UNIVERISTY: INSTITUTE OF SOCIOLOGY	AAU	DE
12	AUSTRIAN STANDARDS INTERNATIONAL	ASI	AT
13	INSTITUTO DE SOLDADURA E QUALIDADE	ISQ	PT
14	AXIA INNOVATION UG	AXIA	DE
15	ASOCIACIÓN DE INVESTIGACIÓN METALÚRGICA DEL NOROESTE	AIMEN	ES
16	NATIONAL COMPOSITE CENTER	NCC	UK
17	UNIVERSITY OF BRISTOL	UBRIS	UK

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Executive Summary

This deliverable titled D3.1 - *Report with bio-rubber material specification* is the result of task T3.1 – *Bio-rubber materials specifications definition* which is scheduled within the GREEN-LOOP project to be executed during the first months [M01-M04].

This document provides an overview of the initial analysis of the bio-rubber value chain final products in terms of providing competitive performance once the GREEN-LOOP products are put on the market. In addition to roofing, the identified applications for the bio-rubber value chain products in the construction industry are consistent with the anticipated uses. The listed uses are:

- Roofing
- Flooring
- Wall-covering (external and internal)

The analysis is based on the following:

- Legal requirements for products for the mentioned, specific intended uses.
- Available technical specifications for these products, in this case EN standards.
- An overview of declared properties by available competitive products on the market.
- Technological solutions for production and expected properties.
- Framework of the GREEN-LOOP project (e.g., TRL to be achieved within the given time frame).

The main objective of this deliverable is to perform the analysis as described above in order to correctly guide the products’ development with respect to their performance towards successful demonstration at TRL 6 and further, not within the scope of the project, towards the market.

Thus, this document presents a quick overview of legal requirements, consequences, i.e., mandatory CE marking for the EU market, derived requirements for final products, requirements for materials and description of future actions in the development of the bio-rubber value chain GREEN-LOOP products.

In practice, roofing materials are also used as exterior wall coverings, and flooring materials are sometimes used as interior wall coverings. Both uses are subject to legal requirements for their use in the market and must comply with EN 14041 or EN 13956, as applicable. The work performed within this value chain must adhere to these two standards, which define the assessment methods and expression of test results.

Initial material properties are defined and listed in the respective tables of this deliverable. Most relevant properties (the reaction to fire and vibrational properties as essential material performance indicators) are addressed and compiled. In addition, properties of the material deemed essential for the final performance of the product are also specified.

Finally, based on the results of the analysis, is recommended to develop future work regarding the material composition and product manufacture in order to reach the target performance of the products.

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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

GWP_{100} – 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

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1. Introduction

The purpose of this document is to define the expected performance and requirements for products used in the building construction industry. These requirements are defined after a thorough review of the legal framework for construction products and a search for information on related products, i.e., rubber-based products for the same intended use as defined in the GREEN-LOOP project: floorings and walls in buildings.

Currently, rubber-based materials are predominantly used for flooring and as roofing membranes. Through screening of the applications, it became clear that there are two types of materials used in four types of applications: roofing material, which is sometimes extended as an external wall covering, and flooring materials which is sometimes extended to the internal wall coverings. The two types of uses – flooring and roofing - are foreseen in the legal system for putting products on the market via CPR (35/2011).

In practice, additional applications have been identified, including band tiles for outdoor applications such as playgrounds, gaskets in windows and doors, elastic core in the expansion joints, base insulation (seismic insulators) cores and bridge bearings. These cases are noted; however, the focus of the bio-rubber value chain is in applications in flooring and walling, now amended with roofing. Most of these uses are very much connected to safety issues, be it in terms of mechanical performance or in terms of general users' safety. Due to the fact that safety in the CPR system requires more rigorous systems of performance verification and simultaneously narrows the field of available characterization / test methods, compared to products with lesser impact on safety, the work in developing the range of applicable test methods is somewhat questionable, since it can be overridden by the EC decisions, via the procedures of the European Technical Assessments as foreseen in the CPR , once the products reach the product development stage TRL 9.

1.1. Current solutions of the materials on the market

Materials, introduced to the market for use as flooring membranes or roofing membranes are very well-established products, widely used in the world and the EU. They belong to the group of elastomeric materials.

Regarding their standard properties, the system of constant performance verification ensures that the products comply to the declared performance. It is, however, up to the building code in the individual member states to set the required levels and lasses for specific uses. Other possible, previously mentioned uses of the bio-rubber value chain products, as described above, are not part of the predefined requirements for construction products in the EU.

After comparing the target performance of bio rubber GREEN-LOOP's products with available declarations and requirements, it becomes clear that the crucial advantage of the bio-rubber GREEN-LOOP's products is in their environmental footprint rather than their technical performance. This also indicates the need for careful formulation of the materials, including the formulation of the production process. The latter should closely consider environmental footprint calculations in order to develop an environmentally clean production process and, consequently, products.

1.2. Need for new material to boost circularity

In the Oekobau.dat [1] data entry for the generic dataset of the environmental performance of rubber floor coverings with foam coating according to EN 1816 the MFR (materials for recycling) is declared as 0 kg Additionally, looking at the GWP_{100} this parameter is stated as 13.34 kg $CO_{2,eq}/m^2$ for the production phase and 11 kg $CO_{2,eq}/m^2$ for the waste processing phase (module C according to EN 15804:2012+A2:2019). This information indicates that not only materials currently on the market are not recycled, but also that waste processing requires a high level of energy consumption. Additionally, the benefits from reuse or recycling are declared as - 5.55 kg $CO_{2, eq}/m^2$ in terms of GWP_{100}, but the dataset also stated this arises from the potential substitution of electricity, and not the material itself.

Similarly, the dataset for rubber roof covering, profiled according to EN 12199 is declared as the MFR (materials for recycling) as 0 kg/kg. Additionally, looking at the GWP_{100} this parameter is stated as 13.22 kg $CO_{2,eq}/m^2$ for the production phase and 1388 kg $CO_{2,eq}/m^2$ for the waste processing phase (module C according to EN 15804:2012+A2:2019). Again, this information indicates that materials on the market are not recycled, but also the waste processing requires a high level of energy consumption. Likewise, the benefits from reuse or recycling are declared as - 7.09 kg $CO_{2,eq}/m^2$ in terms of GWP_{100}, but also in this case the dataset states this arises from the potential substitution of electricity, and not the material itself.

Based on this information there is an opportunity in developing new, recyclable products, made of recycled materials. However, it should be mentioned that recycled rubber-based materials do exist on the market.

Other identified uses are not present in this database; however, it can be concluded that the wall applications share similar values due to the frequent use of the same product. There are no environmental data found for outdoor tiles.

2. Initial workflow (WP3 Lab scale)

This section documents the initial lab scale framework and workflow in the development of bio-based rubber products for the construction industry.

2.1. Framework

The framework consists of several aspects, relevant for the bio-rubber products, such as:

- What are the legal requirements?
- What is the intended use of the products?
- Which test methods are available for assessment of the products?
- Is a certification needed?
- What are the presumptions when assessing product’s fitness for use?

2.1.1. Legal framework for putting construction products on the market

Putting construction products on the marked is regulated via:

- European legislation: the CPR (305/2011)
- National legislations where CPR does not apply

In the case of the CPR, the construction products must be marked with the CE mark, based on a specific harmonized technical specification. These specifications are either European standards for specific products, which have been published on the list (hENs), issued by the European commission or specific European Assessment Documents (EADs), also published by the European commission.

In the absence of an appropriate European harmonized technical specification, national legislation applies. Various approaches to regulation exist across EU member states. Some approaches rely on non-harmonized standards, while others rely on a system of national approvals or something comparable. Additionally, it must be highlighted that the content is also distinct, particularly in sections addressing product installation.

2.1.2. Characteristics by basic work requirements

Relevant characteristics are grouped into seven basic requirements for the construction works. Not all characteristics are relevant for a specific intended use and the final product. These basic requirements are:

1. Mechanical resistance and stability
2. Safety in case of fire
3. Hygiene, health, and the environment
4. Safety and accessibility in use
5. Protection against noise
6. Energy economy and heat retention
7. Sustainable use of natural resources

All the requirements for the performance of construction products are built around this set.

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2.1.3. Test standard and procedures

The following table summarises the test standards and procedures thar regulates the relevant characteristics of the product:

Table 1: List of characteristics and associated test methods to be applied (full list).

Characteristic	Type of characteristic	Method
Reaction to fire – HRR, droplet formation	Chemical	EN 13823, ISO 5660-1
Toxicity of fire gases	Chemical	ASTM E 1678, ISO 19701
Smoke production rate	Chemical	EN 13823
Content of dangerous substances	Chemical	To be selected / developed
Emissions of dangerous substances into indoor air	Chemical	ASTM D814, ASTM D1434
Tensile resistance	Mechanical	ASTM D412, ISO 37:2017
Shear resistance (bond strength)	Mechanical	ASTM D1002
Compression resistance	Mechanical	ASTM D395, ASTM D1414
Hardness	Mechanical	ISO 48 ISO 7619-1, ISO 868, ASTM D1415, ASTM 2240
Electrical resistance	Physical	ISO 1853, ISO 2878, ISO 2951
Abrasion resistance	Mechanical	ASTM D5963
Slip resistance	Physical	EN 16165:2021 (Main standard)
Sound absorption coefficient	Physical	Impedance tube measurements according to BS 4142:2014+A1:2019
Impact sound insulation	Physical	ISO 140-8
Thermal conductivity	Physical	EN 12667 / EN 12664
Thickness	Physical	ASTM D3767
Environmental indicators as per EN 15804	Chemical	EN 15804:2012+A2:2019

2.2. Standardization

To compile a list of final product characteristics, a search of existing standards has been conducted. At the first level, hENs and EADs were analyzed to determine whether there are already available standards that can provide a clear list of properties that bio-based rubber value chain products should have. In addition to hENs and EADs, lists of non-harmonized standards were searched to identify potentially interesting specifications. Finally, a list of test standards extracted from various sources was compiled.

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2.2.1. Harmonized product standards and EADs

In the harmonized field the table of technical specifications is:

Table 2: Identified harmonised technical specifications.

Application	Flooring	Roofing	Wall - indoors	Wall - outdoors
Technical specification	EN 14041	EN 13956	No existing hEN – flooring material is used	No existing hEN – roofing material is used
CE marking	Mandatory in the EU	Mandatory in the EU	n/a	n/a

In addition, at the lower TRL stages there are some expectations for the material properties to be met regarding their resistance to fire and vibration.

2.2.2. Non-harmonized standards

There are several standards that complement the harmonized standards. These standards are:

- EN 1817
- EN 1816
- EN 12199
- ISO 16905
- BS 7976: Parts 1-3

2.3. Data management

The management of this WP’s data will involve several tasks from this and other WPs: task T1.4. “*Inline monitoring and quality controls*”; task T3.3. “*Upgrades and modifications of equipment in manufacturing lines*” and task T7.6. “*Open science and Data management plan*”.

Throughout the duration of the project, IRIS will coordinate the collection of data from the three manufacturing lines onto the ICT (Information and Communication Technology) infrastructure that will be connected to the GREEN-LOOP platform for the purpose of optimizing the entire value chain, as well as the market analyses and identification of replication cases performed in WP7.

Although the activities of sensors installation and data management will be performed by NCC and IRIS until M20, the information gathered by the inline sensors will be processed and analysed for quality control from M10 to M36, contributing to the further development of the ICT platform for WP6's up-scaled processes.

In order to comply with the GDPR, RWTH AACHEN will also publish a Data Management Plan (DMP) that establishes the guidelines for data management (including the data collected, the data processed in the GREEN-LOOP platform, and the results obtained) throughout their entire lifecycle. This information will be published during M06 in deliverable D7.11 "*DMP and open sourcing approach*".

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2.4. Circularity

2.4.1. Material use and environmental footprint

To assure circularity and low environmental footprint of the products it is necessary to:

- Maximize portion of bio—based materials
- Maximize use of recycled materials
- Minimize transport routes
- Minimize pre-processing of waste materials
- Minimize process energy
- Minimize use of fresh water

2.4.2. Waste creation in process of construction

The primary focus should be on designing the product to minimize overordering and waste resulting from cutting and forming. Additionally, the product should avoid potentially hazardous substances so that it can be recycled at the end of its life.

2.4.3. Design for deconstruction

When manufacturing products it is recommended to make sure they can be mechanically fixed to the substrate – bonding should be avoided. If bonding cannot be avoided, it should be done using materials that are easy to remove (mechanically or chemically).

2.5. Modelling

2.5.1. Numerical modelling

Constitutive models for the micromechanics of rubbers are based on hyperelasticity (typically Mooney-Rivlin isotropic or anisotropic materials, and/or Neo-Hookean models)[2], [3]. Those models are commonly represented in major commercial Finite Element software (Abaqus, ANSYS, Patran) and can therefore be used to model parts with simple and complex geometries. Those constitutive models allow to take into account both material and large geometric nonlinearities in finite strains [4]. Predictive methods to assess the acoustic performance of structures with rubber mats are also contained in the formulas of the EN 12354-2 standard.

2.5.2. Tools modelling

3D CAD systems such as Catia V5 with be utilised to model the tools required to press bio-rubber panels for WP3. Other production methods analysis software will be exploited if applicable.

2.6. Manufacture

On the issuing date of this document manufacturing process discussions with consortium partners and relevant industry members are still ongoing. Also, due to the nature of a research project with novel materials the actual production techniques and processes may be subject to future changes. The manufacturing process is expected to be refined further after the lab scale testing in early 2023, however, the manufacturing process can be defined currently as:

1. **Lignin extraction (NIC) to produce lignin flour**, with ultrasound techniques being researched to enhance the extraction process.

2. **Rubber functionalisation (UBRIS) to produce devulcanized rubber.** Lab testing of different techniques will inform the final route, but this could use microwaves (IDENER) or ultrasound (IRIS) for devulcanization.

3. **Extrusion of the rubber, lignin, and further additives (UBRIS, small scale & NCC, large scale) to produce a filament or pellets of homogenised material**. Ultrasound may be applied during the extrusion process to enhance the process.

4. **Pressing of the homogenised material (UBRIS, small scale samples & NCC, bio-rubber panel).**

Along the process, the in-line control developed by IRIS will aim to collect process data in order to optimize key properties of the material to reach the final target performance of the products at TRL 6.

3. Products' properties

3.1. Material level

At the material level, the characteristics are focused on fire and smoke toxicity as well as vibrational characteristics. These characteristics are evaluated at the coupon-level tests, which provide material characterization that influences TRL 6 product characterization.

Table 3. Characteristics of material in the bio-rubber value chain.

Characteristic	Expected (target) value
Reaction to fire - heat release	THR < 800 MJ/m^2
Reaction to fire – critical heat flux	> 20 kW/m^2
Reaction to fire – peak smoke production rate	< 150 m^2/s
Smoke toxicity	Non-toxic
Dynamic stiffness	> 20 MPa and 0.15 loss factor (tanδ) @10 Hz
Porosity	Pore size < 5 um

Note: values are arbitrary at this stage of the product.

3.2. Lignin

The primary role of the lignin in the product formulation is to enhance the reaction to fire performance of the final product. The major features describing Kraft lignin (and thus making it highly potential renewable material for the development of the flame-retardant lignin-rubber composites) are high thermal stability, char-forming capability, and aromatic structure with the numerous reactive hydroxyl groups. Kraft lignin-polymer composites were found to be thermally stable with a potential to increase flame-retardant properties by introducing only high molecular weight lignin (HMW, 5 kDa) without small oxidized lignin fragments responsible for its low thermal stability and its strong combustion [5]. Moreover, Kraft lignin incorporation into the natural rubber compounds improve tensile mechanical properties of the materials, without additional modifications leaving a possibility for the further advancements incorporating the tailored-lignin (grafting functionalities/compounds with excellent flame-retardant properties) [6].

The average molecular weight of the lignin used is 3 – 10 kDa, and considering the HMW-affected properties, lignin-bio-rubbers have a high potential to meet the characteristics listed in Table 3. Specifically, the expected effect of using lignin is significantly delaying the ignition time and increasing of the critical heat flux and almost certainly to reducing smoke toxicity. The magnitude of the effect, however, will be evaluated in next steps of the project.

3.3. Current Materials

WP3 aims to recycle rubber into construction panels. Tables 4 and 5 below show the characteristics, that flooring and roofing panels are expected to have and how they are assessed. The values, stated, are derived from an overall Internet based market survey of competitive products.

Table 4: Characteristics of flooring, as set out in EN 14041, EN 1816 and EN 1817.

Characteristic	Expression of the result	Typical value range
Reaction to fire	Class	Efl-s1 - Bfl-s1
Content of dangerous substances	Not actively added / actively added-threshold	-
Emission of VOCs, except formaldehyde to indoor air	See EN 14041	Content of pentachlorophenol <5 ppm
Emission of formaldehyde to indoor air	Class E1 or E2	E1
Water tightness	Sign of water penetration	No sign of water penetration
Dynamic coefficient of friction	$\mu < 0.30$: y/n	≥ 0.30, pass Slip resistance: DS
Electrical behaviour	Various modes	Static electricity: vertical resistance does not exceed $10^9\,\Omega$ - $10^{10}\,\Omega$ Static electricity propensity: <2 kV
Thermal conductivity	λ (W/mK)	0,17 - 0,24

Table 5: Characteristics of roofing, as set out in EN 13956, Annex ZA.

Characteristic	Expression of the result	Typical value range
External fire performance	Class	F_{roof}
Reaction to fire	Class	E
Water tightness	Threshold value	Pass
Tensile properties	MLV	7-8 N/mm2
Root resistance (only if used as root barrier)	Threshold value	pass
Resistance to static loading (only for covered sheets)	MLV	>20 - >25 kg
Resistance to impact (only for covered sheets)	MLV	200 - 300 mm (A) 1700 - 2000 mm (B)
Tear resistance (only mechanically fastened)	MLV	15-40 N
Joint strength	MLV	40-50 N/50 mm peel 200 N/50 mm shear
Durability (only exposed sheets)	Threshold value	1000h pass
Foldability	MLV	-40°C - -45°C
Dangerous substances	No dangerous substances	None

3.4. Bio-rubber GREEN-LOOP target properties

Bio-rubber value-chains’ products can be used in construction industry in a variety of intended uses in walls or floors. As mentioned before, the identified intended uses are:

- Flooring for use indoors
- Wall lining for use indoors (same material is used, as is used in flooring)
- Roofing
- Wall covering for use outdoors; these are often made as continuous layer extending over the roof

These uses are indicated in Figure 1. Depending on the intended use of a specific product a set of relevant characteristics is assigned, as stated in following sections:

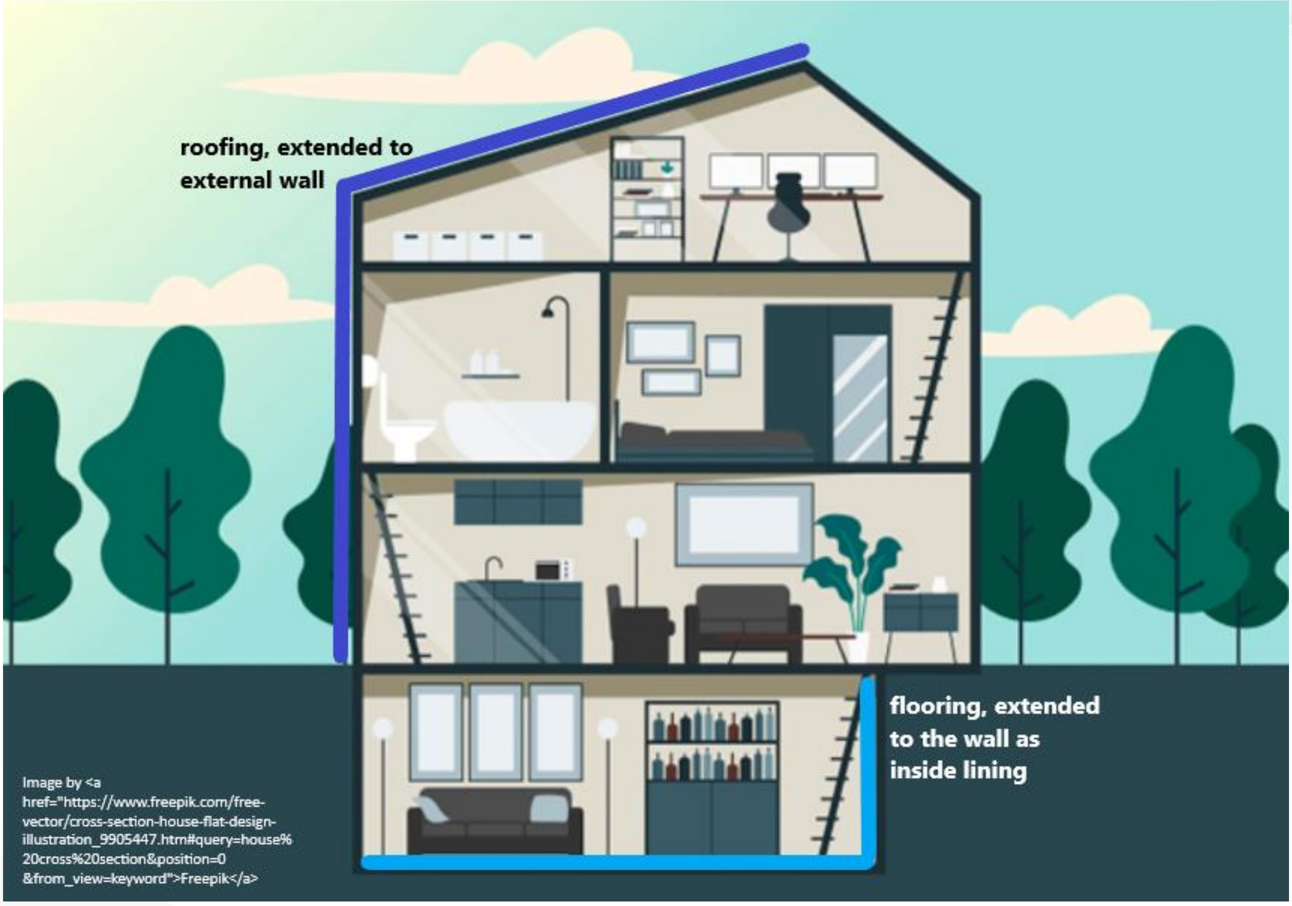

Figure 1. Schematic drawing of the intended installation of the bio-rubber based products.

Table 6 shows the characteristics that should be reached at TRL 6 and gives their value and units. These are the target characteristics for the development of bio-rubber value chain products in frames of the GREEN-LOOP project. These properties and targets are likely to change during the execution of WP3 as the results obtained during the manufacturing process will help in the development and refining of the bio-rubber products.

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Table 6: List of typical characteristics and typical values.

Product	Bio-rubber	
Chemical Properties	**Value**	**Units**
Reaction to fire - class	Class (A1-E)	-
Heat release rate	< 200	kW/m^2
Smoke production rate	< 0,1	m^2/s
Toxic gases in smoke	To be determined	ppm
Content of dangerous substances	Isobutylene, Ethylene	$\mu g/m^3$
Emission of dangerous substances - VOCs (indoor air)	< 1.0	mg/m^3
Environmental indicators, GWP	< 5.9 [7]	kg $CO_{2,eq}$ / kg
Physical Properties	**Value**	**Units**
Thermal conductivity	< 0.3	W/mK
Thickness	(As declared)	mm
Electrical resistance	In the range of 10^9	GΩ
Sound absorption coefficient	< 0.2	-
Impact sound insulation	< 12	dB
Mechanical Properties	**Value**	**Units**
Bond strength (shear)	1.3	MPa
Compressive modulus	2.1	MPa
Hardness	< 82	Shore-A
Tensile strength	3.5	MPa
Standards to address		
EN 14041, EN 13956 (see *Table 2*)		

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4. Future workflow (WP6 Upscaling)

Future work (WP6) will be aiming to upscale the production to an economically viable and sustainable real product. This will be carried out considering characterization, standardization, data management, circularity of bio-rubber products, modelling and manufacture as specified below.

4.1. Characterization

In the Table 1Table 7 a list of relevant characteristics is given. Some of these characteristics (VOC emissions, thermal conductivity, and electrical resistance) will remain to be assessed in at the higher TRL stages (above TRL 6) as they are deemed not to critical for the upscaling or performance. The assumption may change, however during the project.

Table 7: List of characterization methods and their test conditions for the bio-rubber products.

Chemical properties	Method	Test conditions
Reaction to fire - class	Class (A1-E)	As per EN 13501-1
External fire performance	Class (A1-E)$_{ROOF}$	As per EN 13501-5
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 - VOC (indoor air)	Selection to be done, list EN 14041	As appropriate for the method
Environmental indicators, GWP	EN 15804:2012+A2:2019	(calculation)
Chemical 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 6	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
Standards to address		
EN 14041, EN 13956		

4.2. Standardization

Both applications of bio-rubber value chain, i.e., flooring and roofing are standardized through standards for roofing, which should have sabe requirements as external walling, and standards for flooring, however, there are additional properties to be assessed, such as the environmental performance.

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There is no identified need for developing a specific standard for the products of for test methods. At the time there is also no need for modifying any of the test methods. Nonetheless, should such a need occur during the characterization of the product, it will be described and reported in the respective deliverable.

In the case major deviation from the harmonized product standards is identified, another harmonized route (creation of an EAD) should be selected. In this case the deviations should be identified, and an EAD creation requested at the EOTA (European Organization for Technical Assessments). It is recommended to suggest modifications and test methods as well as supply the test results along the route. Once an EAD is created, an ETA can be issued on its basis and the CE marking can be applied. Although the route is fast, it still takes expectedly a few years to pass the whole procedure.

4.3. Data management

Regarding future activities related to task management, IRIS will continue to develop the ICT infrastructure, assist NCC in the selection and installation of sensors connected to it, and upgrade the platform's capabilities to accommodate the scaling up of the final processes. In the meantime, RWTH Aachen will develop and publish a DMP that will serve as the foundation for the management of information processes by the ICT infrastructure and the GREEN-LOOP platform.

4.4. Circularity

This section explains the methods that will be used for sustainability assessment, an important aim for WP6 and GREEN-LOOP.

4.4.1. Life Cycle Assessment

Life Cycle Assessment (LCA) is a method to quantify the environmental impact of a product or service across all, or selected stages of its lifecycle. The stages involved in conducting an LCA are governed by the ISO14040 standard [8] which provides the guidelines and framework for the process. The key stages involved in conducting an LCA are provided in Figure 2.

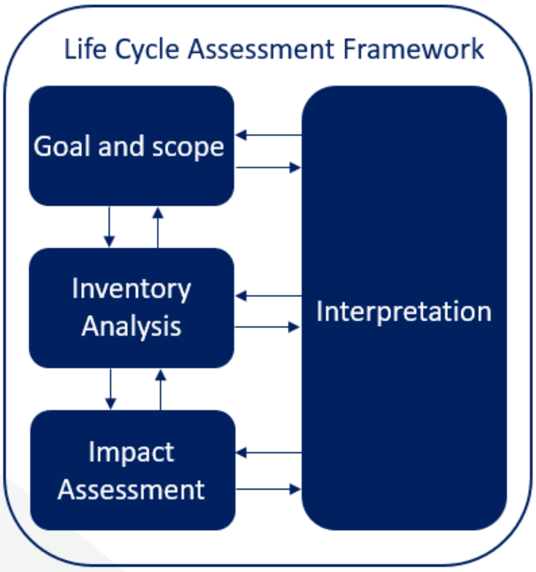

Figure 2. The key stages of LCA as governed by ISO 14040

LCA will be a crucial part in benchmarking the environmental impact of current methods, as well as modelling the proposed methods of bio-rubber manufacture. The key considerations within this value chain include:

Materials:

- Origins, composition, and quantities of lignin
- Origins, composition, and quantities of recycled rubber

Manufacture:

- Energy requirements
- Use of chemicals, other raw materials, and consumables
- Generation of process waste and emissions

Use phase:

- Are there any environmental advantages to using the product over its life?
- Are there any energy savings using the product in comparison to the standard products?
- Are there any improvements to the product’s characteristics (such as durability) in comparison to the standard product?

End of Life:

- Ability to re-use or recycle materials
- Requirements to re-use or recycle materials

The generation of a process maps will inform LCA data capture requirements by highlighting where measurements or data capture activities need to be undertaken. The data collected will be used as an input for the LCA calculations using LCA software (SimaPro/GaBi), and the results will be written up into the LCA assessment of deliverables D2.3 and D2.4 titled “*Energy, exergy, LCA, SLCA and LCC assessments*”. Conducting LCA as part of the GREEN-LOOP project enables more sustainable decisions to be made and will provide benchmark data which can be used to identify process improvements for future iterations of the manufacturing process.

4.4.2. Circular Economy

Although LCA can determine environmental impact, it is not able to indicate the level of circularity of materials or products. A metric to help understand circularity has been developed by the Ellen McArthur Foundation [9]. This metric is known as a Materials Circularity Indicator (MCI). The Circular Economy Model that MCIs are based upon, promotes the creation of a continuous flow of materials around the system, where materials are obtained from re-used, recycled or renewable sources, as well as ensuring there are suitable end of life routes to return materials back into either the technical/engineering loop (Technosphere) or back into the environment (Biosphere). It also considers how intensely a product has been used over its lifetime. This circular economy model is best visualised in the diagram in Figure 3 [10].

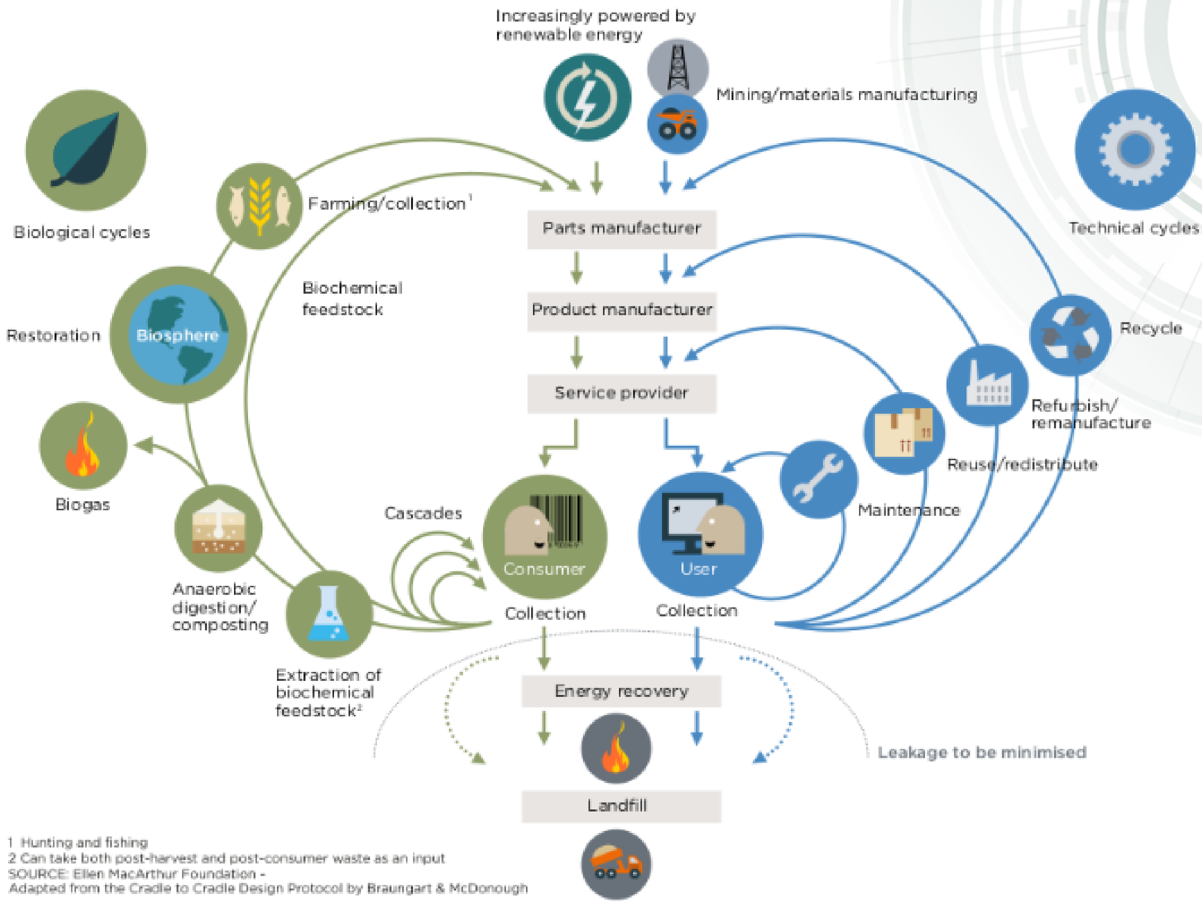

Figure 3. The circular economy model as constructed by the Ellen McArthur Foundation

They key stages of the value chain that are considered within MCI's are:

- **Material source** (proportion of virgin/recycled/re-used renewable material)
- **Utility during use phase** (*How long and intensely is the product used and what is its durability*?)
- **Destination after use** (*What proportion goes to landfill/energy recovery/recycling/reuse?*)
- **Efficiency of recycling** (*How efficiently does the process convert used material to feedstock for new materials?*)

This investigation can be conducted for bio-rubber produced within GREEN-LOOP to understand the level of circularity that can be achieved through material innovation, as well as informing parts of the value chain that may need to be improved upon to increase the circularity of the materials going forwards.

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4.5. Modelling

4.5.1. Numerical modelling

Mooney-Rivlin models consider the hyperelastic behaviour of rubber-type materials under uniaxial, shear and biaxial loading. Incompressible versions of the Mooney-Rivlin models are representative of the tension/compression and planar behaviour of both natural and vulcanised rubber systems [11]. Ad-hoc inverse identification techniques of the Mooney-Rivlin constitutive parameters are available as stand-alone functions in Matlab and Python libraries, or also embedded as modules within commercial finite element code as ABAQUS and ANSYS. These models will be used to benchmark the hyperelastic properties of the materials developed within WP3 against known databased of rubber systems.

The EN 12354-2 standard includes a semi-analytical model to predict the reduction of transmitted impact noise by tiles and carpet/rubber coverings in architectural environments. The impactor tests provide data to extract a sound reduction index according to ISO 717-2, that is then used to extract the normalized impact sound pressure level of the floor. This metrics can be used to benchmark the mats developed within WP3 against available database related to analogous mat floorings commercially available.

4.5.2. Tooling modelling

As previously described, 3D CAD systems such as Catia V5 will be used to model the tools required to press bio-rubber panels. The NCC has a dedicated composite tooling department and tooling modelling for WP3 and will follow specific NCC processes for tooling design.

4.6. Manufacture

The manufacturing process can be split into three sections: lignin extraction (Figure 4), rubber functionalisation (Figure 5) and composite production (Figure 6).

4.6.1. Lignin extraction

Approximately 50 million tons/year of Kraft lignin are produced as a side product during the lignocellulose pulping process in paper industry. Lignin, being the most abundant renewable source of aromatics, has a great potential to replace the petroleum-based chemicals such as toxic halogenated flame retardants in polymers used so far thus supporting the sustainable development of polymers and additives [12], [13].

Lignin is extracted using NaOH and Na_2S aqueous solution at elevated temperature, roughly between 170°C and 175°C. The resulting black liquor is acidified to initiate lignin precipitation, which is filtered and freeze dried with liquid nitrogen. Lignin powder (without pre-treatment, fractionated/modified) will be examined as a flame retardant in bio-rubber composites to determine the optimal material performance.

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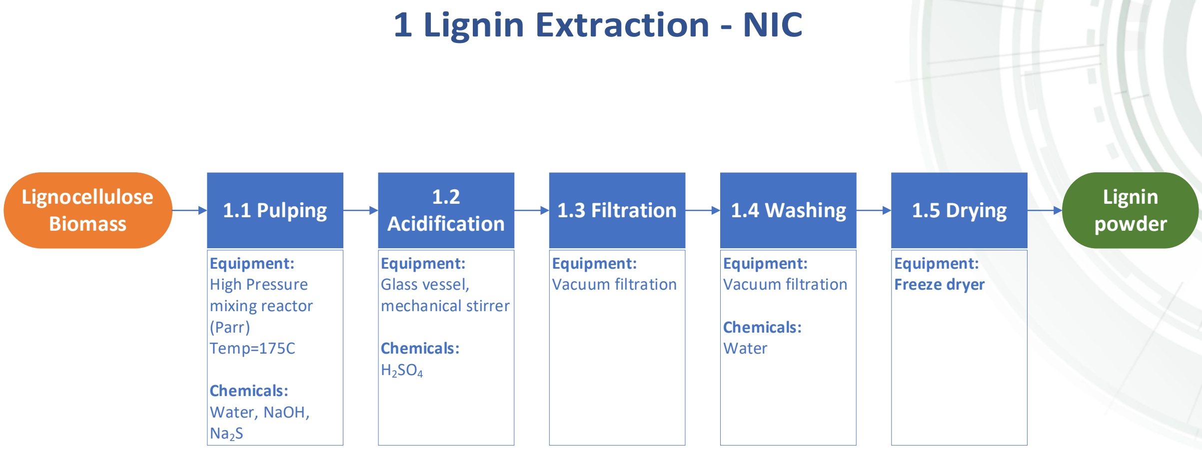

Figure 4: Process flow outlining steps required for lignin extraction

4.6.2. Rubber Functionalization

The rubber feedstock targeted during the project will be from recycled sources, in particular from recycled tire waste. It is estimated that globally 1.5 billion tires reach their end of life each year [14] . This is primarily made up of natural rubber and synthetic rubbers such as styrene butadiene rubber (SBR), butadiene rubber (BR) and butyl rubber (IIR). In order to process recover rubber waste as you would virgin material it is necessary to devulcanize the rubber back to its initial state. There are several ways to do this outlined in the literature such as chemical, ultrasound, microwave, and thermomechanical techniques. Different approaches will be explored in the lab testing phase to determine the best method to use.

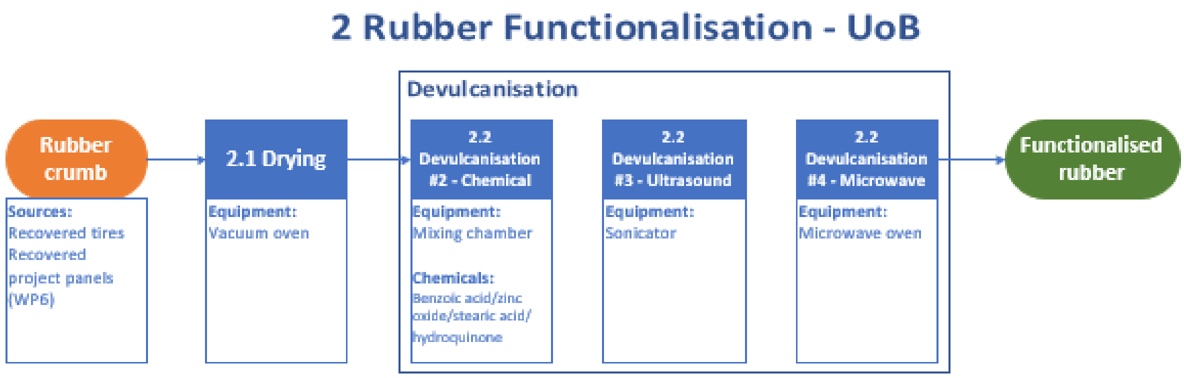

Figure 5: Process flow outlining the steps required to process the recycled rubber crumb

4.6.3. Rubber Composite Production

Once devulcanized, the rubber can be processed in an equivalent manner to virgin rubber feedstocks. It is at this stage that the devulcanized rubber will be compounded with the lignin, and other additives such as micro silica or processing oils may be included to aid material processing or to improve final performance. Initial

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trials will be done by UBRIS using a micro-compounder, and after that, it will then be scaled up using a twin screw extruder to produce enough material for compression moulding of panels.

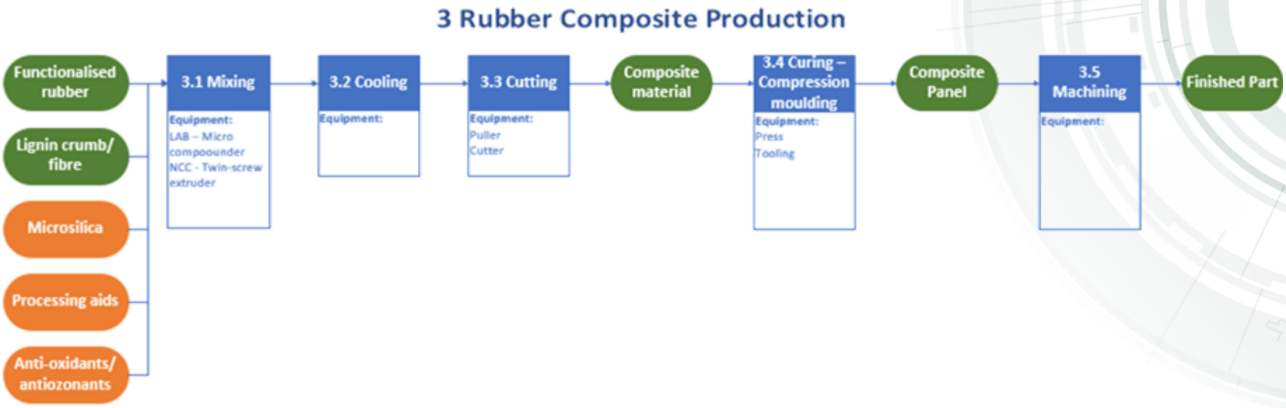

Figure 6: Process flow outlining the steps required to produce the rubber composite

The compression moulding will be done at NCC using a hydraulic press and metallic tooling to produce panels of most likely 400 mm x 400 mm dimensions, with fixing/joining details to be confirmed when NCC has a target industrial panel.

Panels will be pressed at the NCC using a HARE press. A few details about the HARE press:

- Thermoplastic IR lamp preheat shuttle system
- Compression moulding (Thermoset / Thermoplastic)
- Wet pressing (No dispensing Shuttle / Required dosing machine)
- RTM Injection (Required additional Injection machine)
- Data Acquisition (Press positions and TC data)
- Fume Extraction

Some specifications of the HARE press:

- Platen Size = 0.6m x 0.6m (Maximum Tool area = 0.5m x 0.5m)
- Maximum Ram Stroke / day light = 0.5m
- Max Pressing Pressure = 1,000 kN
- Max Closing speed = 300 mm/s
- Max Tool weight = 250 kg
- Maximum Platen Temperature = 400°C / 2°C per min (No Closed loop control)

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Figure 7: The HARE press at the NCC

5. Conclusions

The analysis of the requirements for the bio-rubber value chain products for the construction industry has been completed. It has shown that multiple uses are possible depending on future testing results. An initial broad set of requirements for the products’ properties has been determined. The expected characteristics of the products are listed in the Table 6.

The next step is lab scale production of a bio-rubber panel at UBRIS. Testing this panel should help define a shortlist of real construction panels to focus the project. The actual formulation will be adjusted to meet better defined targets of commercial panels in the market.

Sustainability is an important aim so LCA and energy measurements will be used to compare to commercial panels and GREEN-LOOP's test panels whenever possible. To improve the sustainability of these panels, the raw materials used will be recycled tyres and waste product lignin. Environmental factors such as energy, chemical and water consumption will be considered to minimize the footprint of the products. End-of-life activities is one of the core foci of this project, so an analysis of the final products recyclability will be considered. To this aim, mechanical fixings are preferred for easy dis-assembly, and then the panel will be shredded and considered for use in a second product.

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