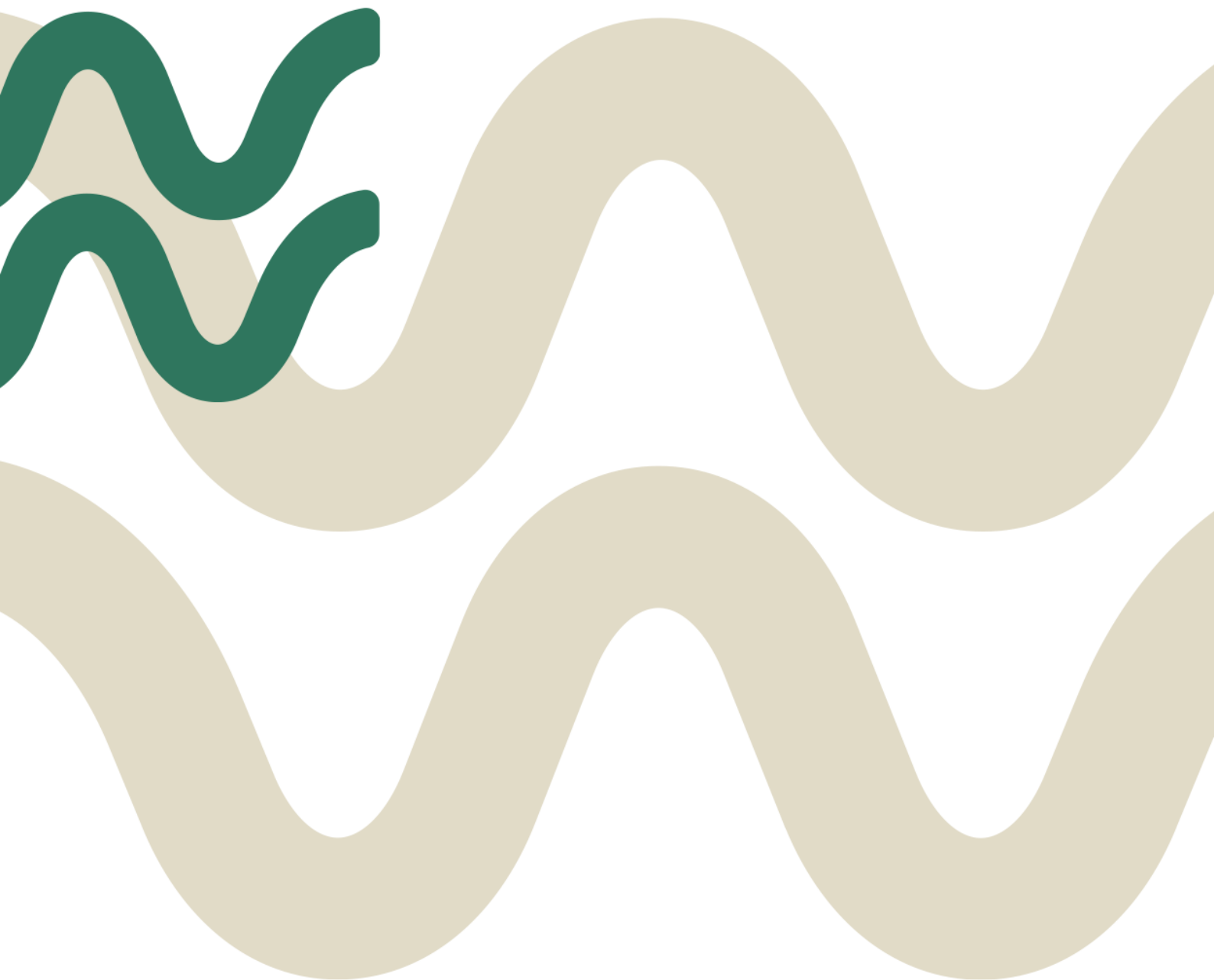




D5.3 – Samples manufactured with the green resins



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Executive Summary

This report summarizes the experimental work done in the period M12-M24 concerning the *Task 5.4: Development and characterization of MDF, plywood and CLT panels*. After the study and development of resins for use in plywood and MDF, containing lignin (supplied by BTG) in different percentages (phenol replacement), the first representative panel samples were prepared using these green resins.

Plywood boards were prepared using resins with a high lignin content (75% and 100% phenol replacement) and previously designed conditions in *Task 5.3: Resin application test and upscaling*. After production, the quality of plywood boards was tested by the Class 3 treatment (Europe Standard EN 314-2) for outdoor environment. To perform the test, the plywood boards were first cut into test pieces, half of this was used for the Class 3 treatment and the other half for mechanical testing. During the Class 3 treatment, many of the test pieces became unglued/delaminated, thus not reaching this class/standard. Also, the determined mechanical properties did not reach the values required by the standard. Different alternatives were tested in an endeavor to obtain a plywood product that meets the European standards established for plywood boards. These alternatives were a.o. adaptation of the dosage and the percentage of lignin in the resin. The resin using SPL2 lignin showed to be the most promising resin and was therefore chosen for the alternative study using a phenol substitution of 50%, 75% and 100%. The results showed that the plywood boards manufactured with the resin having a 50% phenol substitution achieved the values for Class 1 treatment with a dosage of 200 and 240 g/m² and with a curing factor of 1.5 min/mm.

The manufacturing of MDF boards has started using MDF resins with a phenol substitution of 25% by the four lignin's received from BTG. MDF boards obtained were characterized using European Standard UNE-EN 319. The results showed that the MDF boards did not reach the European Standard Internal Bond values in dry conditions. However, when the MDF were undergoing treatment for use in wet conditions, both the swelling percentage and the Internal Bond values reached the requirements of the Standard. The determination of

formaldehyde release of the formed MDF boards was also determined, indicating a high formaldehyde release content for all MDF boards.

The manufacture of MDF resins with a higher percentage of lignin will be carried out in the coming period their corresponding MDF boards will be manufactured and characterized.

Plywood manufactured samples

Different resins were developed with the focus on possible application in plywood and MDF industry. First of all, application testing was carried out to make a resin for plywood products. Foresa Tech has performed a reaction/procedure using a plywood reference resin in which different lignin samples provided by BTG *Lignin A = SPL1; Lignin B = SPL2; Lignin C = MP; Lignin D = LPL*, were tested as phenol substitutes in different amounts. The phenol substitution tested in the resins were 25%, 50%, 75% and 100%.

The first representative samples manufactured with plywood resins were obtained using resins with high lignin content, namely 75% and 100% of phenol substitution.

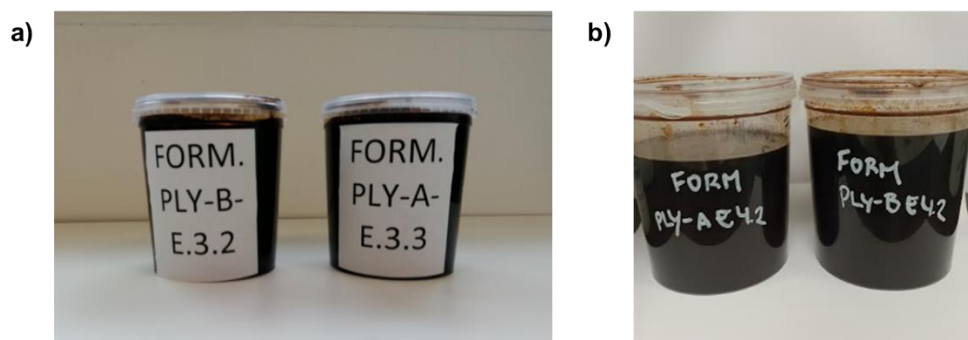


Figure 1. Example of two formulation of plywood resin with a phenol substitution of a) 75% lignin and b) 100% lignin

Development and characterization of plywood products

Foresa Tech has developed plywood panels using resins established in sub-task 5.3. Until now and to investigate the affordability of plywood forming, resins with a phenol substitution of 75% and 100% lignin were used.

For the development of plywood products, the following conditions have been tested:

- **TEMPERATURE:** Two different temperatures have been tested: 100 °C and 120 °C
- **DOSAGE** The coating can vary in dosage, leading to variations in the resistance and durability of the plywood according to the customer's needs. Taking this into account two different dosages were tested: 160 and 200 g/m².
- **CURE FACTOR:** Three different curing factors have been tested: 0.5; 1 and 1.5 min/mm.

First at all, the starting conditions were:

Temperature **100 °C**; Dosage **160 g/m²** and cure factor of **0.5 min/mm**

The results showed that plywood boards obtained at these conditions got broken or delaminated after the pressing process. Therefore, new conditions were applied:

Temperature **120 °C**; Dosage **200 g/m²** and cure factor of **0.5 min/mm; 1.0 min/mm; 1.5 min/mm;**

Under these conditions, it was possible to obtain plywood products after the press cycle and their subsequent characterization was carried out.



Figure 2. Plywood products a) obtained and b) unglued after pressing process.

a) Determination of glueing quality

Three classes are established for the quality of the glueing, which are based on its moisture resistance as indicated below:

- Class 1: Dry indoor environment

This type of glueing is suitable for normal indoor environmental conditions.

- Class 2. Covered outdoor environment.

This type of glueing is suitable for outdoor applications that will be protected and is able to withstand exposure to outdoor for a short period of time. It is also suitable for indoor applications when moisture conditions exceed those corresponding to the class 1 level.

- Class 3: Outdoor conditions

This type of glueing is designed to withstand long-term exposure to outdoor conditions.

To determine the quality of the glueing of the plywood boards, the specifications corresponding to the European Standard EN 314-2 were followed. The plywood boards obtained were cut according to the Standard to obtain the corresponding test pieces. To determine to which class the glueing quality belongs, each line of glue must meet the two criteria of average strength values and percentage of breakage, as shown below.

Table 1. Requirements for determination of glueing quality

Average f_v resistance (N/mm ²)	Average breakage rate
$0,2 \leq f_v \leq 0,4$	≥ 80
$0,4 \leq f_v \leq 0,6$	≥ 60
$0,6 \leq f_v \leq 1,0$	≥ 40
$1,0 \leq f_v$	Not expecifications

The determination of the Class requires different treatments for the test pieces, which are specified below in accordance with UNE-EN 314.

- **Class 1:** Test pieces are immersed in water for 24 hours at a temperature of (20 ± 3) °C.
- **Class 2:** The test pieces are immersed in boiling water for 6 hours and then cooled in water at a temperature of (20 ± 3) °C for at least 1 hour.
- **Class 3:** The test pieces are immersed in boiling water for 4 hours; then it is dried in an oven for 16 to 20 hours at a temperature of (60 ± 1) °C; then immersed in boiling water for 4 hours and finally cooled in water at (20 ± 3) °C for at least 1 hour.

First, the Class 3 treatment was carried out for the different test pieces. During the treatment, some of test pieces became unglued/delaminated, indicating that they could not withstand the process.

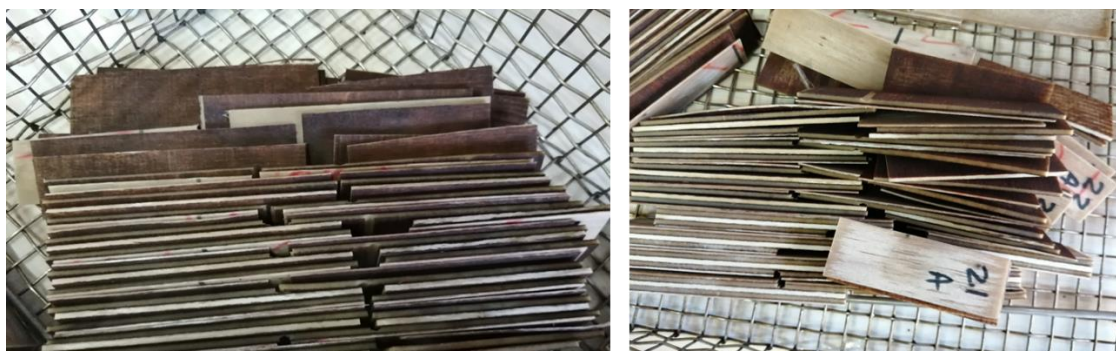


Figure 3. Test pieces unglued after Class 3 treatment.

For those test pieces that could be analyzed, the results for the plywood boards formed by the resins with 75% substitution showed that both lignin SPL1 and SPL2 as phenol substitutes have a slightly higher resistance value than the resin made with lignin LPL. Nevertheless, none of the plywood boards achieved the standard properties for Class 3 treatment. In the case of plywood board made by resin containing MP lignin, no plywood was obtained due to the delamination after the pressing process.

The following tables show the data obtained for those test pieces that could undergo treatment. Samples that have no value are those that became unglued/delaminated after Class 3 treatment or even after pressing process.

Table 2. Results with a press temperature of 120°C and dosage 200g/m² using 75% of substitution plywood resins.

RESULTS	Lignin	Replaced	1.5 min/mm		1.0 min/mm		0.5 min/mm	
			Line 1	Line 2	Line 1	Line 2	Line 1	Line 2
\bar{x} (PLY-E0)		0%	1.21	1.12	1.17	1.03	0.46	0.76
\bar{x} (PLY-AE3)	BTG NW SPL1	75%*	0.38	0.36	0.42	0.34	-	-
\bar{x} (PLY-BE3)	BTG NW SPL2	75%*	0.33	0.45	0.31	0.26	-	-
\bar{x} (PLY-CE3)	BTG NW LPL	75%*	0.22	0.27	0.40	0.34	-	-
\bar{x} (PLY-DE3)	BTG NW MP	75%*	-	-	-	-	-	-

In the case of the plywood board formed by the resins with 100% substitution, the results showed that most of the test pieces came out unglued after Class 3 treatment. As in the previous case, the plywood boards using the resin with MP lignin (100% substitution) became already delaminated after the press cycle and Class 3 treatment could not be carried out. Data was obtained for plywood boards using resin containing SPL1 and SPL2 lignin, however, these values are very far from reaching the requirements of the Standard.

Table 3. Results with a press temperature of 120°C and dosage 200g/m² using 100% of substitution plywood resins.

RESULTS	Lignin	Replaced	1.5 min/mm		1.0 min/mm		0.5 min/mm	
			Line 1	Line 2	Line 1	Line 2	Line 1	Line 2
\bar{x} (PLY-E0)		0%	1.21	1.12	1.17	1.03	1.17	1.21
\bar{x} (PLY-AE4)	BTG NW SPL1	100%*	0.21	0.19	-	-	-	-
\bar{x} (PLY-BE4)	BTG NW SPL2	100%*	0.24	0.23	-	-	-	-
\bar{x} (PLY-CE4)	BTG NW LPL	100%*	-	-	-	-	-	-
\bar{x} (PLY-DE4)	BTG NW MP	100%*	-	-	-	-	-	-

Given the results obtained, alternatives have been studied to obtain manufactured plywood boards that can achieve Standard requirements. These alternatives were:

- Process: Modification of the conditions of pressing process.
- Class category change: Plywood boards that reach standard values for Class II or Class I instead of Class III.
- Higher formaldehyde: Using resins with higher free formaldehyde so these resins will be more reactive.
- Percentage substitution change: Using resins with lower percentage of lignin to reach standard values.

The data shown above suggests that the use of SPL2 lignin in the resins gives rise to plywood boards whose resistance is greater compared to the rest of the lignins. SPL1 lignin could also be considered for this alternative study given its values are close to those obtained with SPL2 lignin. Therefore, to carry out the suggested alternatives, new resins were manufactured using SPL2 lignin as a substitute for phenol in amounts of 50%, 75% and 100%. In addition, two different dosages (200 g/m² and 240 g/m²) and three different treatments for Class 3, Class 2 and Class 1 were tested.

The test pieces obtained after Class 3 treatment did not reach the specifications of the Standard neither with a dosage of 200 g/m² nor with that of 240 g/m². Samples that have no value are those that became delaminated after Class 3 treatment or even after pressing process.

Table 4. Results obtained after Class 3 treatment.

CLASS 3 Treatment	Lignin	Replaced	1.5 min/mm		1.0 min/mm		0.5 min/mm	
			Line 1	Line 2	Line 1	Line 2	Line 1	Line 2
DOSAGE: 200 g/m²								
\bar{x} (PLY-E0)		0%	0.86	1.05	1.01	0.90	0.81	0.95
\bar{x} (PLY-BE2.2)	BTG NW SPL2	50%	0.53	0.67	0.25	0.17	-	-
\bar{x} (PLY-BE3.2)	BTG NW SPL2	75%	-	-	-	-	-	-
\bar{x} (PLY-BE4.2)	BTG NW SPL2	100%	-	-	-	-	-	-
DOSAGE: 240 g/m²								
\bar{x} (PLY-E0)		0%	1.16	0.99	1.01	1.11	1.10	0.91
\bar{x} (PLY-BE2.2)	BTG NW SPL2	50%	0.59	0.69	0.30	0.24	-	-
\bar{x} (PLY-BE3.2)	BTG NW SPL2	75%	0.19	0.13	-	-	-	-
\bar{x} (PLY-BE4.2)	BTG NW SPL2	100%	-	-	-	-	-	-

The test pieces obtained after Class 2 treatment did not reach the specifications of the Standard using a dosage of 200 g/m². However, when the dosage was increased to a 240 g/m², using the resin with 50% substitution and with a curing factor of 1.5 min/mm the values were close to reaching the requirements of the Standard (with average breakage rate of ≥ 40) Samples that have no value are those that became detached after Class 2 treatment.

Table 5. Results obtained after Class 2 treatment.

CLASS 2 Treatment	Lignin	Replaced	1.5 min/mm		1.0 min/mm		0.5 min/mm	
			Line 1	Line 2	Line 1	Line 2	Line 1	Line 2
DOSAGE: 200 g/m²								
\bar{x} (PLY-E0)		0%	1.11	0.89	0.99	0.92	0.97	1.00
\bar{x} (PLY-BE2.2)	BTG NW SPL2	50%	0.41	0.67	0.30	0.26	-	-
\bar{x} (PLY-BE3.2)	BTG NW SPL2	75%	0.22	0.32	0.14	0.11	-	-
\bar{x} (PLY-BE4.2)	BTG NW SPL2	100%	-	-	-	-	-	-
DOSAGE: 240 g/m²								
\bar{x} (PLY-E0)		0%	1.24	1.03	1.19	1.15	1.02	0.98
\bar{x} (PLY-BE2.2)	BTG NW SPL2	50%	0.63	0.72	0.31	0.28	-	-
\bar{x} (PLY-BE3.2)	BTG NW SPL2	75%	0.32	0.31	0.12	0.17	-	-
\bar{x} (PLY-BE4.2)	BTG NW SPL2	100%	-	-	-	-	-	-

In the case of the test pieces obtained after Class 1 treatment, for both dosages of, the resin with a 50% substitution and with a curing factor of 1.5 min/mm, the plywood boards did achieve the specification of the Standard. Even when using a curing factor of 1.0 min/mm it is possible to get close to the requirements suggested by the Standard.

Worth highlighting is that test pieces made with the resin having 75% substitution and applying a curing factor of 1.5 min/mm, can achieve the Standard values with average breakage rate of ≥ 40 .

Samples that have no value are those that became detached after Class 1 treatment.

Table 6. Results obtained after Class 1 treatment.

CLASS 1 Treatment	Lignin	Replaced	1.5 min/mm		1.0 min/mm		0.5 min/mm	
			Line 1	Line 2	Line 1	Line 2	Line 1	Line 2
DOSAGE: 200 g/m²								
\bar{x} (PLY-E0)		0%	1.01	1.41	1.34	1.32	1.26	1.04
\bar{x} (PLY-BE2.2)	BTG NW SPL2	50%	0.97	0.80	0.54	0.58	0.41	0.39
\bar{x} (PLY-BE3.2)	BTG NW SPL2	75%	0.56	0.53	0.28	0.31	0.13	0.10
\bar{x} (PLY-BE4.2)	BTG NW SPL2	100%	0.3	0.24	-	-	-	-
DOSAGE: 240 g/m²								
\bar{x} (PLY-E0)		0%	1.54	1.58	1.42	1.46	1.29	1.37
\bar{x} (PLY-BE2.2)	BTG NW SPL2	50%	0.80	0.86	0.68	0.57	0.38	0.29
\bar{x} (PLY-BE3.2)	BTG NW SPL2	75%	0.62	0.61	0.24	0.29	-	-
\bar{x} (PLY-BE4.2)	BTG NW SPL2	100%	0.17	0.12	-	-	-	-

MDF manufactured samples

During the period M12-M24, studies have started on the development of MDF resins varying the percentage (phenol substitution) of the different lignin's provided by BTG: *Lignin A = SPL1*; *Lignin B = SPL2*; *Lignin C = MP*; *Lignin D = LPL*.

The first tests were carried out replacing 25% phenol in the final MDF resin by the different lignin's. Complete characterization was performed and given in deliverable 5.1-2, MDF boards have been produced by applying the resins.

Development and characterization of plywood products

Foresa Tech has developed MDF boards using resins established in sub-task 5.3. Until now and to investigate the affordability of MDF forming, resins with a phenol substitution of 25% by lignin were used.

For the development of MDF products, the following conditions have been carried out:

- **TEMPERATURE:** 220°C
- **PERCENTAGE OF RESIN USED:** 15%.
- **CURE FACTOR:** 20 s/mm

Under these conditions, it was possible to obtain MDF products, although some of the boards came out broken after the press cycle, as can be seen in the pictures below.

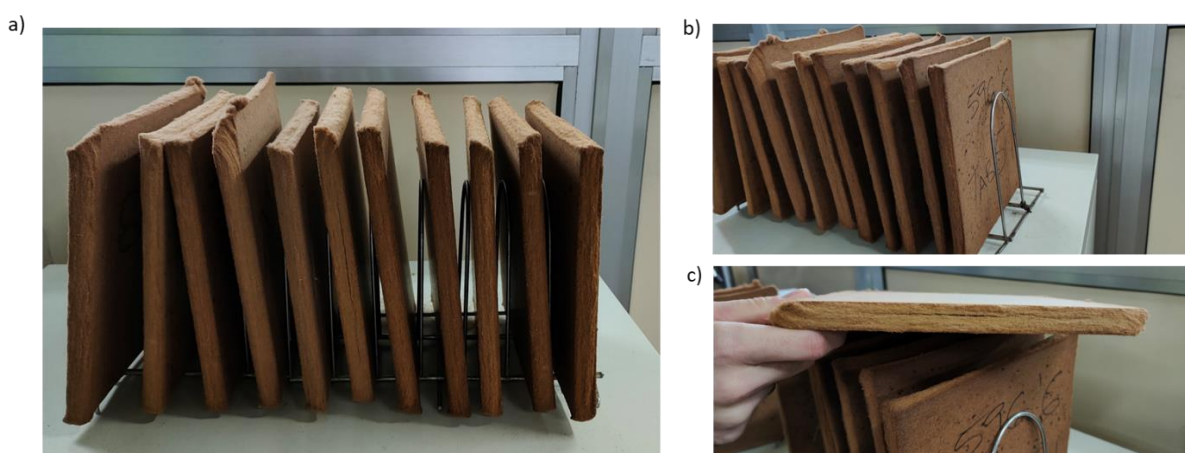


Figure 4. MDF boards a), b) obtained and c) panels broken after pressing process with lignins substituted with 25%.

a) Determination of tensile strength (internal bond)

Determination of the different properties of the MDF board(s) was carried out by following European Standard that regulates the determination of tensile strength perpendicular to the plane of the board (Internal Bond) UNE-EN 319. The boards were sanded to a final thickness of 16 mm and the sampling of test pieces was carried out by cutting square shapes with a side length of 50 ± 1 mm.

The MDF boards manufactured must comply with the general requirements of Standard EN 622-5. The table below shows the required properties of MDF boards (thickness of 16 mm) used in dry and wet environments.

Table 7. Requirements of boards for use in dry environments and wet environments (EN 622-5).

Property	Method	Units	Thickness ranges
			>12 at 19
Dry environment			
Swelling in thickness 24h	EN 317	%	12
Internal Bond	EN 319	N/mm ²	0.55
Wet environment: After Boil test			
Swelling in thickness 24h	EN 317	%	8
Internal Bond	EN 319	N/mm ²	0.12

As can be seen in the following table, the reference board meets the requirements of the Standard. However, the rest of the boards do not reach the Internal Bond value of the reference. It should be noted that the board manufactured with the resin using SPL2 lignin has an Internal Bond value close to the Standard values indicating the possibility of achieving the requirements of the Standard.

Table 8. Results of internal bonding determination

IB determination	Lignin	Replaced	Density (Kg/m ³)	IB (N/mm ²)
\bar{x} (MDF-board- E0)			723	0,55
\bar{x} (MDF-board-AE1)	BTG NW SPL1	25%	727	0,31
\bar{x} (MDF-board-BE1)	BTG NW SPL2	25%	696	0,51
\bar{x} (MDF-board-CE1)	BTG NW LPL	25%	-	-
\bar{x} (MDF-board-DE1)	BTG NW MP	25%	709	0,45

To determine both the percentage of swelling and absorption of the formed MDF boards, the test pieces were immersed in water at room temperature for 24 hours. Considering thickness and weight before and after the treatment of the different test pieces, it is possible to determine the swelling and absorption properties respectively.

The results obtained for the different boards are within the requirements of the Standard and are shown in the following table.

Table 9. Results of swelling treatment.

Swelling Treatment	Lignin	Replaced	Density (Kg/m ³)	Swelling 24h (%)	Abs 24h (%)
\bar{x} (MDF-board- E0)			722,85	3,47	16,90
\bar{x} (MDF-board-AE1)	BTG NW SPL1	25%	704,01	3,46	15,92
\bar{x} (MDF-board-BE1)	BTG NW SPL2	25%	669,34	3,83	15,96
\bar{x} (MDF-board-CE1)	BTG NW LPL	25%	-	-	-
\bar{x} (MDF-board-DE1)	BTG NW MP	25%	714,89	2,64	13,48

The moisture resistance of the boards manufactured by the dry process for use in wet environments was carried out by EN 1087-1: *Determination of moisture resistance-Part 1: Boil test*. This treatment was carried out by immersing the test pieces in boiling water for 2 h. After this time, they were cooled in water for 1 hour and finally, test pieces were placed in an oven at 70°C for 16 hours. After treatment, the internal bonding and swelling percentage were determined according to UNE-EN 319.

The internal bond obtained after boil test for the different MDF test pieces meet the requirements of the Standard with an average value of 0.29 N/mm² compared to 0.12 N/mm² of the Standard. Regarding the percentage of swelling, the results obtained differ a little from the requirements of the standard. The results obtained are given in the table below.

Table 10. Results of V-100 treatment.

V-100 Treatment	Lignin	Replaced	Density (Kg/m ³)	V-100 Swelling (%)	V-100 (N/mm ²)
\bar{x} (MDF-board- E0)			747,861	5,26	0,42
\bar{x} (MDF-board-AE1)	BTG NW SPL1	25%	756,011	9,12	0,27
\bar{x} (MDF-board-BE1)	BTG NW SPL2	25%	685,475	9,83	0,31
\bar{x} (MDF-board-CE1)	BTG NW LPL	25%	-	-	-
\bar{x} (MDF-board-DE1)	BTG NW MP	25%	754,657	5,86	0,29

b) Determination of formaldehyde release

Determination of formaldehyde release in MDF boards was determined using the extraction method, also called the perforator method by the standard UNE-EN ISO 12460-5:2015. Formaldehyde is extracted from the test pieces using boiling toluene, and then transferred into distilled water. The formaldehyde contained in the aqueous solution is determined photometrically using the acetyl-acetone method.

To carry out the extraction method in the perforator, approximately 110 g of test pieces are weighed and placed in a round-bottom flask along with 600 mL of toluene. Next, 1000 mL of distilled water is introduced into the perforator and connected to the round bottom flask with the perforator and condenser. The extraction is carried out for 2 hours, from when it starts to boil and then cools for 1 hour.

After this time, all the water contained in the perforator is collected along with the water used for rinsing and placed in a 2000 mL flask for subsequent determination.

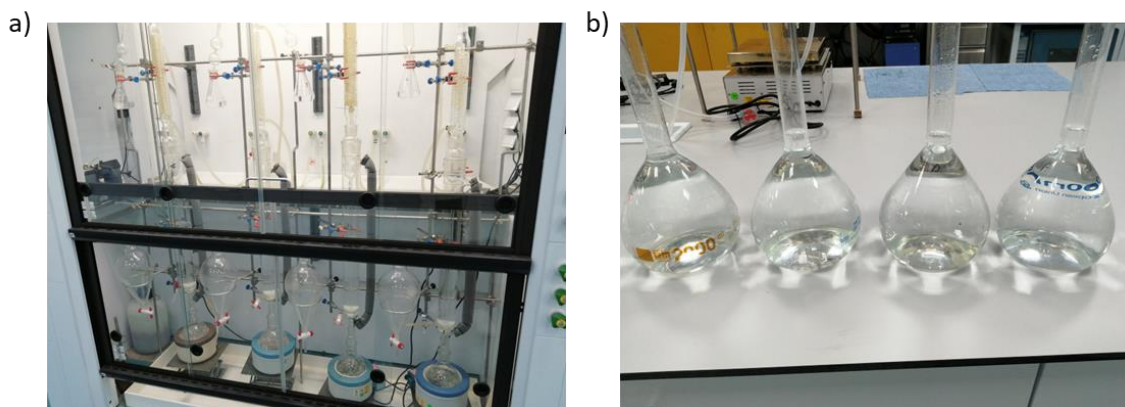


Figure 5. a) Trial in progress using perforator method for MDF samples. b) Aqueous solution obtained after perforator method

The determination of this is based on the Handtzensch reaction, in which formaldehyde in aqueous solution reacts with ammonium ions and acetyl-acetone to form diacetyldihydroxymethyloluril (DDL). DDL has a maximum absorption at 412 nm and its value is determined photometrically.

The formaldehyde content or perforator values is expressed in mg of formaldehyde per 100 g of dry board. Perforator values are applicable to boards conditioned to a moisture content

of 6.5%. In the case of boards with different humidity, the perforator value must be multiplied by the factor F.

For boards whose humidity is between $4\% \leq H \leq 9\%$:

$$F = -0.133 H + 1.86$$

For boards whose humidity is between $H < 4\%$ and $H > 9\%$:

$$F = 0.636 + 3.12e^{(-0.346H)}$$

Due to MDF board CE1 made by the resin with 25% of substitution using LPL lignin was broken after the press cycle, determination of formaldehyde release could not be carried out. As can be seen in the following table, the results obtained showed a high formaldehyde release content for all test pieces.

Table 11. Results obtained for determination of formaldehyde release.

Formaldehyde release	Lignin	Replaced	Humidity %	Factor F	Fomaldehyde content (mg/100mg)	Fomaldehyde content 6.5%
\bar{x} (MDF-board- E0)			8.39	0.743	16.52	12.28
\bar{x} (MDF-board-AE1)	BTG NW SPL1	25%	7.73	0.831	63.64	52.90
\bar{x} (MDF-board-BE1)	BTG NW SPL2	25%	7.20	0.902	63.35	57.19
\bar{x} (MDF-board-CE1)	BTG NW LPL	25%	-	-	-	-
\bar{x} (MDF-board-DE1)	BTG NW MP	25%	9.63	0.847	63.58	53.85

Conclusion

D5.3 describes the procedure and characterization of plywood and MDF boards development in Task: “5.4: Development and characterization of MDF, plywood and CLT panels” with the resins obtained in the Task “5.3: Resin application tests and upscaling”.

Plywood boards were prepared by resins having a high percentage of lignin (75% & 100% substitution) and the quality of the glueing was determined. The results showed that resin using SPL2 lignin was the most promising. Nevertheless, many plywood boards produced did not

reach the values required by the Standard or became unglued/delaminated after undergoing the treatment for outdoor environment (Class 3). Therefore, different alternatives such as a.o. adaptation of the dosage and the amount of lignin in the resin were tested in an endeavor to obtain a plywood product that did achieve the characteristics of the European Standard. Testing these alternatives resulted in plywood boards that achieve the values of Class 1 treatment. The boards were manufactured with a resin using SPL2 lignin, 50% phenol substitution, a dosage of 200 and 240 g/m² and with a curing factor of 1.5 min/mm.

The manufacturing of MDF boards was done by using a resin with 25% of phenol substitution by the four lignin's received from BTG. The characterization of the MDF manufactured showed that values obtained for the mechanical properties did not reach the Standard Internal Bond values in dry conditions. However, when the MDF boards were undergoing testing to determine their use in wet conditions, both the swelling percentage and the Internal Bond values reached the requirements of the European Standard. The determination of formaldehyde release of the formed MDF boards was also carried out, indicating a high formaldehyde release content for all MDF boards. The manufacture of MDF resins with a higher percentage of lignin will be carried out in the coming period in the *Task "5.3: Resin application tests and upscaling"* as well as their corresponding MDF boards in the *Task "5.4: Development and characterization of MDF, plywood and CLT panels and 5.5: Development of a fully sustainable MDF/plywood product"*.

The logo consists of two stylized, overlapping wave shapes in a golden-brown color, positioned to the left of the text.

NewWave



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