BIOBUILD

Thermal Solutions for Green Buildings

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Deliverable D4.5

Report on bio-composite mechanical and physical properties

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Contributors	Paolo Burato, Emanuele Nigrone, Fabio De Francesco, Stefano Maffi (Fellowship) , Antonio Siotto (Master degree student), Giovanni Aminti, Francesco Paloni (Master degree student), Giacomo Mele, Laura Gargiulo, Sabrina Maria Marsala, Sajjad Ghodrati, Marco Cartabia, Sebastian Gritsch
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History of Changes in the Technical Report Part B

Date of change	Section/Page	Nature of change and reason

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Publishable summary

This deliverable reports the results obtained by the physical, mechanical and biological characterization of bio-composites prepared with adhesives of natural origin as linseed oil, lignosulfonate and fungal mycelium. It was characterized three different methodologies, without synthetic glue, for performing bio-composites based on Norway spruce wood fibres impregnated with bio-PCM ethyl ester palmitate.
The biological and physical mechanical characterization were performed on the best combination of three different thesis based on the results obtained by thermal proprieties. Only about SQIM bio-composites, mycelia based on, we received 4 theses, respectively impregnated non pressed (I_NP) and pressed (I_P) and non-impregnated non pressed (NI_NP) and pressed (NP_P). For this reason, due principally to the scaling fabrication with respect to lab scaling of CNR we characterized by all point of view, biological and physical mechanical, only the SQIM bio-composites, nevertheless the evaluation and results obtained in the WP3 from CNR, are resulted di great importance to know the performance of a fungus, found and characterized and used for preparing different bio-composites with hardwood and softwood species impregnate with bio-PCM.
The influence of bio-PCM on these characteristics it is the aim of this deliverable. In fact, the wallboards could be susceptible of biological attacks especially insects due to indoor environment and principally moulds if there are put in service condition the development of humidity. The indoor environment of a building is, in accordance with CEN EN 335, standard use classes 1 and 2. The only biological risk are insect coleoptera and in case of Norway spruce *Hylotrupes bajulus*, subterranean European termites *Reticulitermes lucifugus* and moulds that are developing in the house if the moisture reach high values.
The resistance of the wood-boring beetle *Hylotrupes bajulus* was evaluated in accordance with EN 47:2016. Larvae of category two, weighing 50 to 150 mg, were used, with one larva per sample. Results showed that only the reference specimens and lignosulfonate without PCM reached 12 weeks, while bio-composites with linseed oil, citric acid, and oxalic acid demonstrated resistance to *H. bajulus* after 4 weeks with impregnation and only one week without. Radiographic imaging was conducted using a Gilardoni Radiolight X-ray source, and results were evaluated based on larval survival, gallery formation, and structural degradation. Tests on lignosulfonate adhesives are still pending. The test with mycelia bio-composites is still running and it is waiting for the first check, after 1 month trough RX analyses. The only mycelium thesis test that was stopped before the ending, due the death of all larvae was the I_NP. For the other thesis on mycelium bio-composites the larvae resulted very active after one month since beginning of test and at the moment the tests are ongoing until the first decade of September 2026.
The termites biological test was performed in according to CEN EN 2017 and the results showed that the bio-composites, independently from the adhesive used is not so much suitable for these insects, independently from the presence of bio-PCM, only in some case it was observed some attempts or light attack but not more, such as lignosulfonate without bio-PCM and NI_NP.

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The mould tests concluded showed, for test already concluded at the date of this deliverable, that in most cases, a good resistance o very slight susceptibility to mould but a changing of dimension due to the swollen under effect of high moisture environment with formation of water condensation.

As observed in the physical tests carried out in the laboratory, the impregnation with bio-PCM of the bio-composites fibres leads to a decrease in its mechanical properties and its integrity, also depending on the panel and its composition. The test that decreases more the properties due to the bio-PCM affecting above all is the internal bond test.
However, the data collected permit of defining which types of panels have the greatest affinity with the final use of the project, i.e. interior cladding, which will then be linked with the thermal characterization data. We have been able to see that modified linseed oil is a natural adhesive that is more effective in terms of physical and mechanical characteristics than lignin; in fact, the OX and AC specimens recorded higher values in all the tests in which there was a comparison with the samples with lignin. The AC batch, combining the results of all the tests, is the one with the best characteristics and most similar for the final purpose. The mycelia bio-composites, seems by physical mechanical point of view the solution with the less performance and even in this case it was found a decreasing of an order of magnitude with the presence of bio-PCM with respect to bio-composites without it independently by compression or not.

Introduction

Deliverable 4.5 is a continuation and reveals the properties of the materials processed in Deliverables 4.1-4.4, which demonstrate production of parquet and lignin binder as well as the production of the three types of wallboards namely boards with epoxy linseed oil, lignin and mycelia as binder.
This deliverable describes the biological and physical-mechanical characterization of bio-composites obtained with different adhesives, of plant origine, modified linseed oil, from SLU, lignosulfonate from BOKU and fungal mycelium from SQIM. The characterization has been performed on the best bio-composites obtained, based on thermal proprieties, measured from Lleida University. These tests, carried out on bio-composites performed with and without bio-PCM, gave an information on the influence of bio-PCM on the performance of bio-composites.
The biological characterization is based on evaluation of resistance to coleoptera *H. bajulus* and to subterranean termites *Reticulitermes lucifugus*. Further, the resistance to the moulds was evaluated.
These information about the biological resistance is important especially for the wallboard that in some case, due to the formation of water condensation could facilitate the development of biological decay.
Considering that these bio-composites are in indoor environment, on the walls, they can be attacked preferably by insects and moulds.
The physical-mechanical characteristics were evaluated through various tests: bending three points test in which the elastic modulus (MOE) and the modulus of rupture (MOR) were determined, the tensile test perpendicular to the plane of the panel or internal bonding, with which the cohesion of the panels themselves was evaluated, the hardness test, with which the resistance to penetration was collected and the swelling and water absorption tests were evaluated.
The evaluation of the mechanical and physical properties of a particle board impregnated with phase change materials (PCM) is essential to verify its suitability for use in the application field. In particular, the bending test allows the strength and stiffness of the panel to be determined, highlighting any performance reductions due to the presence of PCM, which can alter the cohesion between the particles. The internal bonding test plays a crucial role as it measures the perpendicular tensile strength and therefore the quality of the internal adhesion: this parameter is particularly sensitive to PCM impregnation, which can interfere with the binder and generate structural discontinuities. The volumetric swelling, on the other hand, makes it possible to evaluate the dimensional stability of the panel in the presence of humidity, a critical aspect considering the possible changes to porosity and hygroscopic interactions introduced by the PCM. Finally, the surface hardness provides indications of resistance to wear and local stresses, which is essential for applications where the panel is

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exposed to direct contact. Overall, the joint analysis of these tests allows to evaluate the balance between the thermal performance provided by the PCM and the preservation of the mechanical and functional characteristics of the material.

1 Biodeterioration and degradation of bio-composites

1.1 Material and methods

1.1.1 Biological resistance against coleoptera *Hylotrupes bajulus*

The resistance against the wood-boring beetle *Hylotrupes bajulus* was evaluated in accordance with EN 47:2016. Larvae, defined in the standard of category two, of weight between 50 and 150 mg, one for sample, were used. In some case, due to the difficulty of extracting the larvae from wood specimens the weight was slightly different but without any influence on the biological test. The reference was performed with larvae put inside Scots pine sapwood while the bio-composites. Two Scots pine sample, due to the small weight of larvae were put in peptone, as source of proteins, impregnated samples as used to do in the living collection.
The larvae were carefully placed into pre-drilled holes prepared in accordance with the standard to facilitate initial penetration. The openings were then sealed with cotton to prevent larval escape while allowing air exchange. The specimens were exposed under controlled laboratory conditions for 12 weeks to promote larval tunnelling and development. Larval survival and development within the wood matrix were monitored non-destructively by X-ray radiography. An intermediate evaluation was performed after 4 weeks to determine early penetration and feeding activity. Final assessment was conducted at the end of the 12-week exposure period. The test results were assessed based on larval survival rate, visible gallery formation, and internal structural degradation detected via X-ray analysis.
At the moment of writing this report the tests of lignosulfonate adhesives and the bio-composites performed with fungal mycelium are still to carry out. The different thesis of fungal mycelia are I_NP (impregnated with bio-PCM and non-pressed), I_P (impregnated with bio-PCM and pressed), NI_NP (non-impregnated with bio-PCM and non-pressed), NI_ P (impregnated with bio-PCM and pressed).

1.1.2 Biological resistance against moulds

The resistance to mould growth and surface discoloration was evaluated under controlled laboratory conditions in accordance with AWPA E24-06. Two mould fungi, commonly associated with wood discoloration and moulds indoor, *Aureobasidium pullulans* and *Sydowia polyspora* were selected as test organisms. The specimens (75 x 100 x 20 mm) were suspended inside the chamber approximately above the inoculated substrate to avoid direct contact. The total duration of the test was 8 weeks or when the 5 rank is reached. Mould growth and discoloration on the sample surfaces were visually assessed after 2, 4, 6, and 8 weeks. Fungal colonization was rated using a 0–5 scale, where 0 represents no visible growth and 5 represents complete (90–100%) surface coverage (Table 1). In figures 1 e 2 the equipment for carrying out the test. The difference from the AWPA standard were principally the utilized moulds, it was used the certified discoloration moulds due the fact that it wasn't possible fungi of grade 4 with respect to Italian legislation.

Table 1 Ranking of Mould resistance test

Ranking	Description
0	No visible growth
1	Mold covering up to 10% of the surfaces it provides growth is not so intense or coloured that it obscures the colour of the sample on more than 5% of the surfaces

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2	Mold coverage of between 10% and 30% of the surfaces that produces growth is not so intense or coloured that it obscures the colour of the sample on more than 10% of the surfaces
3	Mold covering between 30% and 70% of the surfaces it produces growth is not so intense or coloured that it obscures the colour of the sample on more than 30% of the surfaces
4	Mold on more than 70% of the surfaces that produces growth is not so intense or coloured that it obscures the colour of the sample on more than 70% of the surfaces
5	Mold on 100% of surfaces or with less than 100% coverage and with intense or coloured growth obscuring more than 70% of the sample colour

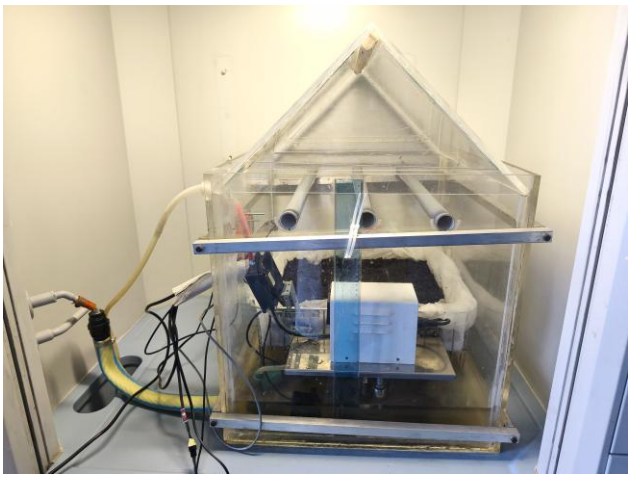

Figure 1 Equipment for evaluation of mould resistance.

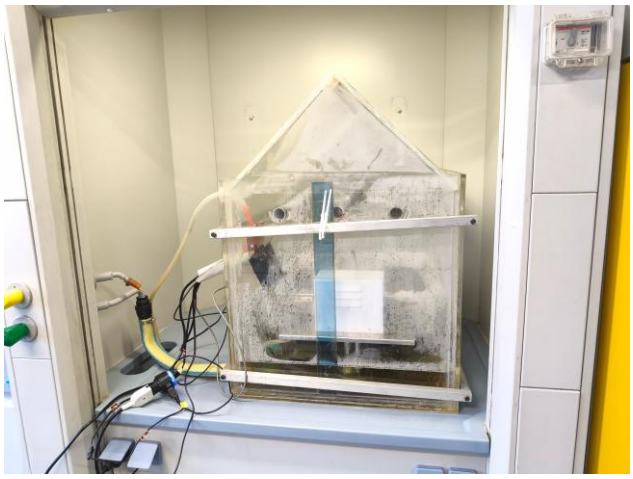

Figure 2 Equipment that is working with the water condensation inside.

1.1.3 Biological resistance against *Reticulitermes lucifugus*

The resistance of bio-composites against termite's attack was evaluated in accordance with EN 117 with a modification. A difference was that only during the first samples the number of termites, 250 workers and 3 soldiers was counted and weighted. Then, successively the workers were weighted and three soldiers added for each sample. The colony of termites collected in University of Florence, in the timber deposit garden, at the moment of the test were of workers and soldier and very few nymphs so it was weighted the corresponded of 250 workers and 3 soldiers for each sample, that corresponded to 1,11 g. The test was performed using the subterranean termite species *Reticulitermes lucifugus*, the Italian subterranean species. The colony was collected at the timber deposit of University of Florence under a beech beam placed on the ground. For each test sample the small colony was introduced into individual glass containers prepared with a moist substrate of sand of pure quartz, to provide suitable living conditions. A ring of glass was buried in the sand and over it a sample was placed. The test is maintained for 8-week exposure period at 70 ± 5% and 20 ± 2 °C. The survival of termites, visual damage ratings observed in test specimens compared to untreated sapwood samples controls were interpreted as indicators of improved resistance to termite attack. Visual assessment was conducted using a standardized rating scale ranging from 0 (no attack) to 4 (severe structural damage) as described in the standard.

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1.2 Results

1.2.1 Biological resistance against coleoptera *Hylotrupes bajulus*

The results are shown in Table 2. In these tests the only reference specimens can arrive to 12 weeks, the biocomposites with the linseed oil with citric acid and oxalic acid as catalyst were results ad resistant to the *H. bajulus* after 4 weeks in case of EP or only a week in case of no EP.
Radiographic imaging was carried out using a Gilardoni Radiolight X-ray source (Italy) operated at 40 kV and 3.0 mA with an exposure time of 2 min for bio-composites and 28 kV 3 mA with an exposure time of 2 min for untreated controls. Images were captured on phosphorus-active plates and digitized using an NDT HD CR 35 scanner (DÜRR, Bietigheim-Bissingen, Germany). The test regarding the mycelium biocomposites, NI_NP, NI_P, I_NP, I_P are at moment of wrting this deliverable attacked and tunneld by living larve and the test will finish on 11th September 2026.

Table 2 Results of biological resistance of bio-composites with linseed oil binder against H. bajulus.

AC	**larva category 2 weight (mg)**	**survival at 1 week (%)**	
6/b	90	no	
7	100	no	
8	22	no	
9	50	no	
10	61	no	
11	145	no	
AC EP	**larva category 2 weight (mg)**	**survival at 1 week (%)**	**RX control at 4 weeks**
1	90	yes	no
2	78	yes	no
3	79	yes	no
4	38	yes	no
5	79	yes	no
6	27	yes	no
OX EP	**larva category 2 weight (mg)**	**survival at 1 week (%)**	**RX control at 4 weeks**
19	50	yes	no
20	130	no	no
21	36	no	no
22	49	no	no
24	43	no	no
OX	**larva category 2 weight (mg)**	**survival at 1 week (%)**	**RX control at 4 weeks**

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13	49	yes	no
14	39	yes	no
15	38	yes	no
16	65	yes	no
17	71	no	no
18	20	yes	no
Pinus sylvestris	**larva category 2 weight (mg)**	**RX control at 4 weeks**	**RX a 12 weeks**
1	64	no	no
2	94	yes	yes
3	101	yes	yes
4	103	yes	yes
5	82	yes	yes
6	56	yes	yes
7	47	yes	yes
8	74	yes	yes
Control peptone	**larva category 2 weight (mg)**	**RX control at 4 weeks**	**RX a 12 weeks**
c1	22	yes	Running tests
c2	13	yes	Running tests
L		**RX control at 4 weeks**	Running tests
1	50	no	Running tests
2	60	yes	Running tests
4	80	yes	Running tests
4	130	yes	Running tests
5	80	yes	Running tests
L EP		**RX control at 4 weeks**	**RX a 12 weeks**
1	60	yes	Running tests
2	65	yes	Running tests
3	100	yes	Running tests
4	100	yes	Running tests
5	70	yes	Running tests
Mycelium I_NP	**larva category 2 weight (mg)**	**RX control at 4 weeks**	**RX a 12 weeks**
1	167	yes	Running tests
2	161	yes	Running tests
3	131	yes	Running tests
Mycelium I_P	**larva category 2 weight (mg)**	**RX control at 4 weeks**	**RX a 12 weeks**
1	100	yes	Running tests
2	140	yes	Running tests

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3	70	yes	Running tests
Mycelium NI_NP	**larva category 2 weight (mg)**	**control at 4 weeks**	**RX a 12 weeks**
1	70	yes	Running tests
2	140	yes	Running tests
3	70	yes	Running tests
4	78	yes	Running tests
5	159	yes	Running tests
6	78	yes	Running tests
Mycelim NI_P	**larva category 2 weight (mg)**	**RX control at 4 weeks**	**RX a 12 weeks**
1	151	yes	Running tests
2	85	yes	Running tests
3	65	yes	Running tests

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Figure 3 Gilardoni Radiolight X-ray source

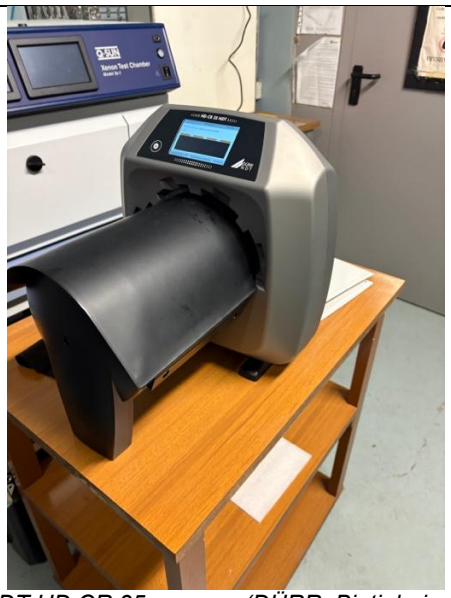

Figure 4 NDT HD CR 35 scanner (DÜRR, Bietigheim-Bissingen, Germany).

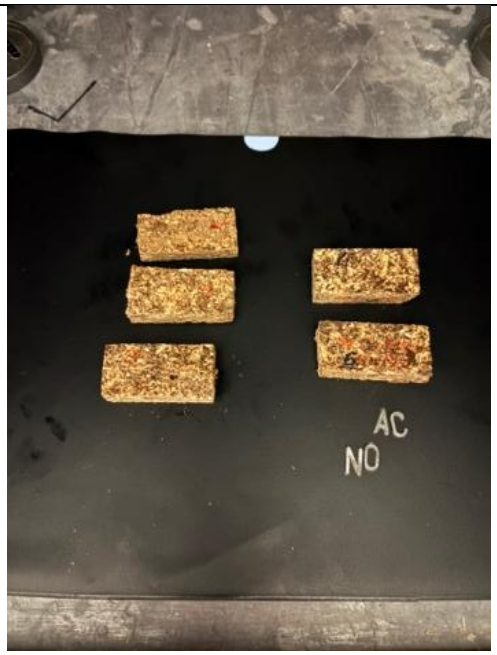

Figure 5 AC without PCM on phosphorus-active plates

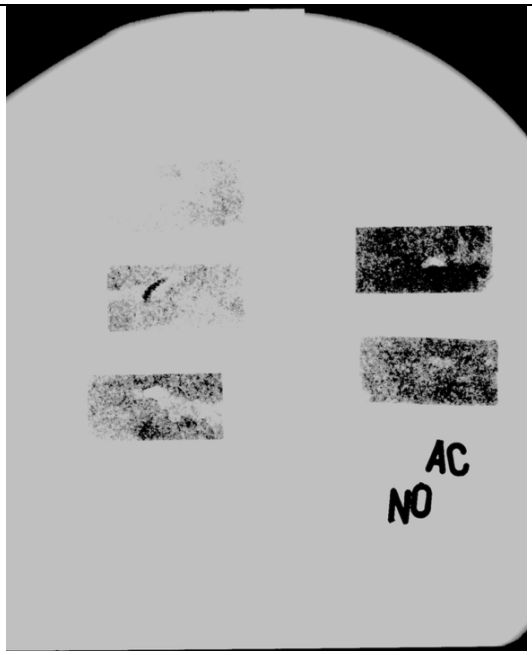

Figure 6 RX of AC without EP

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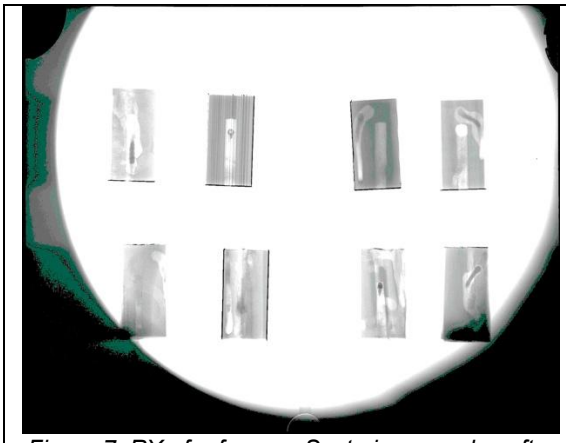

Figure 7. RX of reference Scot pine samples after 4 weeks

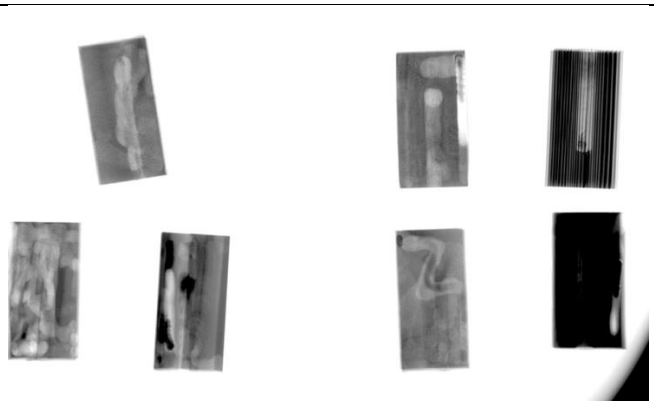

Figure 8 RX of reference Scots pine after 4 weeks.

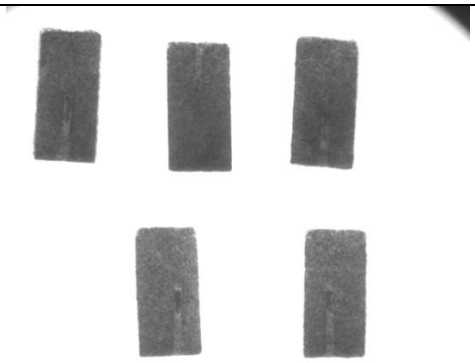

Figure 9 Lignosulfonate bio-composites without PCM after 4 weeks

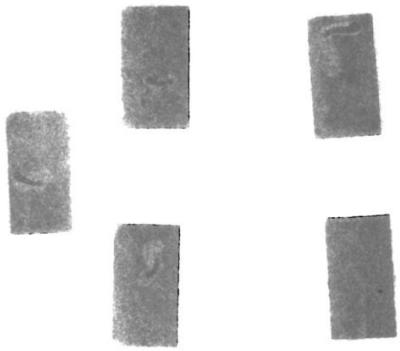

Figure 10 Lignosulfonate bio-composites with PCM after 4 weeks

Figure 11 Mycelium bio-composites I_NP

Figure 12 Mycelium bio-composites NI_P

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Figure 13 Mycelium bio-composites NI_NP

Figure 14 Reference control in sapwood Scots pine.

1.2.2 Biological resistance against moulds

The results are in Table 3, the controls reached the 4 ranking since the second examination, instead in case of linseed oil adhesive and citric acid bio-composites the presence of EP determined a fast rupture of specimens due to the swelling and low cohesion between the wood fibres. Instead, was observed an opposite answer of oxalic acid and EP with respect to only oxalic acid that was broken before the end of the test. The mycelium bio-composites that was examined, respectively NI_NP and I_NP was attacked severely by mould since the second examination.

Table 3 Results of moulds resistance tests

Scots pine controls	1st examination	2nd examination	3rd examination	4rt examination
1	2	4	4	4
2	2	4	4	4
3	2	4	4	4
4	2	4	4	4
5	2	4	4	4
6	2	4	4	4
AC EP				
25	0	0	Broken	- -
26	0	0	Broken	-
27	0	0	Broken	-
28	0	0	Broken	-
29	0	0	Broken	-
Median value	0	0		
AC				
19	0	1	1	1
20	0	1	1	2
21	0	1	1	1
22	0	1	1	1

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23	0	1	1	1
24	0	1	1	1
Median value	0	1	1	1
OX				
7	0	0	0	Broken
8	0	0	0	Broken
9	0	0	0	Broken
10	0	0	0	Broken
11	0	0	0	Broken
12	0	0	0	Broken
Median value	0	0	0	
OX EP				
13	0	0	0	0
14	0	0	0	0
15	0	0	0	0
16	0	0	0	0
17	0	0	0	0
18	0	0	0	0
Median value	0	0	0	0
LIgnin EP	0	1	2	4
L1	0	1	2	4
L2	0	1	1	2
L3	0	1	1	2
L4	0	1	1	2
L5	0	1	1	2
L6	0	1	2	4
Median value	0	1	1	2
Lignin				
l7	0	1	Running test	Running test
l8	0	0	Running test	Running test
l9	0	0	Running test	Running test
l10	0	0	Running test	Running test
l11	0	1	Running test	Running test
Median value	0	0		
NI_P				
1	-	4		
2	-	4		
3	-	4		

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4	-	4		
5	-	4		
6	-	4		
Median value		4		
I_NP				
1	-	4		
2	-	2		
3	-	4		
4	-	2		
5	-	2		
6	-	3		
Median value		2,5		

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Figure 15 Vsualization of mould test from the top of the box without the pitch roof.

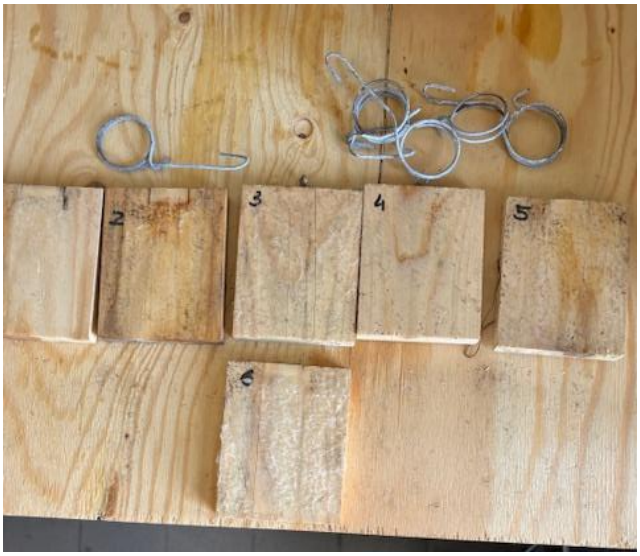

Figure 16 Control sapwood Scots pine at fourth evaluation

Figure 17 Ox specimens at the end of the test

Figure 18 AC with PCM at the end of the test

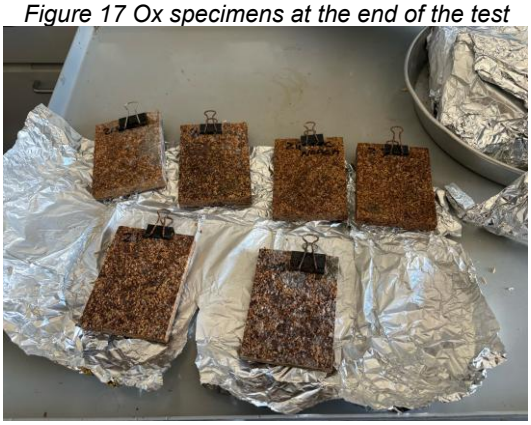

Figure 19 AC at the of the test

Figure 20 Lignin at the end of the test, the specimens are swollen

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1.2.3 Biological resistance against *Reticulitermes lucifugus*

The results are in table 4, all the tests with mycelia bio-composites are still to start, only the NI_NP are finished. In the bio-composites based on linseed oils, with wood fibres impregnated with EP or not the ranking reached only in some cases the level 1, probably due to the difficulty of termites of penetrating them. The lignosulfonate biocomposites resulted , in some case, slightly attacked by termites especially the specimens without EP.

Table 4 Results of EN 117

OX	number survival worker	survival soldiers	% survival	Ranking	Notes
1	0	0	0	0	swollen
2	0	0	0	1	moulds
3	0	0	0	1	swollen
4	25	1	10	0	swollen
5	0	0	0	0	swollen
6	0	0	0	0	swollen
OX EP	**number survival worker**	**survival soldiers**	**% survival**	**Ranking**	**Notes**
1	0	0	0	0	moulds
2	99	2	40	0	moulds
3	7	0	2,8	0	moulds
4	0	0	0	0	moulds
5	0	0	0	0	moulds
L	**number survival worker**	**survival soldiers**	**% survival**	**Ranking**	**Notes**
1	16	0	0	1	swollen
2	42	2	17	1	swollen
3	21	0	8	1	swollen
4	60	1	24	1	swollen
5	69	1	27,6	2	swollen
L EP	**number survival worker**	**survival soldiers**	**% survival**	**Ranking**	**Notes**
1	0	0	0	0	swollen, moulds
2	3	0	0	0	swollen, moulds

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3	0	0	0	0	swollen, moulds
4	23	1	9,2	1	swollen, moulds
5	7	0	0	0	swollen, moulds
AC	**number survival worker**	**survival soldiers**	**% survival**	**Ranking**	**Notes**
1	0	0	0	0	moulds
2	0	0	0	0	moulds
3	0	0	0	0	moulds
4	0	0	0	0	moulds
5	0	1	0	0	moulds
6	0	0	0	0	moulds
AC EP	**number survival worker**	**survival soldiers**	**% survival**	**Ranking**	**Notes**
1	21	0	8	0	swollen, moulds
2	0	0	0	0	swollewn, moulds
3	0	0	0	0	swollen, moulds
4	0	0	0	0	swollen, moulds
5	0	0	0	0	swollen, moulds
6	0	0	0	0	swollen, moulds
Untreaed sapwood	**number survival worker**	**survival soldiers**	**% survival**	**Ranking**	**Notes**
1	32	1	12,8	1	Moulds
2	0	0	0	1	Moulds
3	0	0	0	3	Moulds
4	0	0	0	3	Moulds
5	0	0	0	2	Moulds
6	0	0	0	0	Moulds
Micelium NI_NP					
1	0	0	0	1	Moulds
2	13	0	5,2	1	Moulds

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3	0	0	0	2	Moulds
4	0	0	0	0	Moulds
5	19	1	7,6	1	Moulds
Micelium NI_P	**number survival worker**	**survival soldiers**	**% survival**	**Ranking**	**Notes**
1					Running test
2					Running test
3					Running test
4					Running test
5					Running test
Micelium I_NP					
1					Running test
2					Running test
3					Running test
4					Running test
5					Running test
Micelium I_P					
1					Running test
2					Running test
3					Running test
4					Running test
5					Running test

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Figure 21 Running test

Figure 22 AC PCM specimens at the of the test, completely destroyed.

Figure 23 AC PCM completely swollen at the end of the test

Figure 24 AC no PCM with moulds at the end of the test

Figure 25 Lignosulfonate bio-composites with PCM, on right, and without PCM at the end of the test

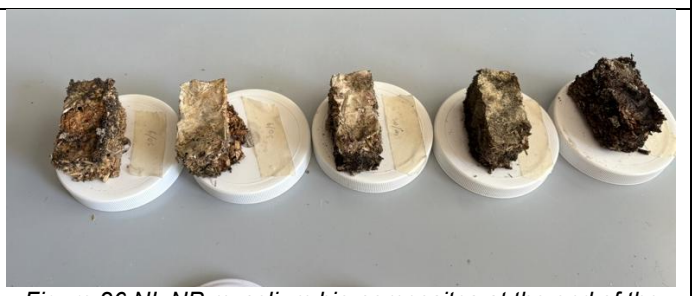

Figure 26 NI_NP mycelium bio-composites at the end of the test. Moulds on the surface

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1.2.4 References

CEN EN 335:2013 Durability of wood and wood-based products - Use classes: definitions, application to solid wood and wood-based products.
CEN EN 47: 2017 Wood preservatives - Determination of the toxic values against larvae of Hylotrupes bajulus (Linnaeus) - (Laboratory method).
CEN EN 117:2023 Wood preservatives - Determination of toxic values against Reticulitermes species (European termites) (Laboratory method).
AWPA E24-06 – Standard Method of Evaluating the Resistance of Wood Product Surfaces to Mold Growth.

2 Physical-mechanical characterization of bio-composites

2.1 Materials

Samples of wood bio-composites made of Spruce particles (*Picea abies*), based on two natural binders: epoxidized linseed oil and catalyst respectively with citric acid (AC) or oxalic acid (OX) as catalyst, and on lignosulfonates were tested.
The specimens tested, if with PCM, were made up of 45% fibres impregnated with palmitic acid ester and the rest untreated fibres to allow an evaluation of the influence that PCM has on the physical behaviour of the materials.
The evaluated samples were divided into lots and named with an acronym and a progressive number that allowed their recognition.
The initials and composition of the specimens in each batch is made clear in the following Table:

Table 5 Bio-composites used in the physical-mechanical tests

Sample acronym	Composition
OX-(n)	Pine particles board linseed oil based with oxalic acid
OX-(n)P	Pine particles board linseed oil based with oxalic acid, impregnated with EP
AC-(n)	Pine particles board linseed oil based with citric acid
AC-(n)-P	Pine particles board linseed oil based with citric acid impregnated with EP
L-(n)_10C	Pine particles board lignin based (10 cm thick)
L-(n)_10P	Pine particles board lignin based (10 cm thick) impregnated with EP
L-(n)_15C	Pine particles board lignin based (15 cm thick)
L-(n)_15P	Pine particles board lignin based (15 cm thick) impregnated with EP
L-(n)	Pine particles board lignin based

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L-(n)P	Pine particles board lignin based impregnated with EP
I_P	Particleboard mycelia-based impregnated with EP and pressed
NI_P	Particleboard mycelia-based pressed
NI_NP	Particleboard mycelia-based non-pressed
I_NP	Particleboard mycelia-based impregnated with EP and pressed

UNI CEN/TS 16368:2014 Light particle board – Specifications gives the requirement for particles boards. In a note on the scope of CEN/TS 16368 it is clearly stated that typical applications for lightweight panels are in furniture and non-structural applications such as doors and packaging. Read in other words, it could mean that the industrial world for the near future is seeking, and/or would like, to create lighter furniture and packaging than the current ones. This challenge is certainly important also because, often, the concept of "lightness" contrasts with that of "robustness" and an example can be had by comparing the requirements of EN 312 for traditional particle boards with those of CEN/TS 16368.
In Table 6 the requirements from UNI CEN/TS 16368:2014, in Table 7 Requirements from EN 312.

Table 6 Requirements for general purpose LP2 lightweight panels (including furniture) in dry environments.

Properties	**Standard**	**UNITS**	**Requirements**					
				Thickness (mm)				
			> 6 a 13	**> 13 a 20**	**> 20 a 25**	**> 25 a 32**	**> 32 a 40**	**> 40**
Three points bending MOR	EN 310	N/mm²	8,0	7,0	6,0	5,0	4,5	4,0
Three points bending MOE	EN 310	N/mm²	1.000	950	900	850	750	650
Internal bonding	EN 319	N/mm²	0,35	0,30	0,25	0,20	0,17	0,17

Table 7 Requirements from CEN EN 312:2010

Properties	**Standard**	**UNITS**	**Requisito**								
					Thickness (mm)						
			< 3	**da 3 a 4**	**> 4 a 6**	**> 6 a 13**	**> 13 a 20**	**> 20 a 25**	**> 25 a 32**	**> 32 a 40**	**> 40**
Three points bending MOR	EN 310	N/mm²	13	13	12	11	11	10,5	9,5	8,5	7
Three points bending MOE	EN 310	N/mm²	1.800	1.800	1.950	1.800	1.600	1.500	1.350	1.200	1.050
Internal bonding	EN 319	N/mm²	0,45	0,45	0,45	0,40	0,35	0,30	0,25	0,20	0,20

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2.2 Methods

The tests that have been carried out on the specimens, following the specifications of the standards, are as follows: three-point bending test (EN 310), volumetric swelling (EN317), perpendicular tensile strength at the panel plane, or internal bond (EN 319) and hardness (EN 1534).

2.2.1 Bending strength in accordance with CEN EN 310:1994

This type of test was carried out on two batches of samples, namely those with oxalic acid and citric acid as catalysts, with and without EP.
The purpose of the test is to define elastic modulus (MOE) and modulus of rupture (MOR).
As required by the standard, the distance between the two supports was defined by multiplying by 20 the thickness of the samples, which had thicknesses ranging between 11.5 mm and 13 mm and an average width of 50 mm.
The length of the specimens was variable but always sufficient to be correctly positioned on the supports.
The measurements were carried out by centesimal calliper, and the test was carried out with the use of a materials testing machine.
In the following table are reported the dimension and the number of samples used for bending three points tests. The total specimens exanimated were respectively 10 linseed oil oxalic acid with bioPCM and 10 without bio-PCM, 10 linseed oil with citric acid with bio-PCM and 10 without bio-PCM, 6 lignosulfunate with bio-PCM and 6 without bio-PCM, 40 mycelia in the variation with, without bio-PCM, pressed and non-pressed. As possible to see by the below tables some combination didn't respect the ratio legnht/ thickness of 20.

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Table 8 Samples used in bending tests

Bio-composites	N	Lenght (mm)	Tickness (mm)
OX	1	50,3	11,6
OX	2	50	11,58
OX	3	50,4	11,55
OX	4	50	11,5
OX	5	50	11,66
OX	6	50,06	11,75
OX	7	49,77	11,77
OX	8	49,9	11,66
OX	9	50	11,6
OX	10	49,8	11,64
OXP	11	50,73	11,82
OXP	12	50,9	11,9
OXP	13	50,95	12,1
OXP	14	51	12,1
OXP	15	50,8	11,59
OXP	16	50,6	11,8
OXP	17	50,75	11,5
OXP	18	50,97	12,3
OXP	19	50,27	13
OXP	20	50,8	12,89

Biocomposites	Lenght (mm)	Tickness (mm)
L1	50	9
L2	50	9
L3	50	9
L4	50	9
L5	50	9
L6	50	9
L_P1	50	9
L_P2	50	9
L_P3	50	9
L_P4	50	9
L_P5	50	9
L_P6	50	9

Bio-composites	N	lenght(mm)	tickness(mm)
AC	21	50,2	11,45
AC	22	50,08	11,9
AC	23	50,07	11,83
AC	24	49,96	11,6
AC	25	49,76	11,55
AC	26	50,23	11,48
AC	27	50,35	11,65
AC	28	49,95	11,9
AC	29	50,07	11,55
AC	30	50,01	11,79
ACP	31	50,05	12
ACP	32	50,1	12,08
ACP	33	50,35	11,65
ACP	34	50,16	11,64
ACP	35	50,3	11,57
ACP	36	50,3	11,64
ACP	37	50,15	11,58
ACP	38	50,3	11,53
ACP	39	50,22	11,61
ACP	40	50,28	11,71

Bio-composites	lenght(mm)	tickness(mm)
I_P1	50	20
I_P2	50	20
I_P3	50	20
I_P4	50	20
I_P5	50	20
I_P6	50	20
I_P7	50	20
I_P8	50	20
I_P9	50	20
I_P10	50	20
NI_P1	50	20
NI_P2	50	20
NI_P3	50	20
NI_P4	50	20
NI_P5	50	20
NI_P6	50	20

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NI_P7	50	20
NI_P8	50	20
NI_P9	50	20
NI_P10	50	20
NI_NP10	50	35
NI_NP9	50	35
NI_NP1	50	35
NI_NP3	50	35
NI_NP2	50	35
NI_NP4	50	35
NI_NP5	50	35
NI_NP6	50	35
NI_NP7	50	35
NI_NP8	50	35
I_NP1	50	35
I_NP2	50	35
I_NP3	50	35
I-NP4	50	35
I-NP5	50	35
I-NP5	50	35
I-NP7	50	35
I-NP8	50	35
I-NP9	50	35
I-NP10	50	35

Figure 27 AC-23 specimen, bending test

2.2.2 Volumetric swelling in accordance with CEN EN 317:1994

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In this test, water absorption and volumetric swelling of the specimens with linseed oil /oxalic acid, linseed oil/ citric acid and the batches with lignosulfonate with or without EP named respectively L-(n) and L-(n)P were evaluated.
The specimens were first measured with the calliper and weighed after being balanced in a standard climate (20°C and 65% RH), the reference measurements are 50x50x12 mm^3.
For a more accurate comparison between the specimens, two sides of each specimen were measured and the line along which the thickness was measured both before and after the test was marked by an indelible mark.
Once the measurement phase was completed, the specimens were placed inside a tank with weights above and were subsequently covered with water, inside which they remained for 24 hours.
After 24 hours, the samples were taken out of the tank and, if still intact, were remeasured on both sides, in thickness and weight.

Figure 28 AC, AC-(n)P, OX, OX-(n)P specimens in diving for volumetric swelling test

2.2.3 Internal bonding in accordance with CEN EN 319

The Internal Bonding test was the only test carried out on all batches of specimens; the purpose of the test is to measure for the samples the tensile strength perpendicular to the panel plane. This quantity provides a quantitative assessment of the internal cohesion of the panel.
The standard dimensions of the specimens are 50x50 mm^2 and the thickness is equal to the thickness of the panel (variable), but also in this case the density of each sample was calculated, to be able to relate it to the performance that was found by the test.
The procedure consisted of adhering the specimen to two aluminium supports on both its two sides by means of hot glue. When the glue had adhered correctly, the supports were hooked to the materials testing machine which applied a tensile to the specimen perpendicular to the panel plane.

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Figure 29 Explanatory figure of the internal bonding test.

2.3 Results

2.3.1 Bending strength in accordance with CEN EN 310:1994

The results are shown in Table 9.

Table 9 Bending test results for linseed oil and lignosulfonate bio-composites

Sample		MOE (MPa)	MOR (MPa)
OX	Mean	205	1,6
	St dev	39	0,3
OXP	Mean	199	1,3
	St dev	28	0,2
AC	Mean	196	1,6
	St dev	53	0,4
ACP	Mean	167	1,1
	St dev	27	0,2
L	Mean	698	3
	St dev	203	1
L P	Mean	972	3,5
	St dev	268	1

The average of the results shows that the samples impregnated with PCM undergo a decrease, although not drastic, in the values of elastic modulus and modulus of rupture. Figure 10 shows the test.
This decrease is more pronounced in specimens containing citric acid while it has a lower decrease in those with oxalic acid. Further the resistance to bending is higher on Lignosulfonate bio-composites without and with bio-PCM. In table 8 the mean and deviation standard results of mycelia bio-composites.

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Table 10 Results of bending test with mycelia bio-composites

		MOE (Mpa)	MOR (Mpa)
I_P			
	Mean	58,7	0,2
	DEV ST	33,9	0,1
NI_P			
	Mean	13,8	0,2
	DEV ST	6,5	0,04
NI_NPI			
	Mean	0,65	0,047
	DEV ST	0,08	0,007
I_NP			
	Mean	2,5	0,07
	DEV ST	1,06	0,02

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Figure 30 Three points bending test equipment

Figure 31 Three points bending test after rupture of sample

Figure 32 Three points bending test after rupture of sample I_P7

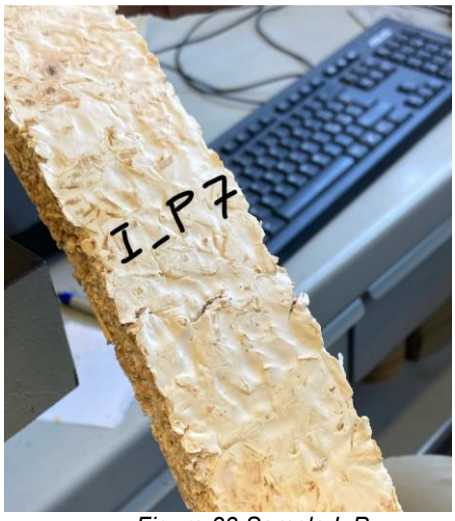

Figure 33 Sample I_P

Figure 34 Bending test on NI_P

Figure 35 NI_P1 At the of bending test

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Figure 36 Bending test on NI_NP sample

Figure 37 NI_NP5 after rupture

Figure 38 Rupture on lignosulfonate sample a with bio-PCM

2.3.2 Volumetric swelling in accordance with CEN EN 317:1994

As clearly shown by the table 6, the samples with oxalic acid and citric acid, with or without EP, withstood the test with excellent results, resulting in the 24 hours still being perfectly intact.
On the other hand, it is very different about batches with lignin, in which water absorption and swelling made measurement difficult. In addition, only 4 lignin specimens and 4 lignin and PCM specimens were evaluated once out of the water, while the others totally lost their cohesion and made evaluation impossible. In the mycelia bio-coomposites the increasing of thickness in the pressed ones was 30% with bio-PCM and 60% without bio-PCM.

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Table 11 Results of volumetric swelling tests

Sample	Increased thickness (%)	Increase in surface area (%)	Water absorbed (%)
OX	9,0	1,2	46
OXP	8,9	2,5	36
AC	5,6	1,4	41
ACP	6,8	1,9	26
L	100,5	20,3	255
LP	116,2	17,5	213
I_NP	3,2	192	314
NI_NP	1,2	224	347
I_P	29,1	189	430
NI_P	59,5	169	384

Figure 39 Two specimens with lignosulfonate-based binder compared, before (right) and after (left) the diving

2.3.3 Determination of tensile strength by internal bonding test

The results were shown in Table 6 and 7. The specimens that returned the best results were those with citric acid without PCM and the last batch of lignin with PCM, which in this case significantly increased performance compared to the reference (while in the other cases performance decreases in the presence of PCM).
PCM influenced some tests by leading to adhesion problems between the sample and the aluminium plates used for the test, this due to its composition that hinders adhesion and gives the specimens a greasy appearance by touch. This was particularly evident in PCM-impregnated oxalic acid specimens.
The results obtained with mycelia bio-composites showed an order of magnitude less than linseed oil bio-composites.
Finally, the tests with demo product of thickness 10 mm 15 mm recorded very low results, shown in Table 8, so much so that some specimens broke during positioning in the machine.

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Table 12 Results of internal bonding tests

Sample		Density (g/cm^3)	Rupture (MPa)
OX	Mean	0,645	0,15
	Dev st	0,031	0,03
OXP	Mean	0,769	0,10
	Dev st	0,037	0,04
AC	Mean	0,657	0,30
	Dev st	0,061	0,09
ACP	Mean	0,813	0,10
	Dev st	0,010	0,04
L	Mean	0,568	0,01
	Dev st	0,008	0,00
LP	Mean	0,739	0,31
	Dev st	0,034	0,01

Table 13 Mycelia based bio-composites.

Sample		Rupture (MPa)
I_NP	Mean	0,039
	Dev st	0,003
NI_NP	Mean	0,014
	Dev st	0,007
I_P	Mean	0,018
	Dev st	0,010
NI_P	Mean	0,006
	Dev st	0,005

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Table 14 Tensile strenght of products from Permanovo DEMO

Internal bonding				
	Density (g/cm3)		Rupture (MPa)	
	Mean	Standard dev.	Mean	Standard dev.
DEMO PERVANOVO 10 mm	**0,304**	0,007	**0,01**	0,003
DEMO PERVANOVO 10 mm PCM	**0,453**	0,010	**0,06**	0,013
DEMO PERVANOVO 15 mm	**0,307**	0,008	**0,01**	0,003
DEMO PERVANOVO 15 mm PCM	**No data available**	No data available	**0,12**	0,022

2.3.4 Hardness in accordance with CEN EN 1534:2023

The hardness test, as for bending, was performed on all batches of specimens measuring 50 x 50 mm thickness. The test consists of placing the specimen on the materials testing machine, on which a tool is mounted that has a ball through which a predetermined known force is applied to the surface of the specimen.
This sphere leaves an imprint on the face of the sample, of which the two diameters are calculated. By means of a formula in which the diameters of the indentation, the diameter of the ball and the force applied are considered, the resulting value of the test is found, this value indicates the ability of the sample to resist penetration by a harder body.

Figure 40 AC-32P Hardness Test Specimen

Figure 41 NI_NP on hardness equipment

The results, tables 13 and 14, reflected what was initially expected, namely that PCM impregnation decreases the mechanical characteristics of the samples. The highest hardness values are found in samples with oxalic acid impregnated with bio-PCM and in those with citric acid and bio-PCM, this shows that in this type of test the bio-PCM increases the hardness of the samples instead of decreasing their characteristics. It the same for lignosulfonate specimens and even, at very lower values for mycelia bio-composites. In the bio-composites performed with mycelia some samples cannot to be attributed a value since the diameter of the print on the

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surface is greater than ones of the sphere used for doing it. The skin of specimen based on fungal mycelium is more flexible than substrate interconnected with fungal mycelium and this fact determine a print on the surface without a rupture of the specimen at the parameters used.

Table 15 Results of hardness tests

Sample		Brinell test HB
OX	Mean	0,463
	Dev st	0,095
OXP	Mean	0,788
	Dev st	0,249
AC	Mean	0,580
	Dev st	0,125
ACP	Mean	0,922
	Dev st	0,292
L	Mean	1,74
	Dev st	0,31
LP	Mean	2,22
	Dev st	0,60

Table 16 Mycelia bio-composites results

Sample		Brinell test HB
I_NP	Mean	0,02
	Dev st	0,002
NI_NP	Mean	0,02
	Dev st	0,005
NI_P	Mean	0,14
	Dev st	0,04
I_P	Mean	0,15
	Dev st	0,1

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Figure 42 Hardness test on mycelium bio-composites

Figure 43 Mycelia specimen after hardness test

Figure 44 Hardness test on linseed oil bio-composites

Figure 45 Hardness test on linseed oil bio-composites

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2.3.5 References

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UNI EN 317:1994 Pannelli di particelle di legno e pannelli di fibra di legno. Determinazione del rigonfiamento dello spessore dopo immersione in acqua.
UNI EN 319:1994 Pannelli di particelle di legno e pannelli di fibra di legno. Determinazione della resistenza a trazione perpendicolare al piano del pannello.
UNI EN 1534:2020 Pavimentazioni di legno e parquet - Determinazione della resistenza alla penetrazione - Metodo di prova.
UNI EN 312:2010 Particleboards SpecificationsUNI CEN/TS 16368:2014 Light particle board – Specifications.

3 Acoustic properties of bio-composites

3.1 Acoustic characterisation of biomaterials

Acoustic characteristics of the developed materials were tested according to the standard ISO 10534-2 (2023) [15]. To evaluate the sound absorption performance of the developed bio-based composite materials, impedance tube measurements were carried out using an ACUPRO two-microphone impedance tube system. The measurements aimed to compare the acoustic behaviour of commercial particleboard as control, bio-based wallboards produced with the three binders, with and without bioPCM, i.e., ethyl palmitate (EP).

3.1.2 Sample preparation

The investigated materials included a commercial particleboard reference (Ref), particleboards manufactured using epoxidized linseed oil with oxalic acid (OA) or citric acid (CA) as hardeners, bioPCM-containing variants of these boards (OA-PCM and CA-PCM), particleboards produced using lignin-based binders (HP-CO1) and its bioPCM-containing variant (HP-TLO1), and lightweight mycelia-based composites produced from poplar particles and fungal mycelium (30Poplar-NonIM-NonComp) and its bioPCM-containing variant (30Poplar-IM-NonComp). In the bioPCM-containing samples, the wood particles were impregnated with ethyl palmitate before board production.
For the particleboard samples, circular specimens with a diameter of *ca.* 34 mm and a thickness of *ca.* 9 mm were prepared using a stationary drilling machine equipped with a circular cutting tool. The final thickness was adjusted by sanding to ensure proper fitting in the impedance tube. Specimen preparation procedure and samples are shown in figure 46.

Figure 46 Stationary drilling machine equipped with a circular cutting tool and the circular cutting tool with a representative circular specimen (left); the groups of tested specimens (right)

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For the mycelia-based composites, a specially designed cutting tool was used to produce specimens with a similar diameter but a thickness of approximately 38 mm (Figure 47). The average dimensions and densities of all tested samples are presented in Table 17. According to the standard, five replicate specimens were prepared and tested for each material type. Prior to acoustic testing, all specimens were conditioned at 22°C and 35% relative humidity.

Figure 47 Cutting tool used for the preparation of specimens from mycelia-based boards

Table 17. Average dimensions and density of the samples. Standard deviation within parentheses.

Sample name	Thickness (mm)	Diameter (mm)	Weight (g)	Density (g/cm^3)
Ref	9.89 (0.11)	33.91 (0.03)	6.09 (0.16)	0.68 (0.01)
OA	9.95 (0.45)	34.02 (0.08)	5.72 (0.68)	0.63 (0.06)
OA-PCM	10.49 (0.3)	33.90 (0.1)	7.26 (0.17)	0.77 (0.03)
CA	10.04 (0.49)	34.04 (0.03)	5.71 (0.61)	0.62 (0.05)
CA-PCM	10.34 (0.34)	33.98 (0.07)	8.26 (0.35)	0.88 (0.02)
HP-CO1	9.08 (0.08)	33.96 (0.03)	4.92 (0.47)	0.60 (0.06)
HP-TLO1	9.25 (0.08)	33.96 (0.05)	6.36 (0.43)	0.76 (0.05)
30Poplar-NonIM-NonComp	36.71 (0.68)	34.22 (0.22)	4.06 (0.42)	0.12 (0.01)
30Poplar-IM-NonComp	36.69 (0.68)	34.76 (0.19)	5.56 (0.22)	0.16 (0.01)

3.1.3 Measurement setup and procedure

The acoustic performance of the materials was evaluated using a two-microphone ACUPRO impedance tube system (Figure 48). The measurements were carried out to determine the normal-incidence sound absorption coefficient of the tested materials. The measurement procedure followed the requirements of ISO 10534-2.
The impedance tube was equipped with two microphones. The distance between the sample surface and the nearest microphone was 50.80 mm, while the spacing between the two microphones was 29.21 mm. Measurements were conducted over a frequency range from ~0 to 6000 Hz with a frequency resolution of 7.5 Hz. For each specimen, the sound absorption coefficient was recorded as a function of frequency. In addition, the software generated reduced 1/3-octave-band datasets that were subsequently used for the calculation of some of the summary acoustic indicators.

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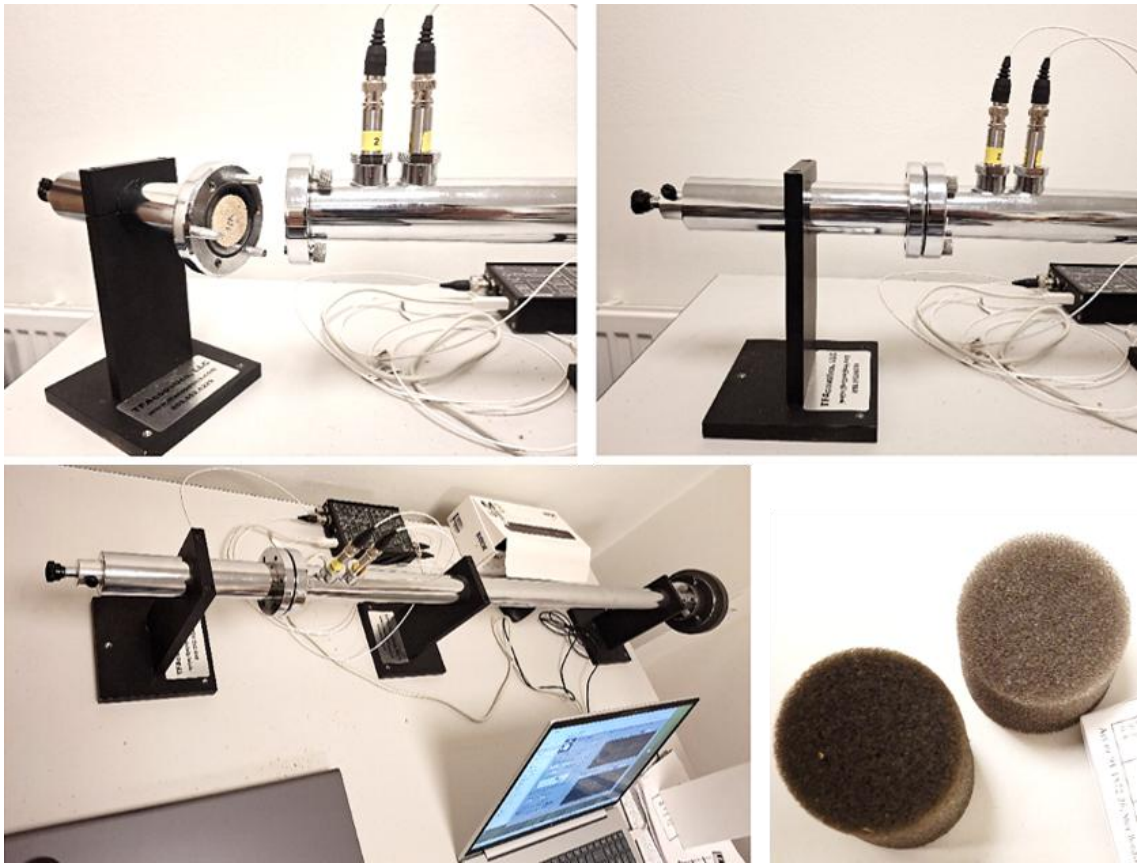

Figure 48 ACUPRO impedance tube showing the locations of the sample, microphones, and speaker. Standard open-cell foam used for impedance tube calibration (down right corner)

Prior to testing, the system was calibrated using reference open-cell foam specimens supplied for impedance tube calibration. For the particleboard materials, a calibration foam with a thickness of *ca.* 9 mm and a diameter of approximately 34 mm was used. For the mycelia-based composites, a calibration foam with a thickness of *ca.* 38 mm and a similar diameter was employed. The calibration specimens are shown in Figure 48.

3.2 Sound absorption coefficient curves

The average sound absorption coefficient (SAC) curves of all investigated materials are presented in Fig. 4. The curves are based on the average values obtained from repeated measurements, while the corresponding standard deviations are shown in Fig. 7 as shaded regions around the mean curves[1].
Figure 39 compares the sound absorption behaviour of all tested materials over the investigated frequency range. The results demonstrate distinct acoustic responses among the different material systems. The commercial particleboard (Ref) exhibited a different absorption profile from the developed bio-based materials, showing relatively high absorption at low frequencies, followed by a decrease in the mid-frequency range and a subsequent increase at higher frequencies.
The bio-based wallboards produced using epoxidized linseed oil and lignin-based binders generally showed lower sound absorption coefficients at low frequencies and a gradual increase in absorption with increasing frequency. In contrast, the mycelia-based composites displayed considerably higher absorption levels over a broad frequency range, particularly in the low- and mid-frequency regions.

[1] The sound absorption curves presented in this report are based on 1/3-octave-band data generated by the instrument software. In a 1/3-octave representation, the measured frequency spectrum is divided into standardized frequency bands, each characterized by a centre frequency (e.g., 125, 160, 200, 250, 315 Hz, etc.). The sound absorption coefficient reported for each band is obtained by averaging the measured spectral data within the corresponding frequency interval. This procedure reduces the influence of local fluctuations and measurement noise while preserving the overall acoustic behaviour of the material [1-3].

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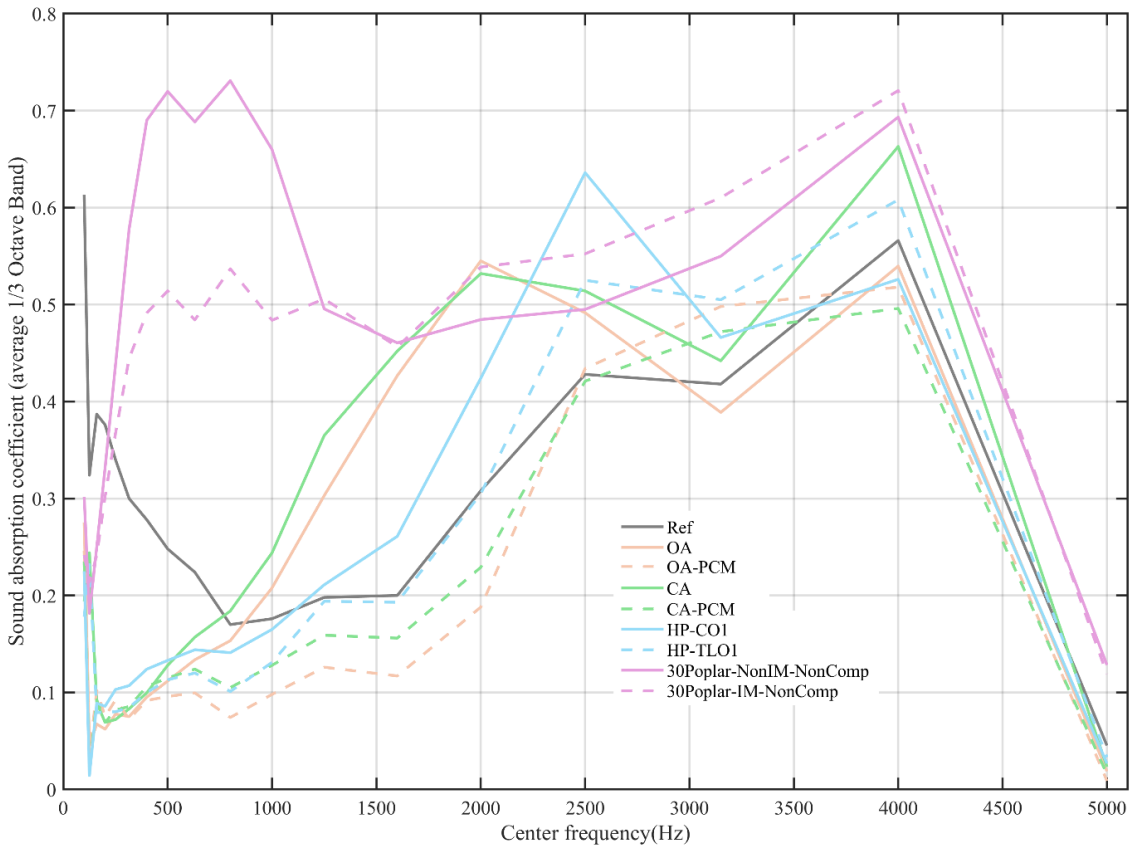

Figure 49 Sound absorption coefficient of all samples (impregnated and nonimpregnated) as a function of frequency

For clarity, the developed materials are further grouped in Figures 49 and 50 according to the presence or absence of bioPCM. The comparison indicates that bioPCM incorporation did not fundamentally alter the overall shape of the absorption curves. However, differences in absorption magnitude were observed for several material systems and frequency ranges. These effects are discussed in detail in the following sections using summary acoustic indicators such as the Noise Reduction Coefficient (NRC), maximum sound absorption coefficient, and frequency-band-averaged absorption coefficients.

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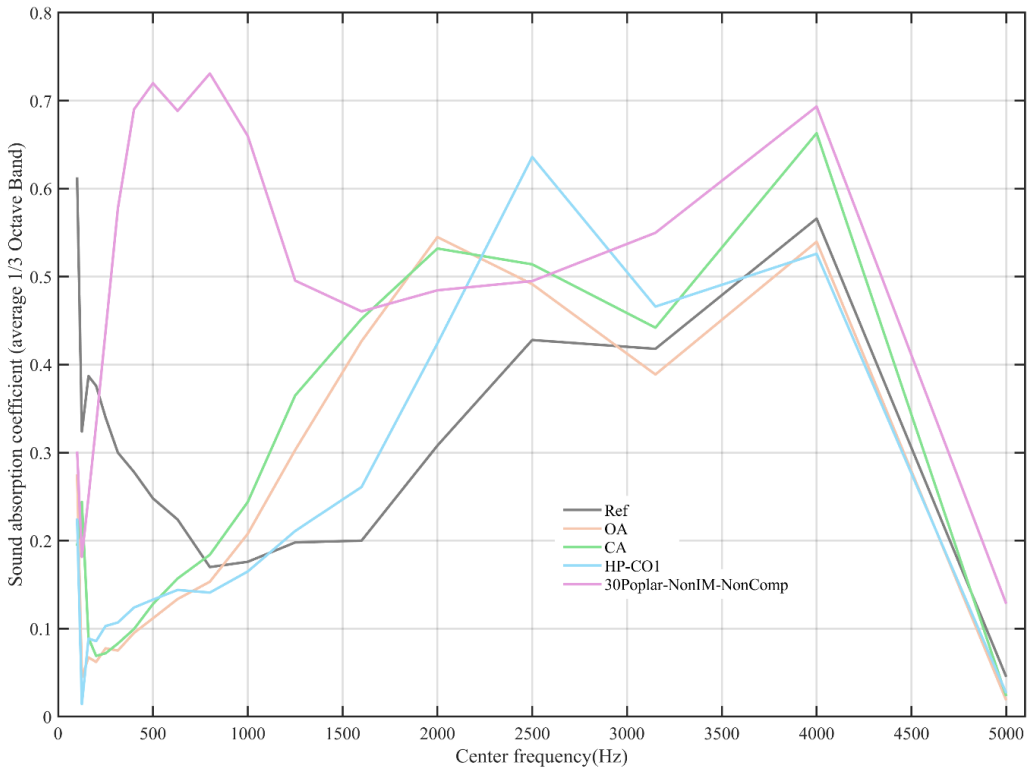

Figure 50 Sound absorption coefficient of all nonimpregnated samples as a function of frequency

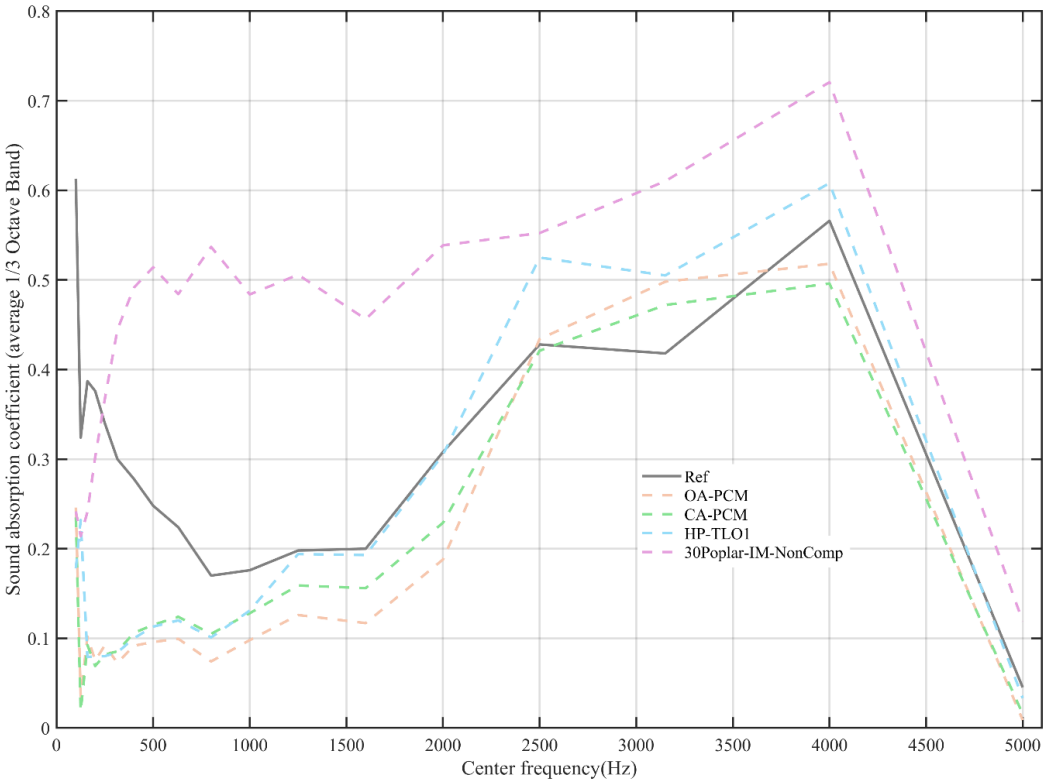

Figure 51 Sound absorption coefficient of all impregnated samples as a function of frequency

The standard deviation bands presented in Figure 52 indicate good repeatability of the measurements. Larger variations were observed in frequency regions where the absorption coefficients were higher.

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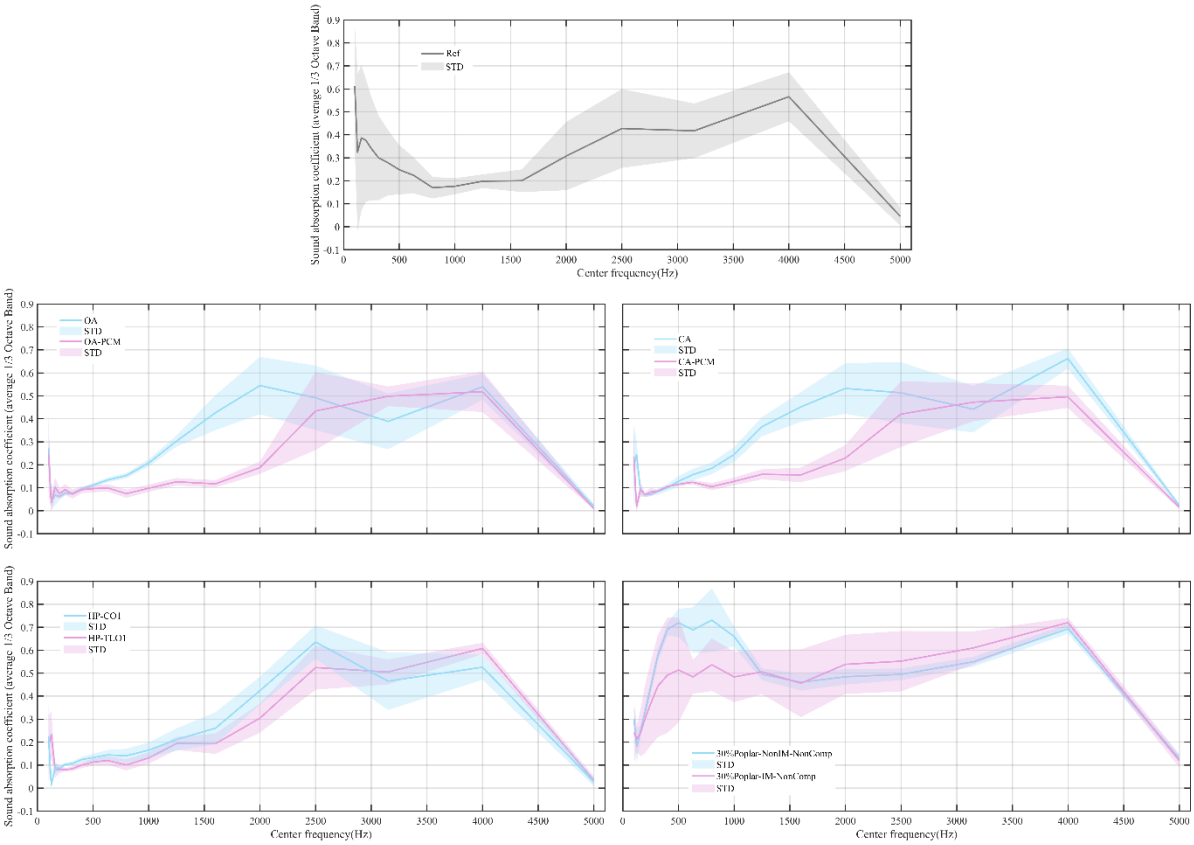

Figure 52 Sound absorption coefficient average (line) and standard deviation (shadow) of pairs of samples as a function of frequency

3.3 Noise reduction coefficient (NRC)

To facilitate comparison among the tested materials, the Noise Reduction Coefficient (NRC) was determined for each sample type. NRC is one of the most widely used single-number indicators of sound absorption performance and provides a simplified representation of the acoustic behaviour of a material [4]. According to ASTM C423, the NRC is calculated as the arithmetic average of the sound absorption coefficients measured at 250, 500, 1000, and 2000 Hz [5]:

$$\mathrm{NRC} = \frac{\alpha_{250} + \alpha_{500} + \alpha_{1000} + \alpha_{2000}}{4}$$

where α represents the sound absorption coefficient at the corresponding frequency. Although NRC does not fully describe the frequency-dependent behaviour of a material, it provides a convenient metric for comparing the overall sound absorption performance of different materials [6] and is therefore widely reported in both scientific literature and industrial applications.

The average NRC values obtained for the studied materials are presented in Figure 8. Among all tested materials, the mycelia-based composite 30Poplar-NonIM-NonComp exhibited the highest NRC value (0.576 ± 0.161), followed by its PCM-containing counterpart 30Poplar-IM-NonComp (0.476 ± 0.088). These values were considerably higher than those obtained for the particleboards, indicating superior overall sound absorption performance.

For the particleboard systems, NRC values ranged from approximately 0.12 to 0.27. The commercial reference board (0.268 ± 0.105) and the lignin-based board HP-CO1 (0.263 ± 0.026) showed the highest NRC values

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within this group. The OA and CA boards exhibited comparable performance, with NRC values of 0.236 ± 0.029 and 0.244 ± 0.017, respectively.
A consistent reduction in NRC was observed following PCM incorporation. The NRC value decreased from 0.236 to 0.118 for the OA system, from 0.244 to 0.138 for the CA system, from 0.263 to 0.157 for the HP system, and from 0.576 to 0.476 for the mycelia-based composite. These results suggest that impregnation with ethyl palmitate reduced the overall sound absorption performance of the investigated materials.

The observed reduction may be associated with partial filling of wood cell lumen, piths and cell wall checks within the wood material structure by the PCM, thereby reducing the available pathways for dissipation of acoustic energy [6-7]. This explanation is consistent with the general understanding of sound absorption mechanisms in porous materials, where interconnected porosity plays a key role in acoustic energy dissipation [8].

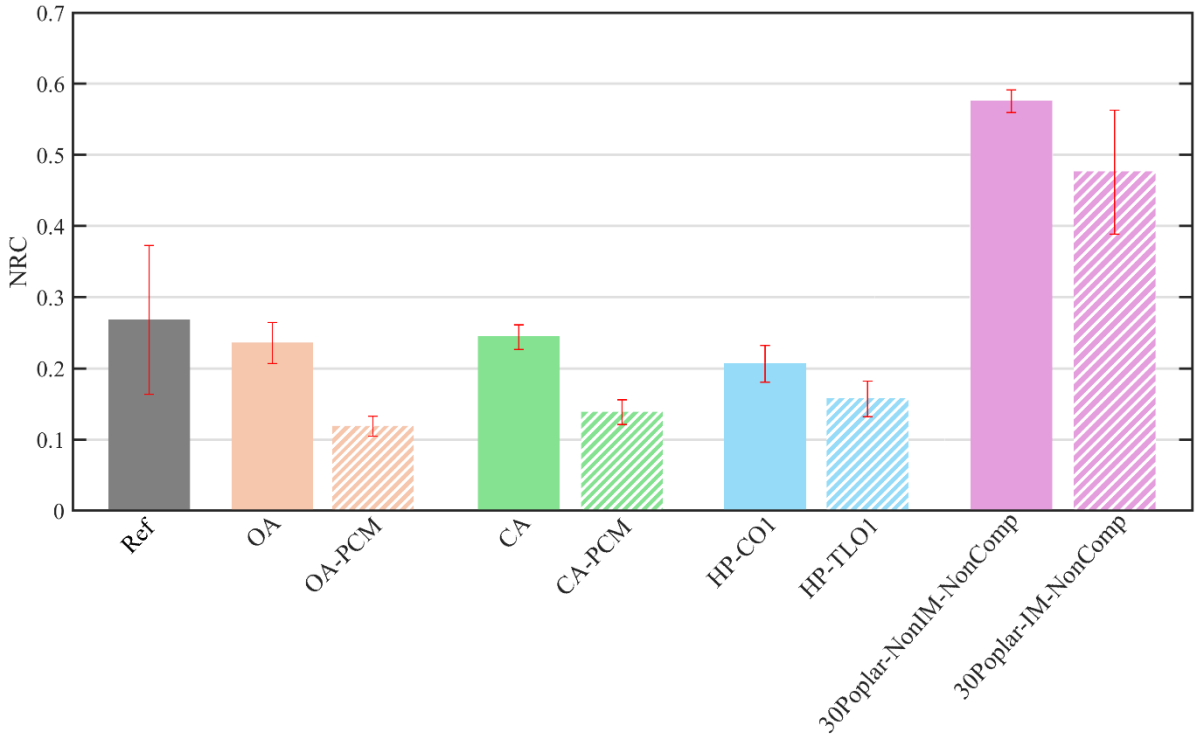

Figure 53 Noise reduction coefficients (NRC) of the tested samples

3.4 Maximum sound absorption coefficient at low-, mid-, and high-frequency ranges

In addition to NRC, the maximum sound absorption coefficient (α_{max}) and the frequency at which this maximum occurred (f_{peak}) were determined for all investigated materials. To reduce the influence of local fluctuations in the measured spectra, a moving-average smoothing procedure was applied prior to identifying the maximum absorption coefficient.
Figure 53 summarizes the average values and corresponding standard deviations of both α_{max} and f_{peak} for all tested materials, while Figure 10 presents the same information grouped according to material type and PCM impregnation.
The maximum sound absorption coefficients ranged from approximately 0.57 to 0.77. The highest values were observed for the mycelia-based composites, with α_{max} values of 0.740 ± 0.026 and 0.766 ± 0.043 for 30Poplar-NonIM-NonComp and 30Poplar-IM-NonComp, respectively. These results are consistent with the NRC analysis and further confirm the superior acoustic performance of the mycelia-based materials.

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For the particleboard materials, the maximum sound absorption coefficients were generally within a relatively narrow range between approximately 0.57 and 0.67. The CA board exhibited the highest αmax among the particleboard formulations (0.672 ± 0.045), while CA-PCM showed the lowest value (0.568 ± 0.027).
Compared with the NRC results, a different trend was observed regarding PCM incorporation. While the NRC values consistently decreased after PCM treatment, the maximum sound absorption coefficient was not always reduced. In several material systems, including OA, HP, and the mycelia-based composites, the PCM-containing variants exhibited similar or even slightly higher αmax values than their non-PCM counterparts.
This apparent discrepancy arises from the different nature of the two acoustic indicators. NRC represents an average sound absorption performance over several frequencies, whereas αmax characterizes only the highest absorption level achieved within the investigated frequency range. Consequently, materials may exhibit a high absorption coefficient at a specific frequency while maintaining lower absorption over the remainder of the spectrum. Such behaviour can lead to relatively high α_{max} values despite lower NRC values.
The frequencies corresponding to the maximum absorption coefficient were generally located in the higher-frequency region, typically between 3000 and 4000 Hz. This observation is consistent with the sound absorption curves presented earlier, which showed increasing absorption performance with increasing frequency for most of the developed materials.
The relatively large standard deviations observed for some materials indicate variability in the exact frequency at which the maximum absorption occurred.

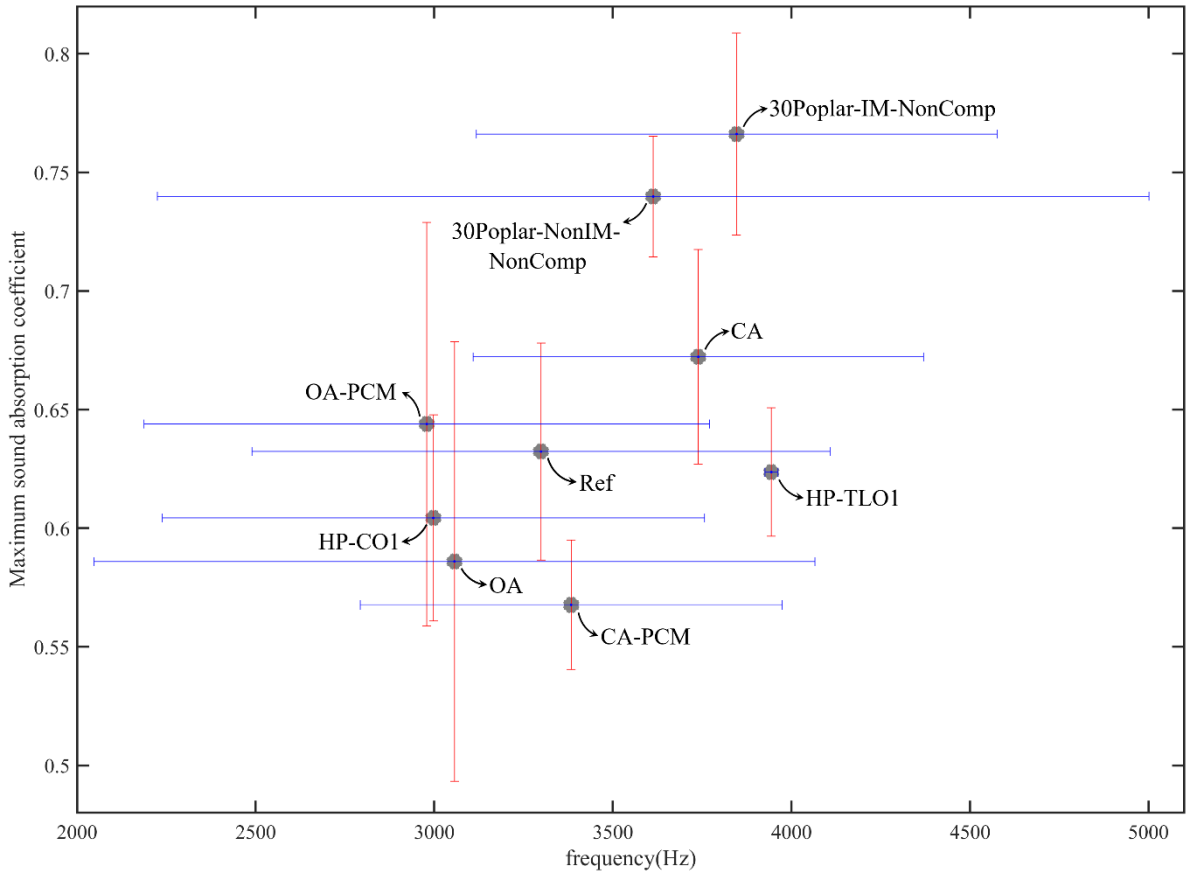

Figure 54 Maximum sound absorption coefficient and corresponding frequency for all samples (non-impregnated and impregnated with bioPCM). Vertical and horizontal error bars indicate the standard deviations of the sound absorption coefficient and frequency, respectively.

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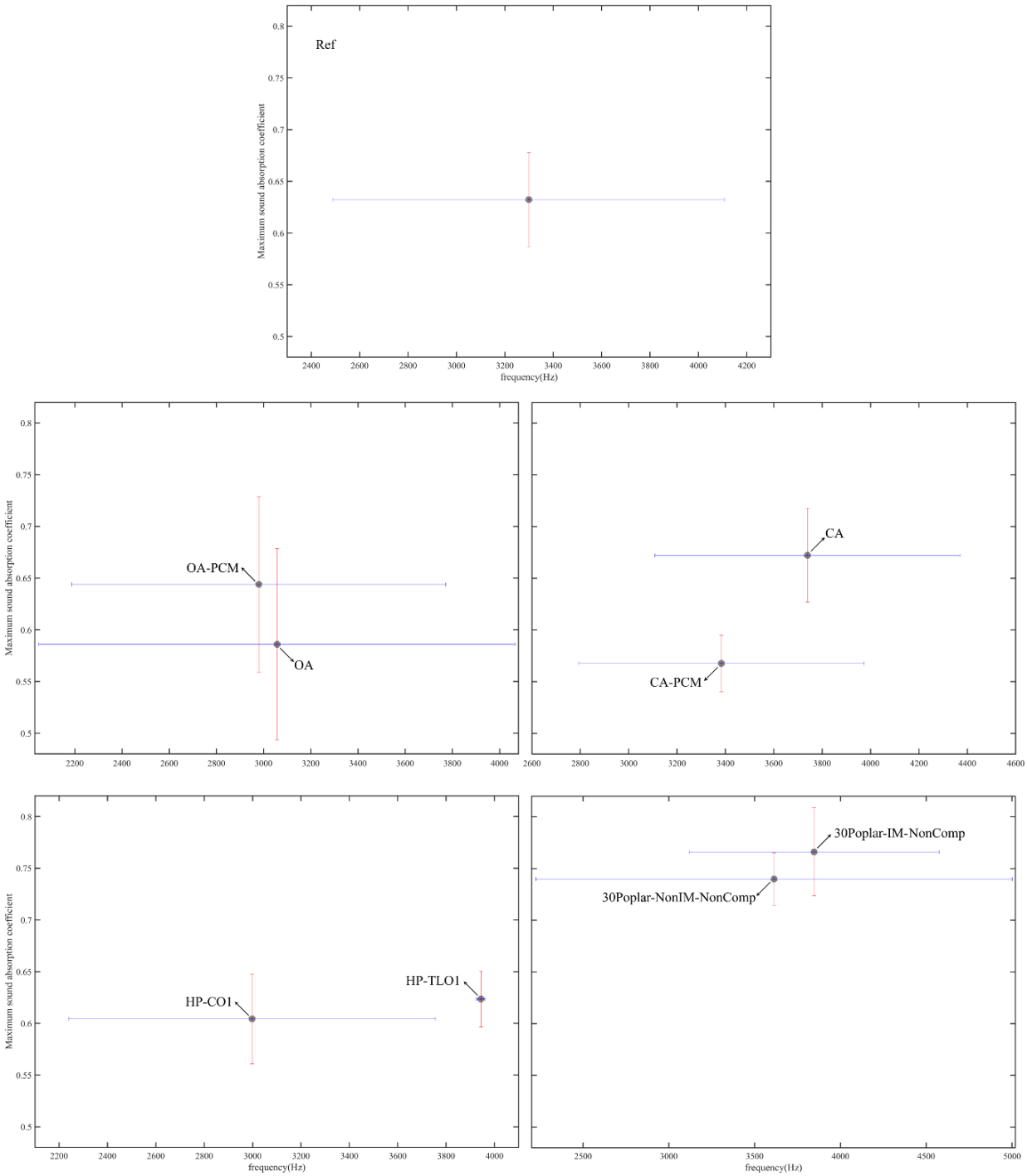

Figure 55 Maximum sound absorption coefficient and corresponding frequency for pairs of samples

To provide a more detailed assessment of the acoustic behaviour of the investigated materials, the average sound absorption coefficient was calculated for low-frequency (100–500 Hz), mid-frequency (500–2000 Hz), and high-frequency (2000–5000 Hz) ranges. The results are presented in Table 18.

While NRC provides a single-number rating of sound absorption performance, frequency-band averaging enables a more detailed evaluation of the frequency regions in which each material is most effective. This approach is commonly used in acoustic-material studies because sound absorption mechanisms and material performance can vary substantially across the frequency spectrum [9].

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The commercial particleboard (Ref) exhibited relatively high absorption in the low-frequency region (0.358 ± 0.249), lower absorption in the mid-frequency region (0.213 ± 0.085), and improved performance again at higher frequencies (0.364 ± 0.226).
Among the particleboard materials produced with bio-based binders, sound absorption generally increased with increasing frequency. The OA, CA, HP-CO1, and HP-TLO1 boards showed relatively limited absorption in the low-frequency region, moderate absorption in the mid-frequency region, and their highest average absorption values in the high-frequency range. This trend is characteristic of relatively dense wood-based materials, where efficient sound absorption is more readily achieved at shorter wavelengths [10].
The CA and HP-based boards exhibited the highest high-frequency absorption among the particleboard formulations, with average values exceeding 0.41. In contrast, their low-frequency absorption remained relatively limited, indicating that these materials are more effective for controlling medium- and high-frequency sound.
The mycelia-based composites demonstrated different acoustic behaviour. Both 30Poplar-NonIM-NonComp and 30Poplar-IM-NonComp exhibited substantially higher absorption coefficients in the low- and mid-frequency ranges than all particleboard materials. The non-PCM mycelia-based composite achieved average absorption coefficients of 0.436 ± 0.199 and 0.587 ± 0.130 in the low- and mid-frequency ranges, respectively, while the PCM-containing counterpart reached values of 0.352 ± 0.199 and 0.501 ± 0.110. These results indicate a broader and more balanced sound absorption performance across the investigated frequency range.
The frequency-band analysis also helps explain the trends observed in the NRC results. The superior NRC values obtained for the mycelia-based composites can largely be attributed to their enhanced low- and mid-frequency absorption performance. Since NRC is calculated using absorption coefficients at 250, 500, 1000, and 2000 Hz, materials with improved absorption within these frequency regions naturally achieve higher NRC values.
The effect of PCM incorporation was found to depend on the frequency range. For the OA, CA, and mycelia-based systems, PCM incorporation reduced the average absorption coefficient in both the low- and mid-frequency ranges. The reduction may be associated with partial filling of pores and voids by bioPCM, thereby reducing the dissipation of acoustic energy within the porous structure.

Interestingly, the high-frequency absorption performance was less affected by PCM incorporation. In some cases, such as OA-PCM, HP-TLO1, and 30Poplar-IM-NonComp, the average high-frequency absorption coefficient remained similar to or slightly higher than that of the corresponding non-PCM material. This observation helps explain why several PCM-containing materials maintained comparable maximum sound absorption coefficients despite exhibiting lower NRC values.
The frequency-band analysis therefore provides an important link between the sound absorption curves, the NRC values, and the maximum sound absorption coefficients.

Table 18. Average sound absorption coefficients of the samples in low-, mid-, and high-frequency ranges. Standard deviation withing parentheses.

Materials	Average sound absorption coefficient at low-frequency (100-500 Hz)	Average sound absorption coefficient at mid-frequency (500-2000 Hz)	Average sound absorption coefficient at mid-frequency (2000-5000 Hz)
Ref	0.3581 (0.2489)	0.2126 (0.0847)	0.3641 (0.2263)
OA	0.1012 (0.0802)	0.2949 (0.1615)	0.3597 (0.2275)
OA-PCM	0.1014 (0.0802)	0.1171 (0.0400)	0.3649 (0.2312)
CA	0.1225 (0.0873)	0.3225 (0.1504)	0.4105 (0.2550)
CA-PCM	0.1004 (0.0697)	0.1499 (0.0488)	0.3511 (0.2154)
HP-CO1	0.1102 (0.0729)	0.2244 (0.1090)	0.4135 (0.2473)
HP-TLO1	0.1184 (0.0779)	0.1740 (0.0771)	0.4177 (0.2371)
30Poplar-NonIM-NonComp	0.4362 (0.1986)	0.5866 (0.1304)	0.4666 (0.2137)

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30Poplar-IM-NonComp	0.3520 (0.1990)	0.5009 (0.1099)	0.5005 (0.2442)

3.5 Density/NRC correlation

To investigate the influence of material density on acoustic performance, NRC was plotted against the density of all tested specimens (Figure 56). A total of 54 individual specimens representing all investigated material types were included in the analysis. A clear negative relationship was observed between density and NRC. Linear regression analysis yielded the following relationship:

$$\mathrm{NRC} = -0.5638\rho + 0.5962$$

where ρ is the density expressed in g/cm³. The strong correlation $R^2 = 0.86$ suggests that density is an important parameter influencing the acoustic behaviour of the developed materials.
The observed trend is consistent with the results presented in the previous sections. The mycelia-based composites, which exhibited the lowest densities among the investigated materials, also showed the highest NRC values and the highest average sound absorption coefficients in the low- and mid-frequency ranges. In contrast, the denser particleboard materials generally exhibited lower NRC values.
From a physical perspective, lower-density porous materials typically contain a larger volume fraction of interconnected voids through which incident sound waves can penetrate. As sound propagates through these porous networks, acoustic energy is dissipated. Consequently, increased porosity often leads to improved sound absorption [11].

It should be noted, however, that density alone does not fully determine acoustic performance. Other structural parameters, including pore size distribution, pore connectivity, tortuosity, and air flow resistivity, are also known to influence sound absorption. Therefore, the observed relationship should be interpreted as a strong empirical correlation within the investigated material systems rather than as a universal predictive model [12-14].

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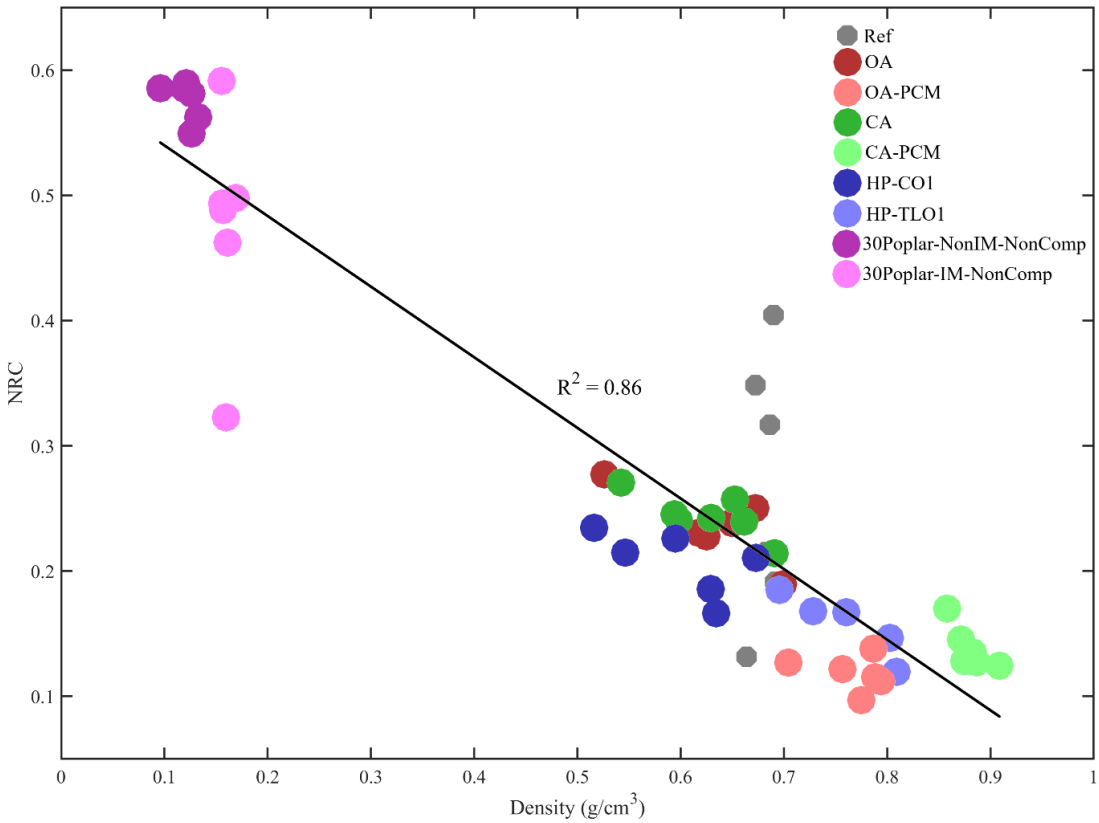

Figure 56 Correlation between material density and NRC for all tested specimens

3.6 Conclusions

The acoustic performance of a commercial particleboard, several bio-based wallboards, and lightweight mycelia-based composites were evaluated. The results showed that the acoustic behaviour of the materials investigated was strongly dependent on both material composition and density. While the particleboard materials exhibited increasing sound absorption with increasing frequency, the mycelia-based composites demonstrated superior broadband performance, particularly in the low- and mid-frequency ranges. Consequently, the mycelia-based composites achieved the highest NRC values among all investigated materials.

The incorporation of bioPCM reduced NRC and frequency-band-averaged sound absorption coefficients, particularly in the low- and mid-frequency ranges, although the maximum sound absorption coefficient was often maintained or slightly improved. A strong negative correlation was observed between density and NRC (R^2 = 0.86), indicating that lower-density and more porous structures provided enhanced sound absorption performance.

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4 Particle boards characterisation with X-ray micro-CT and 3D image analysis

4.1 Particle boards samples and treatments

In the Tables 19, 20 and 21 are reported photos of the panels produced with three different bio-based binders with and without addition of ethil-palmitate as bio-PCM. Photos and size of the scanned samples are also indicated.

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Table 19 Wood particle boards with lignin as binder investigated with X-ray micro-CT

Treatment	Photos		Sample size	
			Thickness (cm)	Volume (cm^3)
dry lignin, no bPcM			1,5	2,65
dry lignin with bPCM			1,5	2,65
MW dried			1,8	4,58

Table 20 Wood particle boards with epoxy oil as binder investigated with X-ray micro-CT

Treatment	Photos		Sample size	
			Thickness (cm)	Volume (cm^3)
no bPcM			1,2	1,36
with 25% bPCM			1,2	1,36

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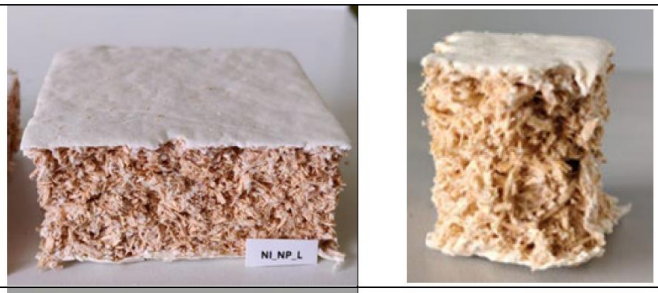
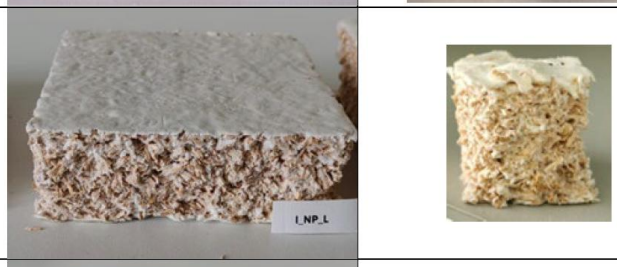
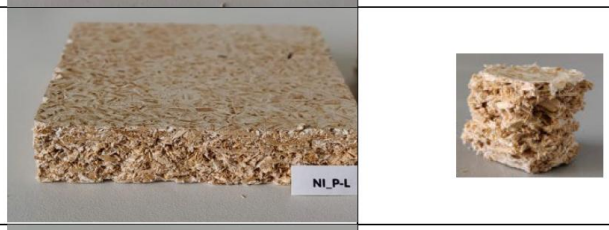
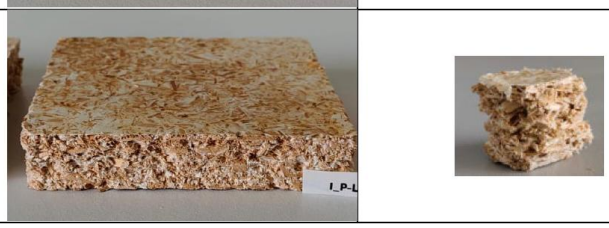
Table 21: Wood particle boards with mycelium as binder investigated with X-ray micro-CT

Treatm.	Photos	Sample size	
		Thickness (cm)	Volume (cm^3)
NI-NP		4,0	50,27
I NP		3,0	21,21
NI-P		2,0	6,28
I_P		2,0	6,28

4.2 Sample board imaging

Cylindrical or cubic samples have been collected from the particle boards with diameter/side equal to the board thicknesses (Tables19, 20 and 21). They have been scanned with a Skyscan 1272 desktop microtomograph (Bruker Corporation) with the setting parameters reported in table 22.

Table 22 Setting parameters used for micro-CT imaging

Sample		Source voltage	Source current	Exposure	Filter	Object to source distance	Voxel size
Binder	**Treatment**	**(kV)**	**(mA)**	**(ms)**	**(mm)**	**(mm)**	**(µm)**
Dry lignin	no bPcM	60	166	2269	Al 0.25	192,8	10,5
Dry lignin	with bPCM	60	166	1464	Al 0.25	142,5	9,5
Lignin-based	MW dried	60	166	1464	Al 0.25	135,2	9,0

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Epoxy oil	no bPcM	60	166	1464	Al 0.25	135	9,0
Epoxy oil	with 25% bPCM	60	166	1053	Al 0.25	93,2	8.0
Mycelium	NI-NP	60	166	2269	Al 0.25	165,3 (4 scans)	9,0
Mycelium	I NP	60	166	2495	Al 0.25	165,2 (4 scans)	9,0
Mycelium	NI-P	60	166	2269	Al 0.25	200,1	10,9
Mycelium	I_P	60	166	2495	Al 0.25	200,1	10,9

4.3 Results

Images of the cross sections of the particle board samples obtained from the micro-CT scans have been first binarised applying the Otsu method, then processed by a de-speckling procedure to remove the noise. Porosity profiles have been calculated by counting the pixels representing pores in each section parallel to the board plane inside the sample, finally the successive opening morphological procedure has been applied to calculate the pore size distribution of the whole sample.
Figures 57, 588 and 59 show cross and coronal sections reconstructed by x-ray micro-CT, as well as a coronal radiograph to evidence the internal structure of the boards. The porosity profile is also showed to measure inhomogeneities inside the board thickness.
In figure 60 are reported the pore size distributions of all the investigated samples while in Table 23 the measured average structural properties are resumed.

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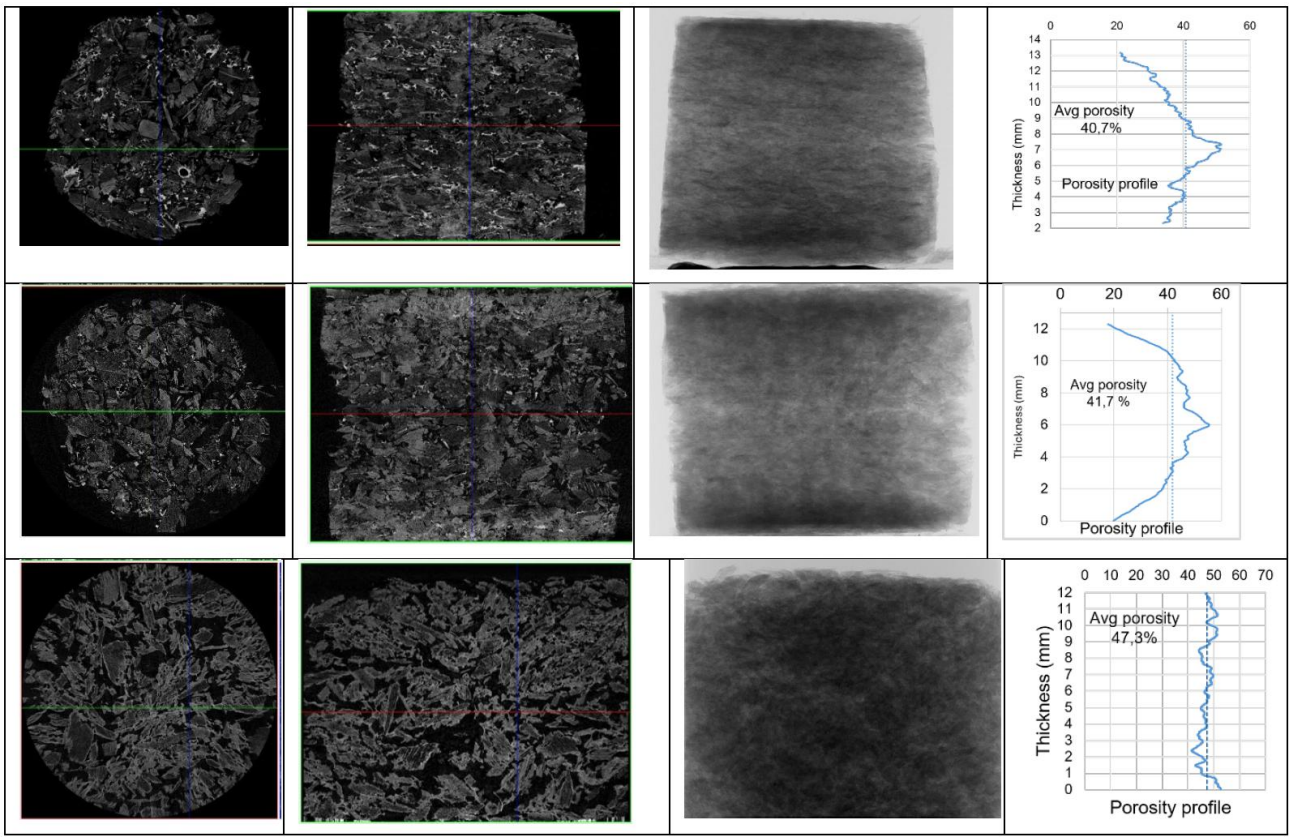

Figure 57 Cross and coronal x-ray sections, coronal radiograph and porosity profiles of lignin binder-based panels. Dry lignin, no bPCM sample in the first row. Dry lignin, with bPCM sample in the second row. Lignin-based binder MW dried, with bPCM sample in the third row.

Dry lignin, no bPCM sample in the first row of figure 57 exhibits weak binder in the middle where porosity is max. Fragile when cut. splits in two. Brighter parts in micro-CT cross sections are lignin accumulation locations.
Dry lignin, with bPCM sample in the second row of figure 57 shows weak binder in the middle where porosity is max. Fragile when cut, splits in two. Brighter spots in micro-CT cross sections indicate lignin accumulation locations.
Lignin-based binder MW dried, with bioPCM sample in the third row of figure 57 indicates a homogeneous panel with high mean pore size due to wood particle aggregate formation. Binder (brighter parts in cross sections) appears uniformly distributed on the outer surface of the wood particle aggregates.

Epoxy oil wthout bPCM sample in the first row of figure 58 is a very homogeneous, compact and rigid panel with low mean pore size due to well compacted wood particles
Epoxy oil wth bPCM sample in the second row of figure 58 is a very homogeneous, compact and rigid panel with low mean pore size and porosity (the lowest among those analysed). Well compacted wood particles.

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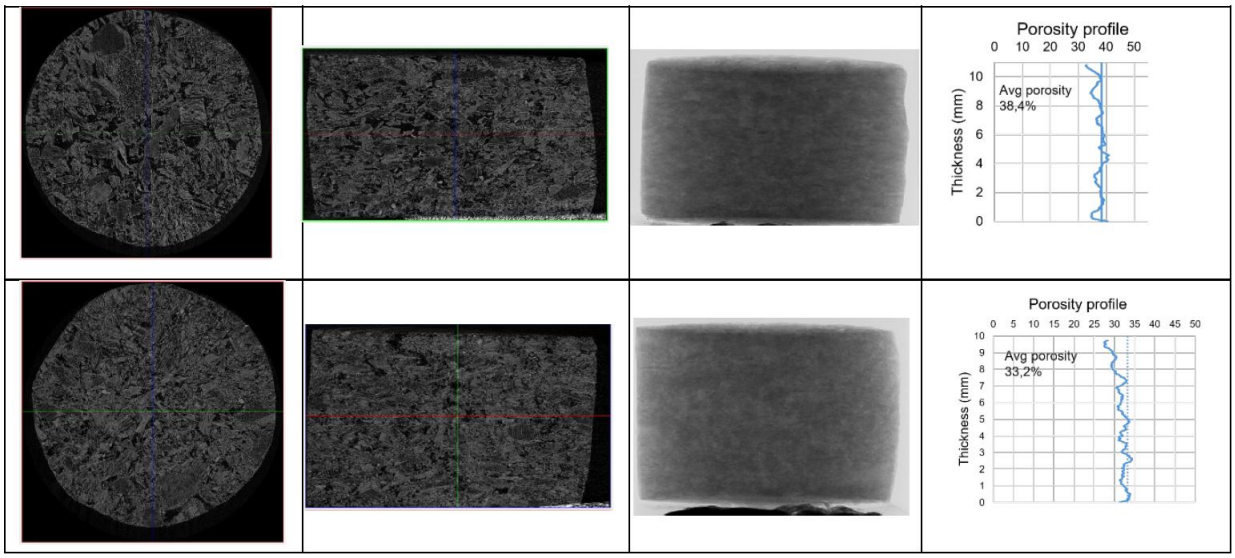

Figure 58. Cross and coronal x-ray sections, coronal radiograph and porosity profiles of panels using epoxy oil as binder. No bPCM sample in the first row. With 25% bPCM addition in the second row.

NI_NP sample in the first row of figure 59 results a homogeneous, extremely soft and light panel with very high mean pore size and porosity (the highest ones among those analysed). Very loose wood particles.
I_NP sample in the second row of figure 59 results a homogeneous, very soft and light panel with very high mean pore size and slightly lower porosity than NI_NP panel. Quite loose wood particles.
NI_P sample in the third row of figure 59 results as a soft and light board with high mean pore size and higher porosity in the middle than on top and bottom of the panel.
I_P sample in the fourth row of figure 59 results as a soft and light board with high mean pore size and just a pronounced higher porosity in the middle than on top and bottom of the panel.

Overall analyses of porosity profiles allow to argue that, except for the case of the lignin-based MW dried sample, pressing treatment introduces structural inhomogeneities inside the sample thickness. Pore size distribution outcomes shown in figure 60 suggest that, except for the Epoxy oil bonded particle boards, addition of bPCM produces presence of larger pores, probably due to a wood particle aggregation effect of such an addition. Addition of bioPCM to mycelium bonded particle boards produced also a secondary effect of a moderate compaction in the not pressed panel case.

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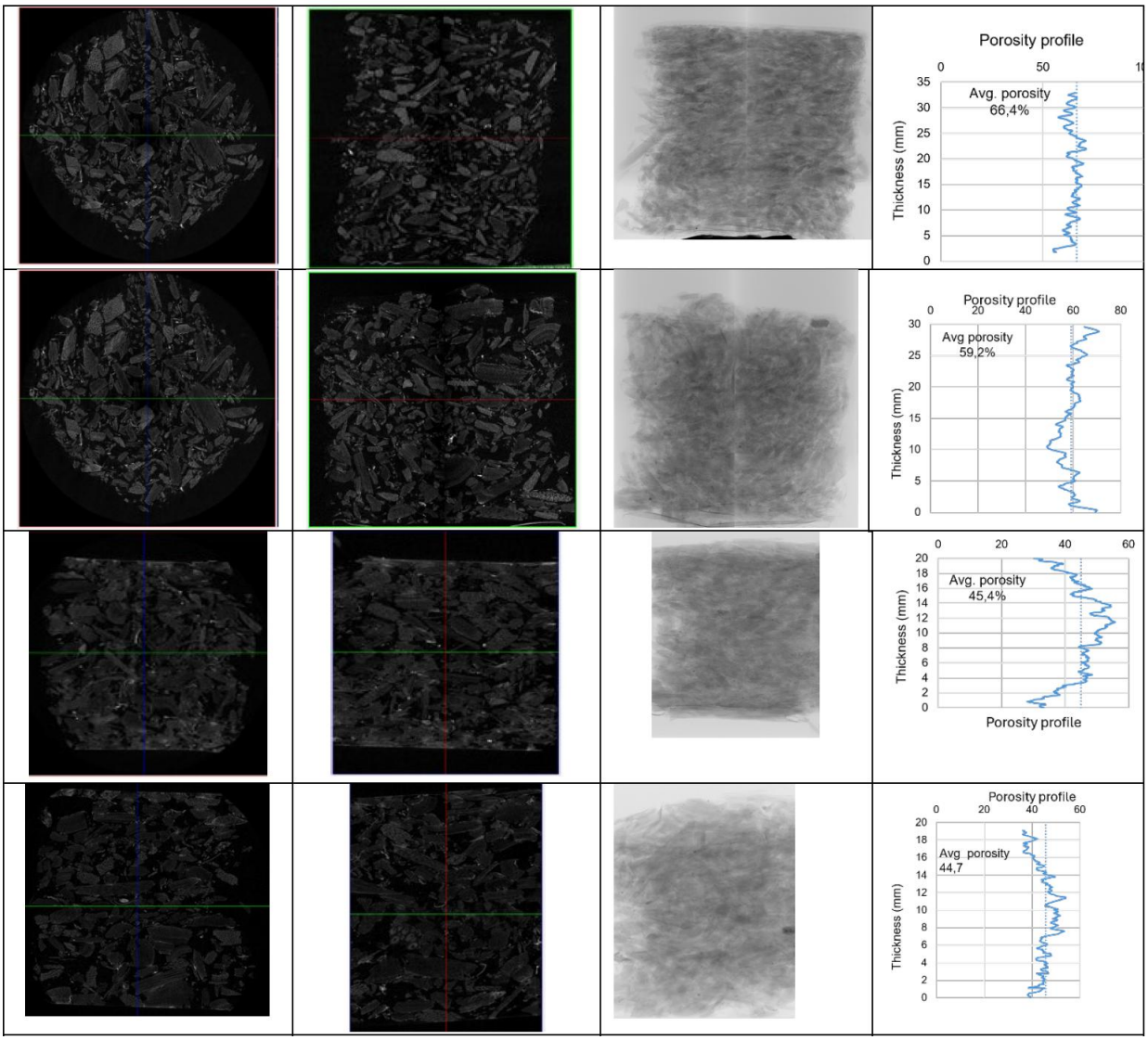

Figure 59 Cross and coronal x-ray sections, coronal radiograph and porosity profiles of panels using mycelium as binder. NI_NP sample in the first row. I_ NP sample in the second row. NI_P sample in third row. I_P sample in fourth row.

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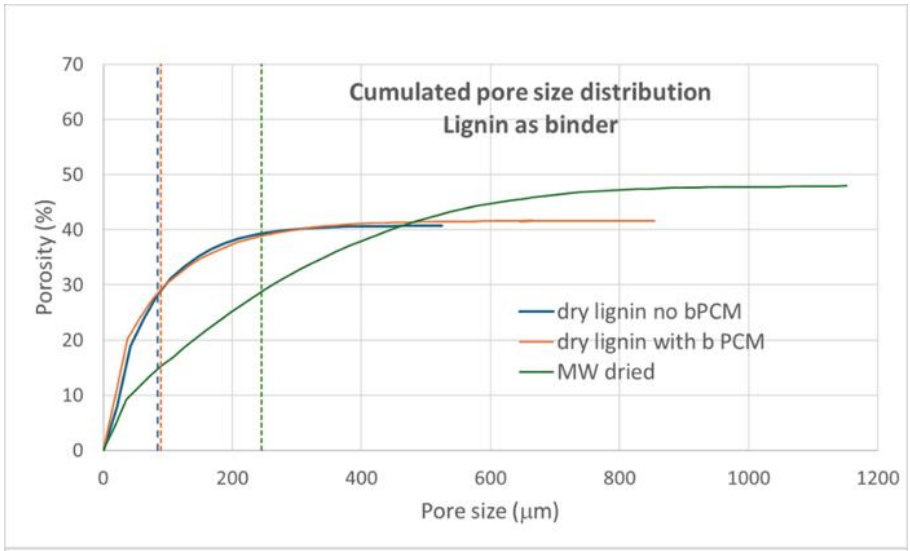

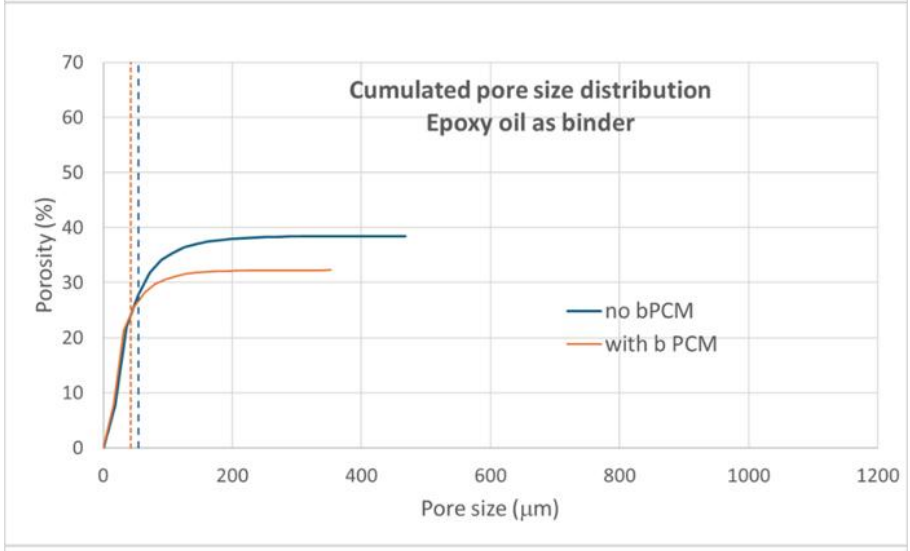

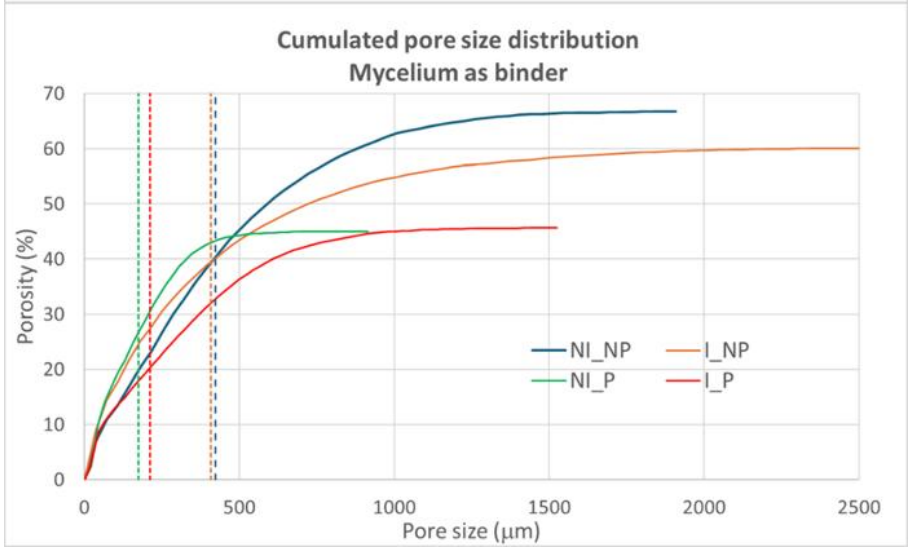

Figure 60 Cumulated pore size distributions of all investigated particle boards. Vertical dotted lines indicate position of mean pore sizes.

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Table 23 Average structural characteristics as measured by image analysis from X-ray micro-CT

Sample		Total porosity	Mean pore size	Surface density
Binder	**Treatment**	**(%)**	**(μm)**	**(1/mm)**
Dry lignin	no bPcM	40,7	83,8	25,4
Dry lignin	with bPCM	41,7	89,6	30,1
Lignin-based	MW dried	47,3	245,4	18,4
Epoxy oil	no bPcM	38,4	54,9	32,5
Epoxy oil	with 25% bPCM	33,2	42,4	35,0
Mycelium	NI-NP	66,4	422,7	11,7
Mycelium	I NP	59,2	407,2	14,1
Mycelium	NI-P	45,1	173,5	15,0
Mycelium	I_P	45,4	210,7	16,0

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5 Fire resistance of bio-composites

5.1 Fire resistance of bio-composites with linseed oil as binder

Wood-based bio-composites possess high thermal and fire sensitivity, which introduces a need of improved fire performance being as important as enhancing mechanical properties. This is important given their increasing practical use. This part of the project studies the resistance to fire of the bio-composites with bioPCM, bonded by the three binders (ELO, LS and mycelia). The flammability performance of the materials was evaluated, with particular focus on additional applications as surface treatments to enhance the composite's fire resistance.

5.1.1 State-of-the-art

Several conventional flame retardants based on halogen and inorganic materials are widely used; however, some of them, in particularly halogen flame retardants, are harmful to human's our health and environment, and thus, they should be restricted in use [1]. With fire protection of bio-composites in focus, significant number of fossil-derived [2] and biobased fire retardants [1, 3] have been reviewed and it is concluded that the flammability of bio-composites is a complex scientific challenge with a diversity of options but lack of a universal solution of the problem. The bio-matrix of the bio-composites is combustible and thus, demanding various approached to *reduce* the flammability and combustibility.

Regarding fire protection of bio-composites with integrated PCM, a scarcity of studies has been found due to fact that these materials have drawn increased research interest just in the recent years. A study [4] improved

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bulk PCMs with adding the flame retardants ammonium phosphate, melamine phosphate, hydromagnesite, magnesium hydroxide, and aluminum hydroxide. The authors found that fire retardancy was improved by adding hydromagnesite and magnesium hydroxide (both at 50 wt %) in the fatty acid mixtures. However, the fatty acid mixtures showed a clear reduction of the melting enthalpy by a factor of 3 when 50 wt % of the flame retardants were added.
Flammability of PCM-containing wood-based composites [5] was evaluated by additions of boric acid, clay and trimethoxymethylsilane coating. The thermal properties of the composites were unchanged by mixing the fire retardants in the composite, not to the bulk PCM. Interpreting the results of the study, it can be concluded that the tested materials can be indicatively classified in class E described in the standard EN 13501-1:2018, i.e., one class lower than the class of wood and wood materials. The best fire protection was achieved by adding *ca.* 50% clay to the composite.

5.1.2 Ethyl palmitate as bioPCM and need of fire protection

Ethyl palmitate is widely used not only as bioPCM but in other application (cosmetics, personal care, lubricants). According to the safety data sheet of Sigma-Aldrich, EP is not flammable liquid (https://www.sigmaaldrich.com/SE/en/sds/aldrich/w245100?srsltid=AfmBOooZ0euHmoAQ_Mo5a2HubcyROkWFeIk9ZtuXjuko-v8ePoLTVOva). However, it is *combustible* being composed of fatty acid esters.
Ethyl palmitate is classified as a combustible material with a slight fire hazard when exposed to heat or flame. Although not highly flammable, it can burn at elevated temperatures and form combustible mists that may become explosive. The primary hazards during a fire involve thermal decomposition, releasing carbon monoxide and acrid smoke. Core risk assessment data are found in the safety data sheet (https://datasheets.scbt.com/sc-205423.pdf)

- Flash point: >113°C.
- Autoignition temperature: No widely published specific data; typical of long-chain fatty esters, it generally requires high temperatures to self-ignite.
- Flammability class: OSHA Class IIIB (Combustible liquid).
- Hazardous decomposition: Burning releases carbon oxides (CO and CO_2) and irritating pyrolysis products.

Control and Mitigation Measures
- Extinguishing media: Use alcohol-resistant foam, dry chemical, carbon dioxide (CO_2), or water spray/fog. Avoid using solid water jets, which can scatter the burning liquid and spread the fire.
- Storage conditions: Store in a cool, dry, well-ventilated area away from strong oxidizers, heat, sparks, and open flames. Keep containers tightly closed when not in use.

EP requires sustained heat to ignite and can burn if exposed to an open flame, but it is not ignited at room temperature. EP has no REACH nr. as the substance or its uses are exempted from registration or the annual tonnage does not require a registration.

5.1.3 Approach

Based on the literature data, adding of fire retardants in the bulk of PCM alters drastically the thermal characteristics of the material and thus is found inappropriate. Another approach can be the treatment of the biomaterial with fire retardants, i.e., impregnation and drying of the wood particles and fibres prior to the impregnation with bioPCM. The approach introduces an extra step, which is demanding considering the energy consumption and time and thus, can influence the Life Cycle Cost Assessment (LCCA) determined in Deliverable 6.2.

Fire retardance of the bio-composites with ELO comprises test of external layer applied on the composite's surface. As shown in Deliverable 3.1, UV-curable coating formulated with ELO and triarylsulfonium hexafluoroantimonate salts (mixed) enabled low-temperature curing of ELO. The coating system is needed to prevent crystallization and/or leaching of the bioPCM but is compatible with a number of fire retardants, thus allowing incorporation of the fire retardant into the coating.

Melamine polyphosphate (MPP, the illustration below) and fire-resistant paper Fleme-Gard™ were tested alone or in combinations with two concentrations of MPP. Melamine polyphosphate (MPP) is a halogen-free

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intumescent flame retardant composed of melamine and polyphosphoric acid. It is widely used to provide fire resistance in engineered materials, coatings, and resins with no use of toxic halogens. MPP is active in both gas and condensed phases because of its nitrogen-phosphorus structure. During burning, melamine degrades and releases inert gases (nitrogen, ammonia, steam) that dilute oxygen and remove heat in the gas phase while the phosphoric acid forms an intumescent char layer that insulates against heat flux, blocks oxygen contact, and inhibits flammable gas formation in the condensed phase. MPP is an effective fire retardant [6]; however, the disadvantage of organo-phosphorus flame retardants is the need of large quantities, which influences negatively the polymer properties [2]. The amount of MPP in fire retardant coatings varies in the range of 10-30% w. [7].

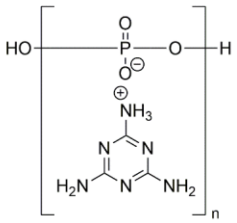

Figure 61 Formula of melamine polyphosphate (from [2])

Flame-Gard is unbleached kraft paper formulated without organo-halogens and free of tri-phenylphosphate. It is a commercial product of Ahlstrom (www.ahlstrom.com) that is aimed at improved flame retardancy of materials to ensure a smooth and uniform material surface with balanced strength properties.

5.1.4 Materials and methods

Fire classification of construction products and building elements in Europe is covered by the Euroclass system and used in the EU construction products framework and support CE marking when the rest of the product requirements are met. It splits fire performance into reaction to fire for products (standard EN 13501-1) and fire resistance for assembled building elements or structures (standard EN 13501-2). For construction products such as wall and floor coverings, insulation the Euroclasses are A1, A2, B, C, D, E, and F. Classes A1 and A2 are the non-combustible classes, illustrated by materials like gypsum boards with thin paper and mineral wool. Classes B and C comprise materials like gypsum boards with thick paper and fire-retardant wood with limited contribution to fire. Wood and wood-based products are placed in class D having acceptable but weaker fire performance. Class E meets only small flame test requirement and has d2 droplet classification while class F comprises materials with no classified fire performance. The classification has also two additional ratings of smoke class from s1 to s3 and droplet class from d0 to d2; s1 and d0 mean no smoke or droplets while s3 and d2 allows more. For example, surface materials, e.g., wallboards, placed on a wall might be classified as C-s1, d0, meaning they have limited contribution to fire, low smoke production, and no flaming droplets.

The standards ISO 5660-1:2015 and ISO 5660-1:2015/Amd1:2019 describe cone calorimeter method for measuring how a material behaves in fire, its heat release rate and dynamic smoke production rate. The Amendment from 2019 modifies the text by the option to add new material or make editorial corrections in ISO 5660-1. ISO 5660-1 provides data on heat release by the time, peak heat release rate, total heat release, smoke production, and mass loss rate for a test specimen exposed to a controlled heat flux. The method shows how quickly a product ignites, burns, and produces smoke under defined laboratory conditions. The advantages of testing according to ISO 5660-1 is the need of less test material and cost-effective testing than measuring simple burning item (SBI by the standard EN 13823) test.

ISO 5660-1 shows reaction-to-fire testing of construction products and materials when quantitative fire behaviour data is needed rather than a simple pass/fail result. It is often used in research, product development, and fire classification work. ISO 5660-1 does not classify the materials to the Euroclasses of itself, but it provides test data that may be used as part of broader fire-performance assessment. Peak heat release rate (pHRR) from the cone calorimeter method (ISO 5660-1) is not directly used as criterion in Euroclasses A1 to F, but it is highly correlated with the PHRR-based index used in EN 13501-1, called FIGRA (Fire Index Growth Rate), which is computed from an SBI test (EN 13719) rather than from cone calorimeter data.

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Research shows that for groups of products, average cone calorimeter HRR curves for each Euroclass; can be determined. In practice it means that the high Euroclasses A1, A2, and B have lower peak HRR, lower total heat release, and slower HRR rise when compared to Euroclass C, D, and E having higher peak HRR, higher total heat release, faster HRR rise. Thus, ISO 5661-1 is used to show *indicatively* the eventual classification of the materials in EN 13501-1.

Cone calorimetry (ISO 5660-1) tests samples with dimensions of 100 × 100 mm and thickness up to 50 mm that are subjected to a specific irradiance level. The surface of the sample is heated and starts to emit pyrolysis gases that ignite by a spark igniter. The emitted gases are collected in a hood and transported away through a ventilation system. The heat release is measured by measuring the oxygen concentration in the emitted smoke. The smoke production is measured continuously throughout the test with a laser system. The following parameters are measured: ignition properties, heat release rate (kW/m^2), total heat release (MJ/m^2), mass loss (g/s), effective net heat of combustion (MJ/kg) and smoke production rate (m^2/s). The value of toxic gases can also be measured by Fourier Transform Infrared Spectroscopy (FTIR).

Bio-composites made of wood particles (sawdust), mechanical pulp fibres and epoxidized linseed oil as a binder and two options of catalyst, i.e., oxalic (OA) and citric acid (CA) were processed to improve the fire resistance of the material. Both composites contain 55% wood particles impregnated with bioPCM (ethyl palmitate). Thus, the amount of pure bioPCM in the composite is 25%. The development of the samples is shown in detail in Deliverable 3.1. According to the ISO 5660-1, the tested bio-composites (Treatments 1-10 below) had dimensions of 11 × 100 × 100 mm with three replicates in each treatment group shown below:

1. Control composite with OA, uncoated
2. Composite with OA, coated
3. Composite with OA, coated, 20% melamine polyphosphate (MPP) in the coating
4. Composite with OA, coated, 40% melamine polyphosphate (MPP) in the coating
5. Composite with OA, fire-resistant paper, coated
6. Composite with OA, fire-resistant paper, coated, 20% MPP
7. Composite with OA, fire-resistant paper, coated, 40% MPP
8. Composite with CA, coated
9. Composite with CA, coated, 20% MPP
10. Composite with CA, coated, 40% MPP

The coating tested was described in Deliverable 3.1. It consists of ELO with either triarylsulfonium hexafluoroantimonate salts (mixed) or bis(4-methylphenyl) iodonium hexafluorophosphate to enable low-temperature curing of ELO. The coated surface was exposed to UV radiation by a LED lamp with a peak emission wavelength of 365 nm and a power of approximately 0.5 W/cm². The distance between the sample surface and the lamp was 3 cm. MPP was solved in the ELO at two concentrations of 20 and 40% and polymerized by UV-irradiation at the same wavelength.

5.1.5 Results and discussion

Descriptive statistic of the results from the tests are shown below. The description comprises mean values and standard deviations within parentheses.

Table 24 Descriptive statistics of results

Treatment	Heat release rate, kW/m^2	Total heat release, MJ/m^2	Mass loss, %	Time to ignition, s	Smoke production, m^2/m^2
1	499.96 (10.44)	156.34 (1.82)	62.0 (1.0)	7.3 (0.6)	1291.5 (63.2)
2	576.68 (7.98)	164.87 (3.29)	62.0 (1.0)	12.3 (1.2)	1433.6 (38.4)
3	554.13 (37.42)	156.60 (7.39)	57.7 (2.3)	18.0 (12.0)	1565.5 (45.2)
4	477.01 (22.28)	142.82 (0.91	55.3 (1.5)	16.0 (3.6)	1585.5 (59.4)
5	475.46 (28.42)	177.18 (9.83)	66.7 (0.6)	5.0 (1.0)	1595.5 (96.2)
6	548.78 (41.86)	158.95 (7.40)	57.7 (2.5)	6.3 (0.6)	1388.4 (72.7)
7	519.45 (55.39)	151.61 (4.89)	55.3 (4.7)	8.3 (0.6)	1446.5 (40.2)
8	494.04 (6.72)	159.95 (15.59)	63.3 (0.6)	12.7 (5.0)	1521.1 (311.5)
9	407.07 (18.11)	149.96 (11.27)	61.3 (0.6)	10.0 (4.0)	1400.9 (106.7)

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10	376.78 (10.97)	136.83 (5.30)	57.7 (2.0)	17.3 (1.2)	1524.6 (68.5)

For EN 13501-1:2019, these cone calorimeter data are only indicative. They are useful for ranking formulations, but they do not by themselves establish the Euroclass because the standard classification is based on the prescribed reaction-to-fire test framework, not ISO 5660-1 alone. From the observed values, the material set looks unlikely to support a high-performing class such as B or C, because the peak heat release rates are generally high and smoke production is substantial. A cautious qualitative indication would be that the better-performing formulations are likely in the lower Euroclass range rather than the top classes.

As a rule-of-thumb class limits for cone calorimeter tests at 50 kW/m^2, the following Table 25 can be used.

Table 25 Rule-of-thumb class limits for cone calorimeter tests at 50 kW/m^2

Euroclass	Time to ignition, s	Heat release rate (maximum), kW/m^2
B	≥ 40	≤ 100
C	≥ 30	≤ 180
D	≥ 15	≤ 250

The heat release maximum rate of all tested composites exceeds 250 kW/m^2, the coated composites with OA and CA containing 20 and 40% melamine polyphosphate (MPP) in the coating showed longer time to ignition, i.e., ≥ 15 s. The above proves the effect of MPP as fire retardant but the effective retention is probably higher than the two tested. Flame-Gard kraft paper formulated without organo-halogens and free of tri-phenylphosphate improves slightly the heat release rate but has negative or no effect on the time to ignition. The tested materials can be *indicatively* classified in Euroclass E, i.e., a class lower when compared to wood-based panels being in class D. The above is explained with the large amount of combustible bioPCM impregnated in the wood particles. The results of the study are similar to these found by [5].

To increase the Euroclass of the test materials to at least class D, more tests are planned. Commercial fire resistance paints will be tested, similar to the approach of SQIM when a fire-retardant (FR) coating system is applied.

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5.2 Fire resistance of bio-composites with lignin as binder

5.2.1 Material

Samples of hot-presses bioPCM-containing composites HP-TL01 and the bioPCM-deficient hot-pressed control HP-C01 were used for this test. Three 100 x 100 mm samples of each variant were climatised at 20 °C, 65 % relative humidity for several days before starting the analysis.

Table26 Material properties of BOKU samples supplied for cone calorimetry. Results of three replicates and the mean (x̄) are given for each variant.

		HP-C01				HP-TL01			
	Unit	1	2	3	x̄	1	2	3	x̄
Mass	g	69.75	65.25	70	68.3	83.18	81.24	81.06	81.83
Thickness	mm	10.3	10.3	10.3	10.3	10.3	10.3	10.3	10.3
Density	kg/m³	677.2	633.5	679.6	663	807.6	788.7	787.0	794.4

5.2.2 Cone calorimetry

Samples were analysed by cone calorimetry according to ISO 5660-1 on a cone calorimeter meeting the standards specifications (TCC 918, Netzsch, Selb). Samples were heated at 50 kW/m² with a 25 mm distance between sample and heater for a duration of 20 minutes.

Usually, a higher material density leads to longer time to ignition, but in this case, it is the other way around. The denser bioPCM samples ignited 13 s earlier than the control, which is probably attributed to the bioPCM, which can contribute to the formation of pyrolysis gases and consequently to ignition. Additionally, the boards containing bioPCM showed a longer time to flame off (744 s) compared to the control board (689 s).

bioPCM containing board (HP-TL01) showed a higher total heat release (THR), total heat release within the first 600 s (THR600), peak heat release rate (peak HRR) and maximum average rate of heat (MARHE) indicating a more intense reaction to fire.

Similarly, the total smoke production (TSP) was about 84 % higher in bioPCM containing boards (1083 m²/m²) compared to the control boards (587 m²/m²). Both materials were nearly converted during the test, with relative mass losses of 87.1 % (HP-C01) and 88.1 % (HP-TL01) (Table).

Table 27 Results of cone calorimeter analysis of BOKU samples subjected to a heat flux of 50 kW/m². Results of three replicates and the mean (x̄) are given for each variant.

		HP-C01				HP-TL01			
	Unit	1	2	3	x̄	1	2	3	x̄
Time to ignition	s	30	27	30	29	14	18	15	16
Time to flame off	s	640	647	689	659	751	748	733	744
THR	MJ/m²	91.8	86.2	94.2	90.7	140.6	138.2	144.0	140.9
THR600s	MJ/m²	70.1	65.7	72.5	69.4	101.0	101.9	103.6	102.1
Peak HRR	kW/m²	211.2	219.1	235.9	222.1	307.3	301.5	307.8	305.5
MARHE	kW/m²	139.2	140.4	144.8	141.5	231.8	229.1	229.7	230.2
TSP gesamt	m²/m²	613.4	561.2	586.8	587.1	1108.8	1105.7	1033.1	1082.5
TSP non-flaming phase	m²/m²	97.1	112.2	107.2	105.5	77.8	100.3	91.5	89.9
TSP flaming phase	m²/m²	516.2	448.9	479.7	481.6	1030.9	1005.4	941.6	992.6
Mass loss	%	86.9	86.9	87.4	87.1	88.1	88.0	88.1	88.1

The parameters for estimation of the fire classification, calculated from cone calorimeter data, are shown Table 17. As expected, the simulated values for THR600s and FIGRA0.4MJ are significantly higher for variant HP-TL01 than for variant HP-C01. The addition of bioPCM leads to an THR600s-increase from 28.4 MJ to

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46.6 MJ (+64%), while the FIGRA0.4MJ rises from 277 W/s to 609 W/s (+120%). Nonetheless, both variants are classified as Euroclass D-s1 according to the simulation.

Table 17Simulated parameters for Euroclass classification according to EN 1350-1, calculated from cone calorimeter data for BOKU panels. Results of three replicates and the mean (x̄) are given for each variant.

		HP-C01				HP-TL01			
	Unit	1	2	3	x̄	1	2	3	x̄
THR600s	MJ	28.3	27.6	29.3	28.4	46.5	45.8	47.5	46.6
FIGRA0.4MJ	W/sec	268.8	278.2	284.1	277.0	622.0	592.1	612.7	608.9
Simulated Euroclass	-	D-s1	D-s1	D-s1	D-s1	D-s1	D-s1	D-s1	D-s1

The values shown in Table 17serve solely to estimate the expected Euroclass. A binding classification according to EN 13501-1 is only possible on the basis of an SBI test according to EN 13823 in combination with the single flame test.

5.2.3 Single flame test

To further strengthen the simulated classification by cone calorimetry, a single flame test was conducted (based on EN ISO 11925-2). Due to limited sample availability the test was performed on cot-offs. The sample size (70 mm x 50 mm) therefore does not meet the specified standard dimensions of 90 mm x 250 mm. Samples were subjected to a flame for 30 s. No sustained ignition or flame spread was observed on any of the tested specimens (Figure). The results therefore suggest that the requirements of the single-flame test for classification in Euroclass D could be met. However, due to the specimen dimensions deviating from the standard, these tests do not replace a standardized test according to EN ISO 11925-2.

Figure 62 BOKU samples after subjecting to the single flame test. Left: bioPCM containing samples. Right: control samples without bioPCM.

5.3 Fire resistance of bio-composites with mycelia as binder

To evaluate the impact of the bio-composites on fire performance, four samples were treated using SQIM's standard fire-retardant (FR) coating system, which is currently applied to commercial SQIM panels. SQIM panels have previously achieved European fire classifications of B-s1, d0 for the Pluma line and B-s2, d0 for the Acoustic line products, according to EN 13823 (Single Burning Item, SBI). The objective of this assessment was to determine whether the incorporation of the bioPCM affected the fire behavior of the final product.

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CONTROL SAMPLE

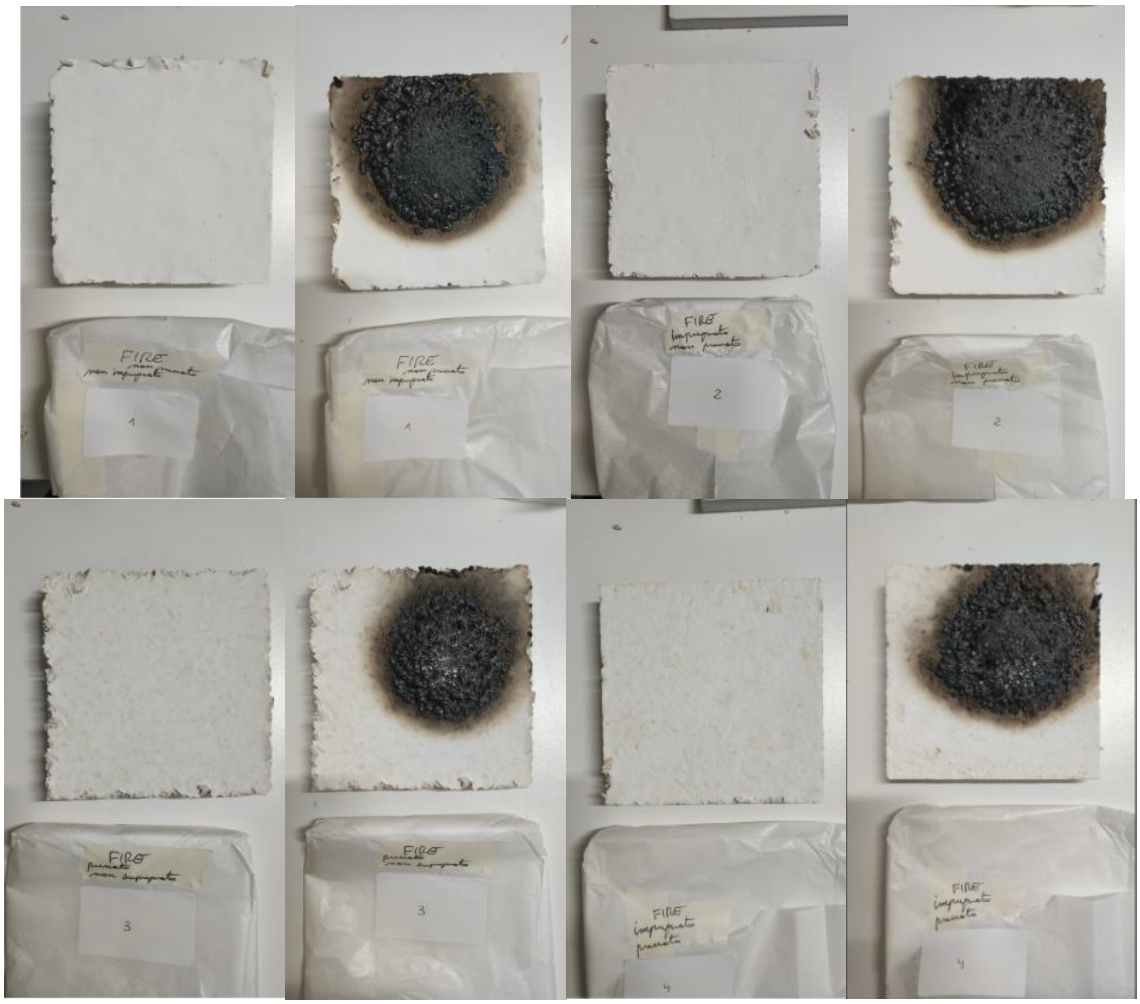

Figura 63 SQIM samples after subjecting to the single flame test. Belov: bioPCM containing samples, above: control samples without bioPCM.

The bio-composite samples were prepared using the standard SQIM FR coating and subjected to internal fire performance testing. Their behaviour was then compared against a reference control panel treated with the same fire-retardant system.
The results showed that the bio-composite samples exhibited fire performance comparable to that of the control panel. No significant differences were observed during the tests, and the samples demonstrated promising behaviour overall.

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Based on these preliminary evaluations, the incorporation of the bio-composites does not appear to adversely affect the fire performance of the final product when used in combination with SQIM's standard fire-retardant coating system.

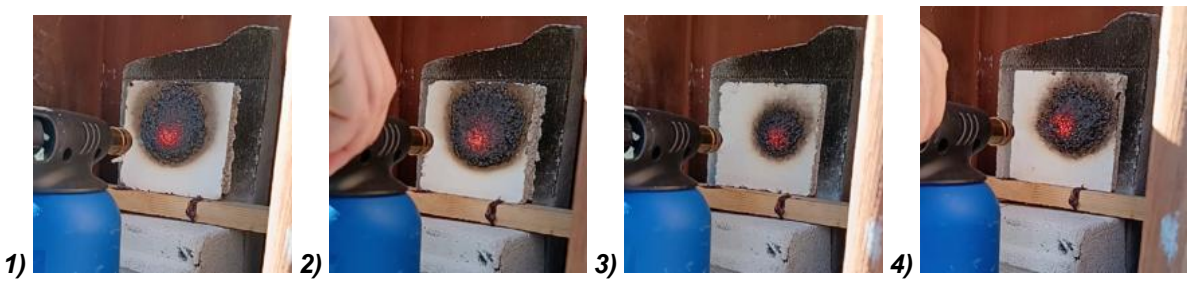

Figure 64 Fire performance test of internal SQIM FR coating

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