



## D5.2 - Development of new plywood resins



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## Executive Summary

This report summarizes the experimental work performed in the period M12-M24 concerning *Task 5.3: Resin application tests and upscaling*. A full development of plywood and MDF resins will be carried out using lignin samples provided by BTG: *Lignin A = SPL1; Lignin B = SPL2; Lignin C = MP; Lignin D = LPL*.

These four lignin's were characterized in *Task "5.1 Lignin characterization"*. They have been characterized by measuring the necessary parameters/properties for the implementation and subsequent replacement (phenol) in plywood and MDF resins. In the subsequent data evaluation, the two important points of interest were: What are the actual properties of the lignin materials and how will they behave when introduced into the resin recipe. The parameters that have been analysed were a.o. the water content, density, viscosity, pH, phenol content, elemental analysis (C, H, N) and metal analysis. Analyses have also been carried out to understand the reactivity, e.g. by applying thermal degradation testing, differential scanning calorimetry- and curing ramp measurements. Worth mentioning was that during the analysis and resin preparation all lignin sample showed good homogeneous properties.

After the lignin's were analyzed, their use in a general resin recipe was verified. The substitution of 25%, 50% and 75% of phenol in the resin was tested with each of the lignin samples. All the reactions could be carried out without problems in the polymerization, except for the 75% replacement with the SPL samples. At 75% replacement, the SPL samples generated a too high viscosity in the reactor, preventing polymerization. Once the screening tests were performed, resin systems were developed for plywood and MDF applications as described in this report.

The plywood resin development was done by replacing phenol with the different lignin's in amounts of 25%, 50%, 75% and even 100%. As the percentage of lignin (phenol substitution) increased in the final resin, more time was required to achieve complete dissolution of the lignin. A complete determination of the physical & chemical properties for the different

plywood resins was carried out by measuring a.o. the density, viscosity, pH, % of (free) phenol and (free) formaldehyde. Moreover, analyses have also been carried out to better understand the reactivity, e.g. by applying thermal degradation testing, differential scanning calorimetry- and curing ramp measurements. The results showed a high content of free formaldehyde in the resins with a phenol replacement of 75%. The lignin's are less reactive than phenol and therefore some formaldehyde remains unreacted in the resin system. To reduce the content of free formaldehyde in the final resins, the recipe was modified resulting in a reduction of approximately 38% of free formaldehyde. Resins production with 100% of phenol substitution were produced using this modified recipe. The developed resins will be used in *Task 5.4: Development and characterization of MDF, plywood and CLT panels*.

Concerning the MDF resins, the synthesis started with a recipe applying a phenol substitution of 25% using the four different lignin's. A complete characterization including viscosity, density, pH, hardening time, solid content, free formaldehyde, and phenol content was carried out. Analyses have also been carried out to understand the reactivity, e.g. by applying thermal degradation testing, differential scanning calorimetry- and curing ramp measurements. A complete characterization helps to determine the capacity of a resin for subsequent use in the manufacturing of MDF boards. All MDF resins analyzed showed a high content of free formaldehyde. Due to this, the recipe will have to be modified to reduce the presence of free formaldehyde. The development of MDF resins with a higher lignin percentage is ongoing. The developed resins will be used in *Task 5.4: Development and characterization of MDF, plywood and CLT panels*

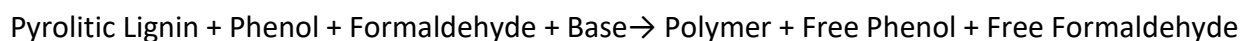
## Substitution of phenol for bio-phenol in resins

In the previous delivery D5.1, Foresa Technologies was able to make a wide range of characterization of the different lignin's provide by BTG: *Lignin A = SPL1*; *Lignin B = SPL2*; *Lignin C = MP*; *Lignin D =LPL*. Moreover, it was possible to make a reaction of a generic reference resin with different degrees of phenol substitution, 25%, 50% and 75%. However, it was not possible to obtain the 75% substitution for the case of the SPL samples. This issue was due to the fact that SPL samples generated a too high viscosity in the reactor, preventing polymerization.

This report presents the development and the study of a new formulation resin for plywood and MDF application following the scheme below:



And then, the modification of the plywood/MDF recipe where part or all the phenol is replaced by pyrolytic lignin:



## Plywood resins

A specific plywood standard recipe was synthesized that allows phenol to be replaced by different percentages of pyrolytic lignin. Four different degrees of substitution were applied, namely: 25%, 50%, 75% and 100%.

It was found that in the case of 75% substitution the content of free formaldehyde obtained was too high. Therefore, in an endeavor to reduce the percentage of free formaldehyde, the recipe was modified for resins with a high phenol substitution of 75% and 100%.

## Characterization resins

In the table below, the physical & chemical properties of the plywood resins developed are shown. For the resins all analysis were performed in trifold, the average value for each property is given in the table.

Table 1. Characterization resins [Ht (Hardening time); SC (solid content); FF (free formaldehyde); FP (Free phenol)]

	Lignin	Replaced	$\eta$ , cP (25°C)	$\rho$ , g/cm <sup>3</sup> (25°C)	pH (25°C)	H t, min	SC, %	Miscibility	FF, %	FP, %
$\bar{x}$ (E0)		0%	278	1,211	11,8	29,8	46,4	1000	0,06	0,001
$\bar{x}$ (AE1)	BTG NW SPL1	25%	317	1,213	12,0	35,2	46,0	1000	0,26	0,033
$\bar{x}$ (BE1)	BTG NW SPL2	25%	354	1,210	11,9	42,0	47,0	1000	0,18	0,05
$\bar{x}$ (CE1)	BTG NW MP	25%	327	1,225	11,8	41,8	47,8	1000	0,30	0,043
$\bar{x}$ (DE1)	BTG NW LPL	25%	295	1,205	11,0	38,4	47,0	1000	0,53	0,053
$\bar{x}$ (AE2)	BTG NW SPL1	50%	351	1,208	11,6	31,5	47,4	1000	0,70	0,050
$\bar{x}$ (BE2)	BTG NW SPL2	50%	373	1,208	11,4	32,1	47,7	1000	0,68	0,053
$\bar{x}$ (CE2)	BTG NW MP	50%	334	1,212	11,4	25,2	49,4	1000	0,64	0,031
$\bar{x}$ (DE2)	BTG NW LPL	50%	203	1,207	11,3	28,1	49,0	1000	0,53	0,051
$\bar{x}$ (AE3)	BTG NW SPL1	75%	329	1,206	11,1	30,8	49,6	1000	1,53	0,004
$\bar{x}$ (AE3.M)	BTG NW SPL1	75%	240	1,194	11,3	53,6	43,2	1000	0,47	0,004
$\bar{x}$ (BE3)	BTG NW SPL2	75%	399	1,204	10,4	34,6	47,7	1000	1,51	0,004
$\bar{x}$ (BE3.M)	BTG NW SPL2	75%	303	1,186	11,4	58,7	47,4	1000	0,40	0,023
$\bar{x}$ (CE3)	BTG NW MP	75%	353	1,214	11,4	45,4	52,1	1000	1,19	0,003
$\bar{x}$ (CE3.M)	BTG NW MP	75%	341	1,196	11,4	39,0	48,4	1000	0,45	0,003
$\bar{x}$ (DE3)	BTG NW LPL	75%	283	1,195	10,5	94	50,9	1000	0,95	0,023

$\bar{x}$ (DE3.M)	BTG NW LPL	75%	245	1,191	11,1	57,3	46,5	1000	0,49	0,017
$\bar{x}$ (AE4.M)	BTG NW SPL1	100%	305	1,193	10,8	57	48,9	1000	0,34	0.004
$\bar{x}$ (BE4.M)	BTG NW SPL2	100%	485	1,186	10,3	61	49,5	1000	0,25	0,002
$\bar{x}$ (CE4.M)	BTG NW MP	100%	399	1,207	11,4	50	49,7	1000	0,47	0.003
$\bar{x}$ (DE4.M)	BTG NW LPL	100%	384	1,198	11,5	58	47,8	1000	0,51	0.0021

### Viscosity

In the graphs collected, it is observed that in all plywood resins, the final viscosities were in the same range of 250 – 350 cP. This is normal because all resins were made with the same recipe and polymerization stops in the same viscosity range.

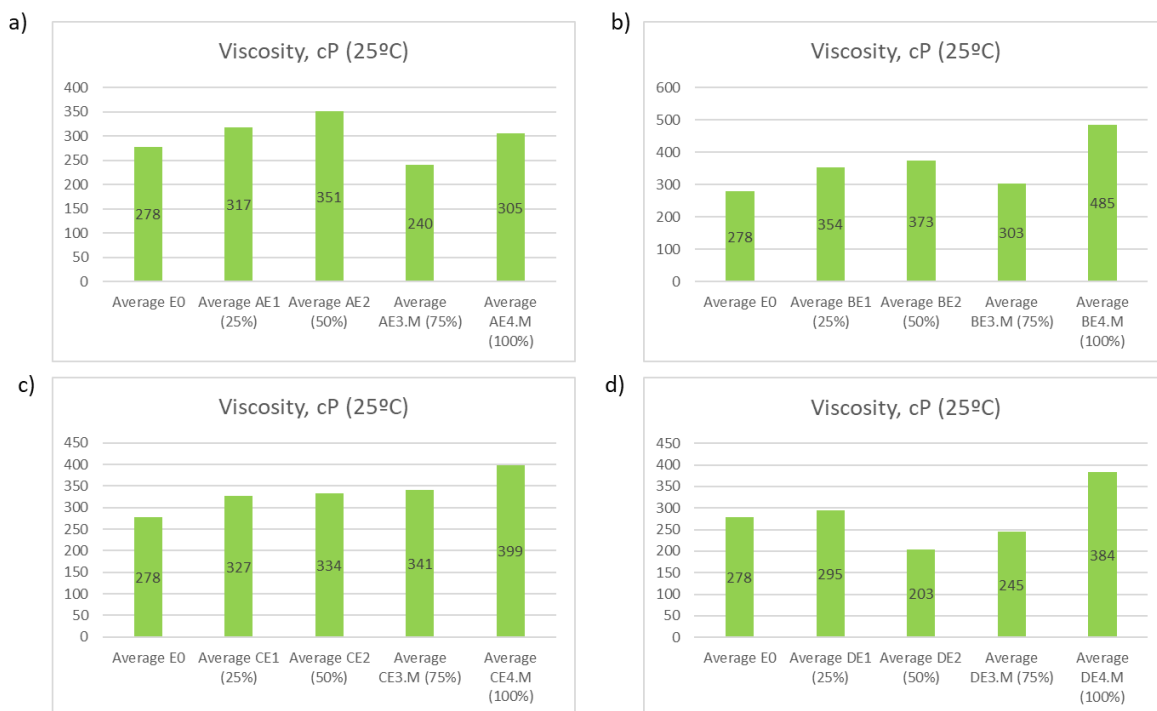


Figure 1. Viscosity of a) reference resin (E0), lignin BTG NW SPL1 resin replacing 25% (AE1), 50% (AE2), 75% modified (AE3.M) and 100% modified (AE4M); b) reference resin (E0), lignin BTG NW SPL2 resin replacing 25% (BE1), 50% (BE2), 75% modified (BE3M) and 100% modified (BE4M); c) reference resin (E0), lignin BTG NW LPL resin replacing 25% (CE1), 50% (CE2), 75% modified (CE3M) and 100% modified (CE4M); d) reference resin (E0), lignin BTG NW MP resin replacing 25% (DE1), 50% (DE2), 75% modified (DE3M) and 100% modified (DE4M).

Density

As can be observed in the following graphs, no significant changes were detected in the densities of the different samples. In all resins characterized, the density obtained was around 1.200 g/cm<sup>3</sup>.

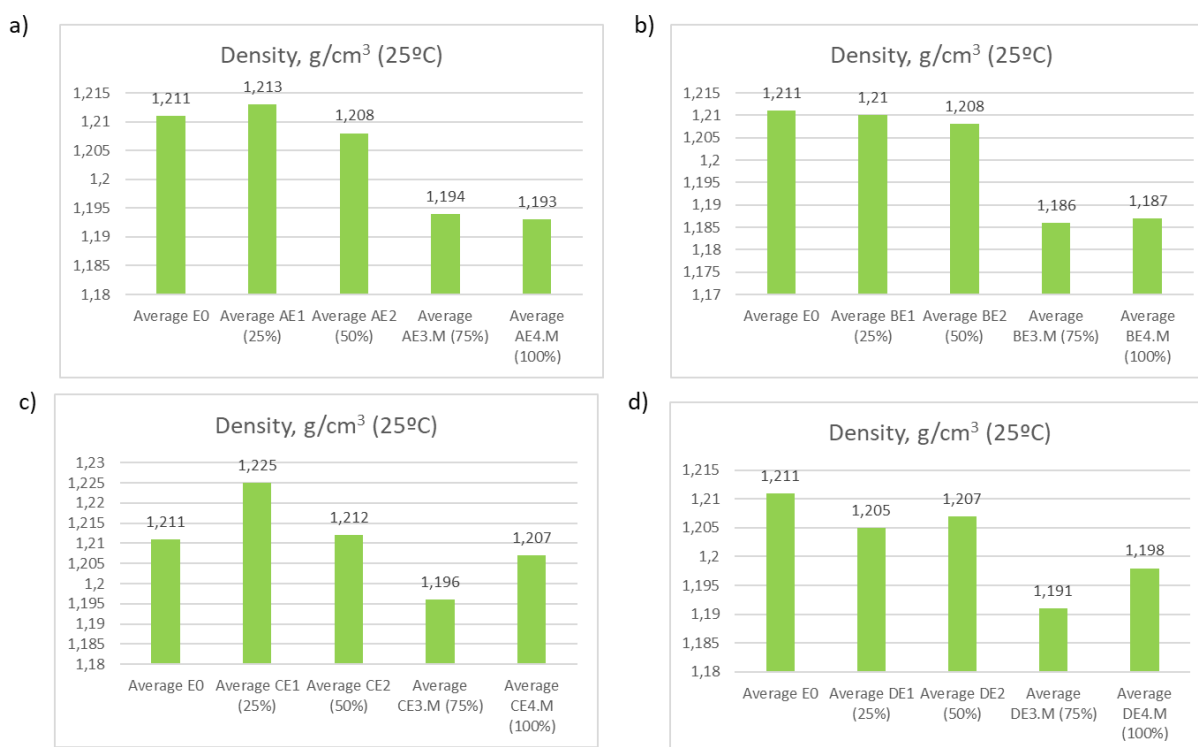


Figure 2. Density of a) reference resin (EO), lignin BTG NW SPL1 resin replacing 25% (AE1), 50% (AE2), 75% modified (AE3.M) and 100% modified (AE4M); b) reference resin (EO), lignin BTG NW SPL2 resin replacing 25% (BE1), 50% (BE2), 75% modified (BE3M) and 100% modified (BE4M); c) reference resin (EO), lignin BTG NW LPL resin replacing 25% (CE1), 50% (CE2), 75% modified (CE3M) and 100% modified (CE4M); d) reference resin (EO), lignin BTG NW MP resin replacing 25% (DE1), 50% (DE2), 75% modified (DE3M) and 100% modified (DE4M).

*pH measurement*

Due to plywood resins are condensed with sodium hydroxide and this parameter is fixed during polymerization, all resins have a similar and quite high pH value.

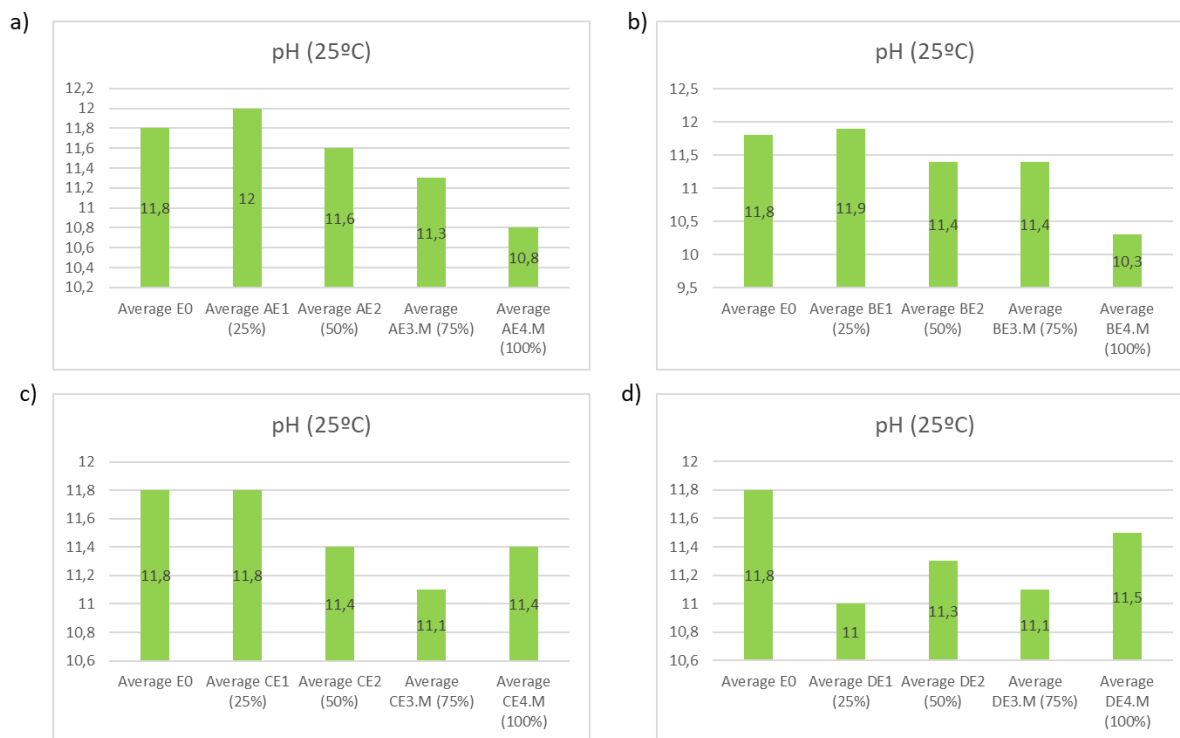


Figure 3. pH of a) reference resin (E0), lignin BTG NW SPL1 resin replacing 25% (AE1), 50% (AE2), 75% modified (AE3.M) and 100% modified (AE4M); b) reference resin (E0), lignin BTG NW SPL2 resin replacing 25% (BE1), 50% (BE2), 75% modified (BE3M) and 100% modified (BE4M); c) reference resin (E0), lignin BTG NW LPL resin replacing 25% (CE1), 50% (CE2), 75% modified (CE3M) and 100% modified (CE4M); d) reference resin (E0), lignin BTG NW MP resin replacing 25% (DE1), 50% (DE2), 75% modified (DE3M) and 100% modified (DE4M).

### Hardening time

In the study of hardening time, it was observed that when increasing the degree of substitution, this parameter also increases. This is due to the fact that the lignin is less reactive and as the percentage of lignin increases, the amount of more reactive phenol decreases resulting in a less reactive final resin.

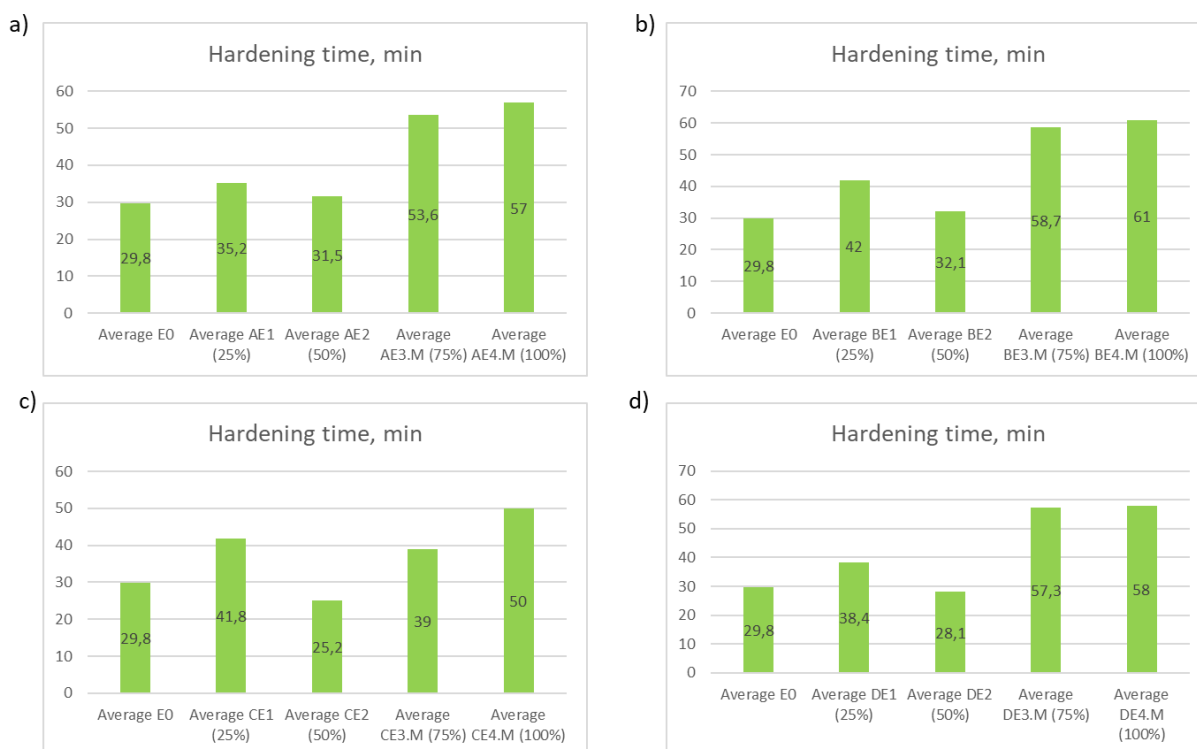


Figure 4. Hardening time of a) reference resin (E0), lignin BTG NW SPL1 resin replacing 25% (AE1), 50% (AE2), 75% modified (AE3.M) and 100% modified (AE4M); b) reference resin (E0), lignin BTG NW SPL2 resin replacing 25% (BE1), 50% (BE2), 75% modified (BE3M) and 100% modified (BE4M); c) reference resin (E0), lignin BTG NW LPL resin replacing 25% (CE1), 50% (CE2), 75% modified (CE3M) and 100% modified (CE4M); d) reference resin (E0), lignin BTG NW MP resin replacing 25% (DE1), 50% (DE2), 75% modified (DE3M) and 100% modified (DE4M).

*Solid content*

The solids content was determined by placing 3 g of sample in an oven at 135°C for 1 h. In the following graphs it is observed that there are no significant variations between the samples regarding the solid content.

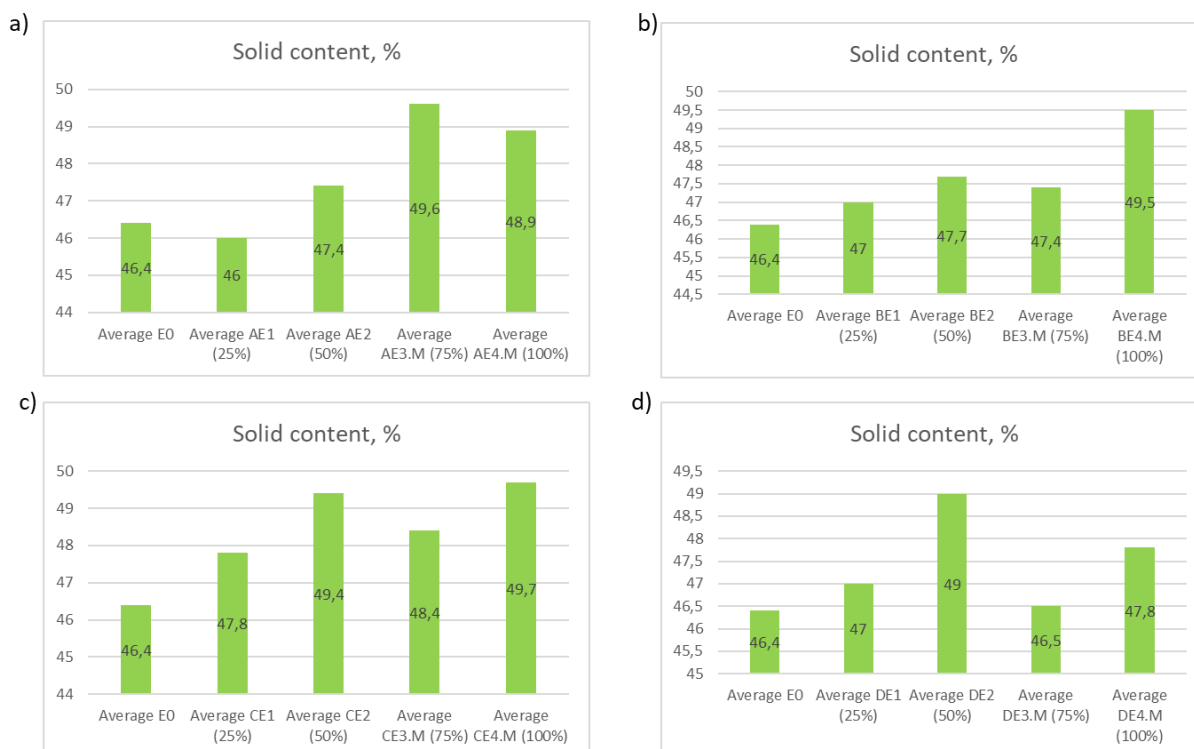


Figure 5. Solid content of a) reference resin (E0), lignin BTG NW SPL1 resin replacing 25% (AE1), 50% (AE2), 75% modified (AE3.M) and 100% modified (AE4M); b) reference resin (E0), lignin BTG NW SPL2 resin replacing 25% (BE1), 50% (BE2), 75% modified (BE3M) and 100% modified (BE4M); c) reference resin (E0), lignin BTG NW LPL resin replacing 25% (CE1), 50% (CE2), 75% modified (CE3M) and 100% modified (CE4M); d) reference resin (E0), lignin BTG NW MP resin replacing 25% (DE1), 50% (DE2), 75% modified (DE3M) and 100% modified (DE4M).

Free Formaldehyde and Phenol content

Lignin is less reactive than phenol and therefore, as the percentage of lignin increases the phenol content decreases resulting in a less reactive final resin and amounts of unreacted formaldehyde. Therefore, in the recipe using 75% and 100% of phenol substitution, less formaldehyde was applied. In the figure below, the free formaldehyde content before and after resin modification is shown.

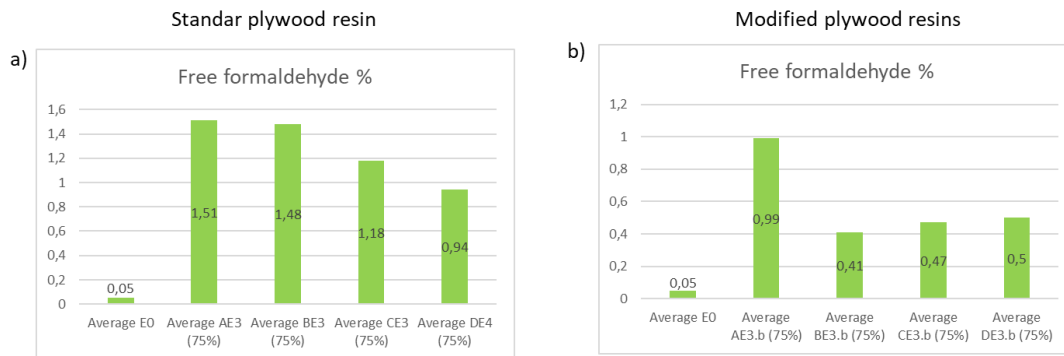


Figure 6. Free Formaldehyde of resins for a 75% of substitution of: a) standard plywood resin and b) modified plywood resins for free formaldehyde reduction.

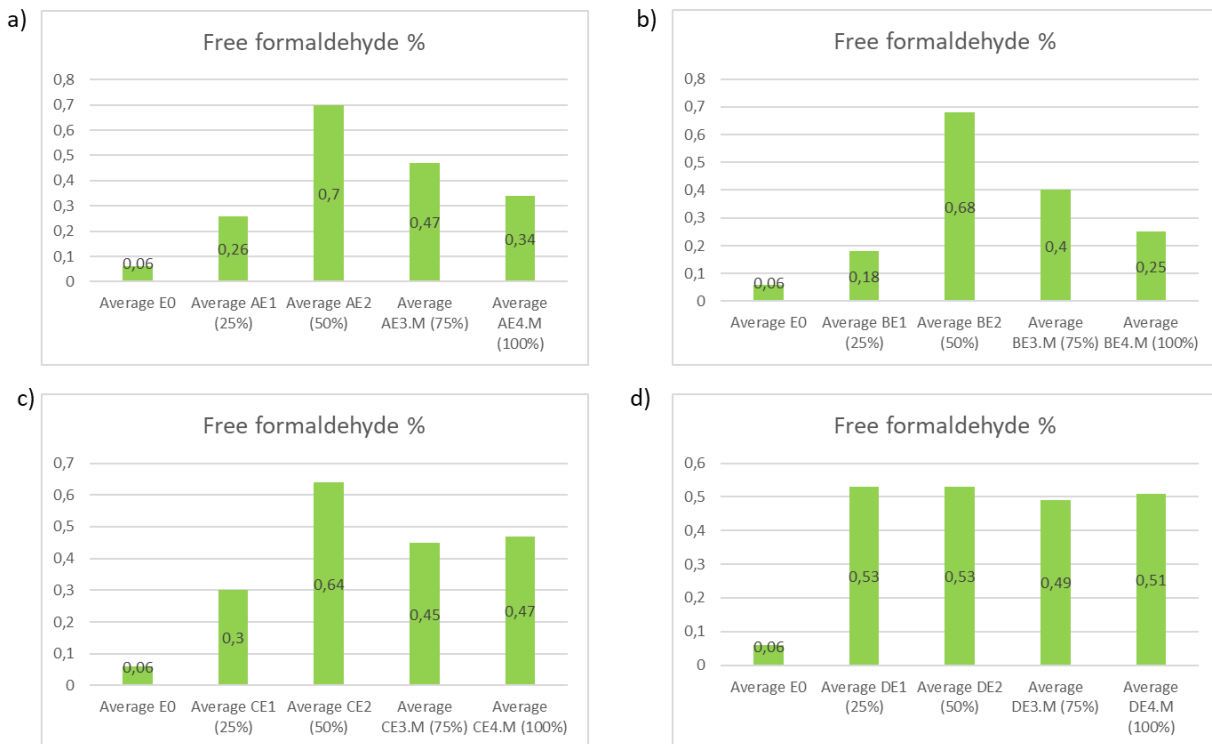


Figure 7. a) Reference resin (E0), lignin BTG NW SPL1 resin replacing 25% (AE1), 50% (AE2), 75% modified (AE3.M) and 100% modified (AE4.M); b) reference resin (E0), lignin BTG NW SPL2 resin replacing 25% (BE1), 50% (BE2), 75% modified (BE3M) and 100% modified (BE4M); C) reference resin (E0), lignin BTG NW LPL resin replacing 25% (CE1), 50% (CE2), 75% modified (CE3M)

and 100% modified (CE4M); d) reference resin (E0), lignin BTG NW MP resin replacing 25% (DE1), 50% (DE2), 75% modified (DE3M) and 100% modified (DE4M).

Plywood resins were developed with different grades of phenol substitution. So, when the percentage of lignin increased, the phenol content decreased and with this the amount of free phenol in the resin. This is illustrated in the figures below.

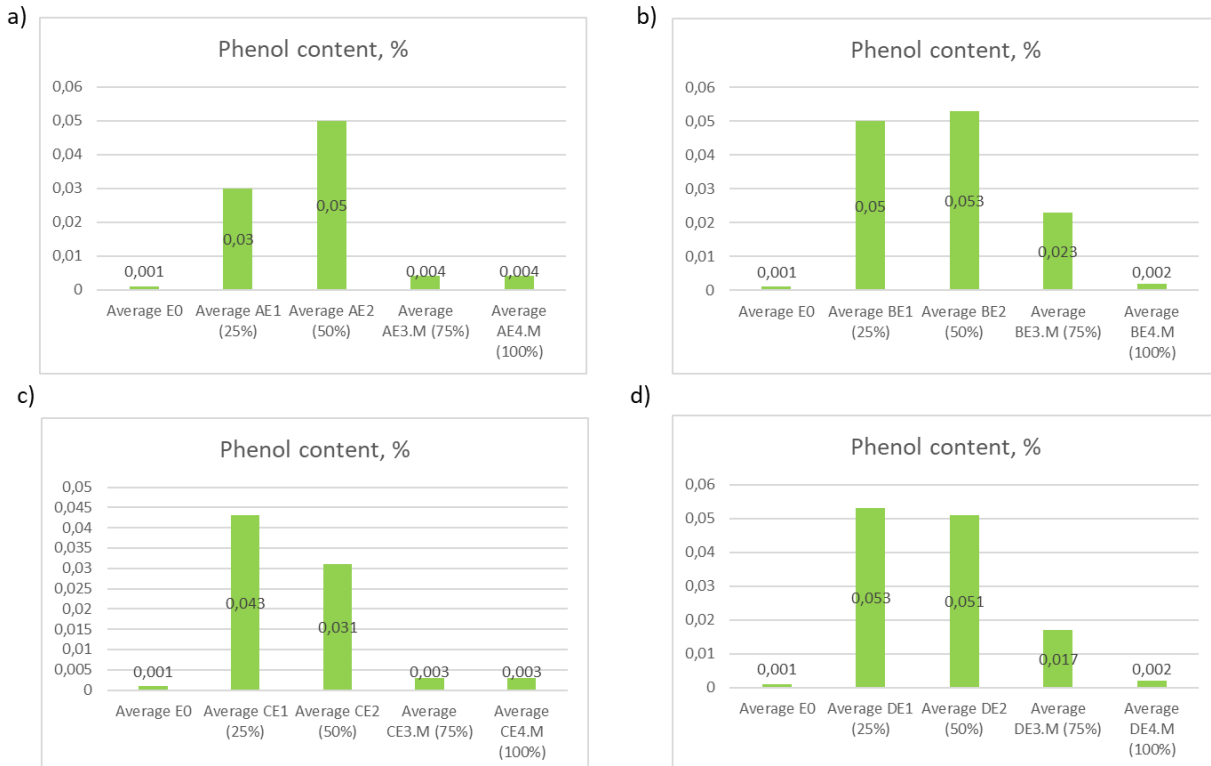


Figure 8. a) Reference resin (E0), lignin BTG NW SPL1 resin replacing 25% (AE1), 50% (AE2), 75% modified (AE3.M) and 100% modified (AE4M); b) reference resin (E0), lignin BTG NW SPL2 resin replacing 25% (BE1), 50% (BE2), 75% modified (BE3M) and 100% modified (BE4M); C) reference resin (E0), lignin BTG NW LPL resin replacing 25% (CE1), 50% (CE2), 75% modified (CE3M) and 100% modified (CE4M); d) reference resin (E0), lignin BTG NW MP resin replacing 25% (DE1), 50% (DE2), 75% modified (DE3M) and 100% modified (DE4M).

## Gel Permeation Chromatography (GPC)

Gel permeation chromatography (GPC) analysis was performed to evaluate the molecular weight of each resin. The earlier performed GPC study of each lignin indicated a weight for the SPL1 and SPL2 lignin in a range of 2700 to 3000 Da, 900 Da for LPL lignin and 450 Da for the MP lignin. The molecular weight of the plywood resins slightly decreased according to the type of lignin used following the next order SPL1 > SPL2 > LPL > MP. The repeatability between the different batches was also confirmed, obtaining very similar values.

- Resins with 25% of substitution

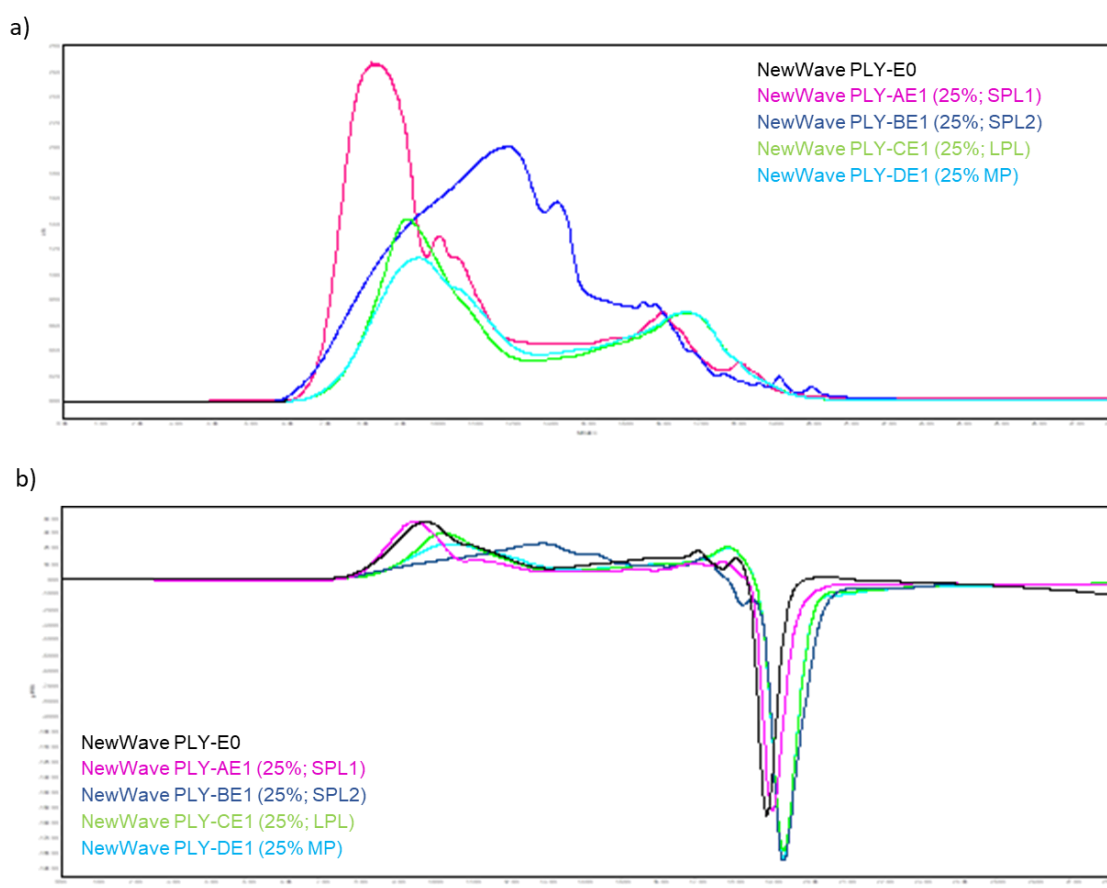


Figure 9. Molecular weight distribution in a) UV detector and b) IR detector of reference resin (E0), BTG NW SPL1 lignin resin replacing 25% (AE1), BTG NW SPL2 lignin resin replacing 25% (BE1), BTG NW LPL lignin resin replacing 25% (CE1), BTG NW MP lignin resin replacing 25% (DE1).

Table 2. Results of molecular weights of reference resin (E0), BTG NW SPL1 lignin resin replacing 25% (AE1), BTG NW SPL2 lignin resin replacing 25% (BE1), BTG NW LPL lignin resin replacing 25% (CE1), BTG NW MP lignin resin replacing 25% (DE1).

Sample	Mn (Da)	Mw (Da)	PDI
NewWave PLY-E0 (0%)	2866	463071	161,58
NewWave PLY-AE1 (SPL1; 25%)	2741	374512	152,10
NewWave PLY-BE1 (SPL2; 25%)	2668	410080	153,68
NewWave PLY-CE1 (LPL; 25%)	2457	420789	138,74
NewWave PLY-DE1 (MP; 25%)	2379	398741	141,78

▪ Resins with 50 % of substitution

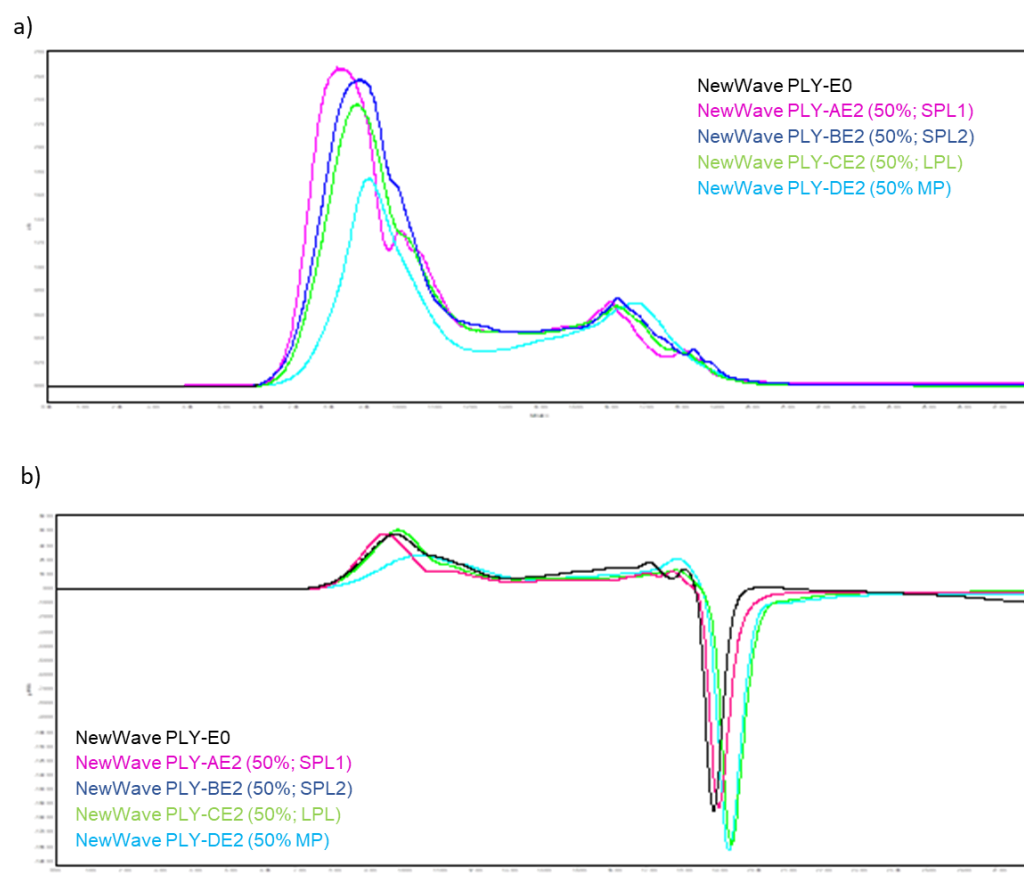


Figure 10. Molecular weight distribution in a) UV detector and b) IR detector of reference resin (E0), BTG NW SPL1 lignin resin replacing 50% (AE2), BTG NW SPL2 lignin resin replacing 50% (BE2), BTG NW LPL lignin resin replacing 50% (CE2), BTG NW MP lignin resin replacing 50% (DE2).

Table 3. Results of molecular weights of reference resin (E0), BTG NW SPL1 lignin resin replacing 50% (AE2), BTG NW SPL2 lignin resin replacing 50% (BE2), BTG NW LPL lignin resin replacing 50% (CE2), BTG NW MP lignin resin replacing 50% (DE2).

Sample	Mn (Da)	Mw (Da)	PDI
NewWave PLY-E0 (0%)	2866	463071	161,58
NewWave PLY-AE2 (SPL1; 50%)	2674	423671	137,54
NewWave PLY-BE2 (SPL2; 50%)	2535	303689	119,78
NewWave PLY-CE2 (LPL; 50%)	2315	478431	45,93
NewWave PLY-DE2 (MP; 50%)	2437	378459	99,87

■ Resins with 75% of substitution

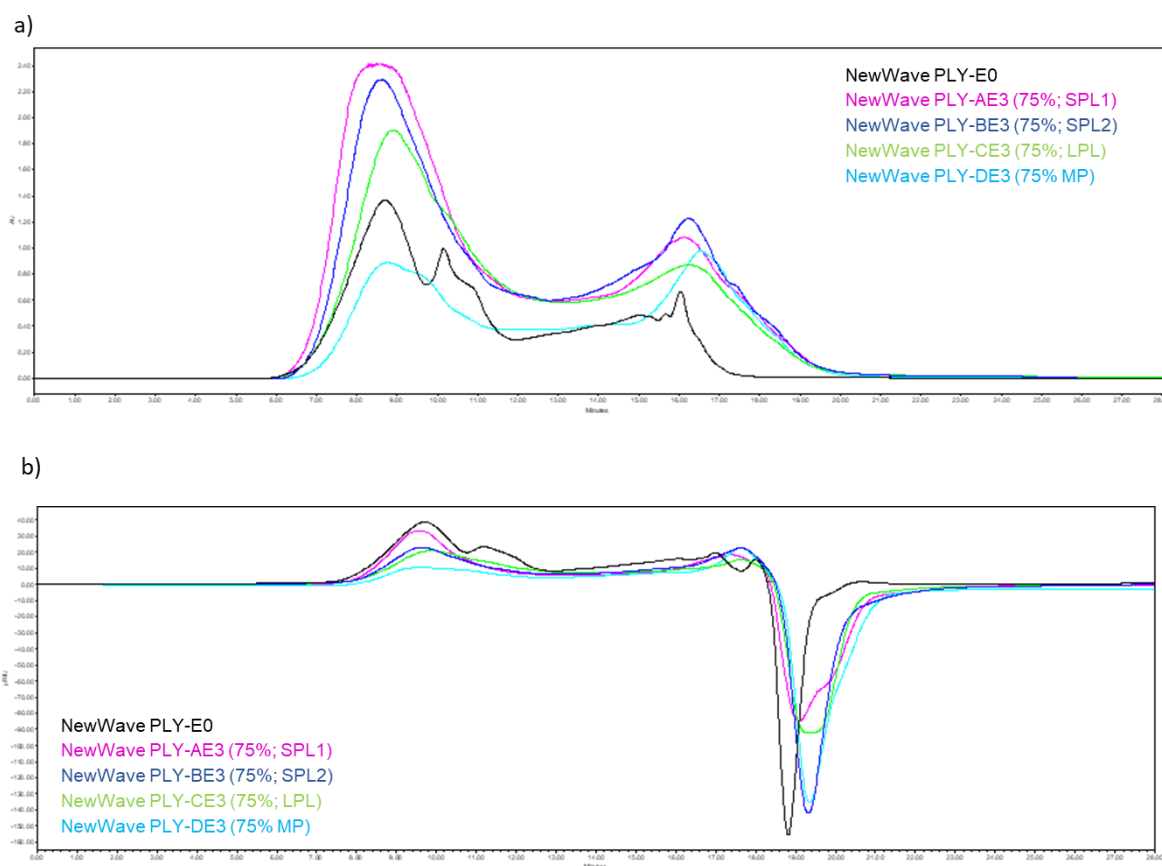


Figure 11. Molecular weight distribution in a) UV detector and b) IR detector of reference resin (E0), BTG NW SPL1 lignin resin replacing 75% (AE3), BTG NW SPL2 lignin resin replacing 75% (BE3), BTG NW LPL lignin resin replacing 75% (CE3), BTG NW MP lignin resin replacing 75% (DE3).

Table 4. Results of molecular weights of reference resin (E0), BTG NW SPL1 lignin resin replacing 75% (AE3), BTG NW SPL2 lignin resin replacing 75% (BE3), BTG NW LPL lignin resin replacing 75% (CE3), BTG NW MP lignin resin replacing 75% (DE3).

Sample	Mn (Da)	Mw (Da)	PDI
NewWave PLY-E0 (0%)	2866	463071	161,58
NewWave PLY-AE3 (SPL1; 75%)*	1663	285403	171,59
NewWave PLY-BE3 (SPL2; 75%)*	1523	305197	200,13
NewWave PLY-CE3 (LPL; 75%)*	2537	322703	127,21
NewWave PLY-DE3 (MP; 75%)*	1201	164825	137,22

Resins with 100% of substitution

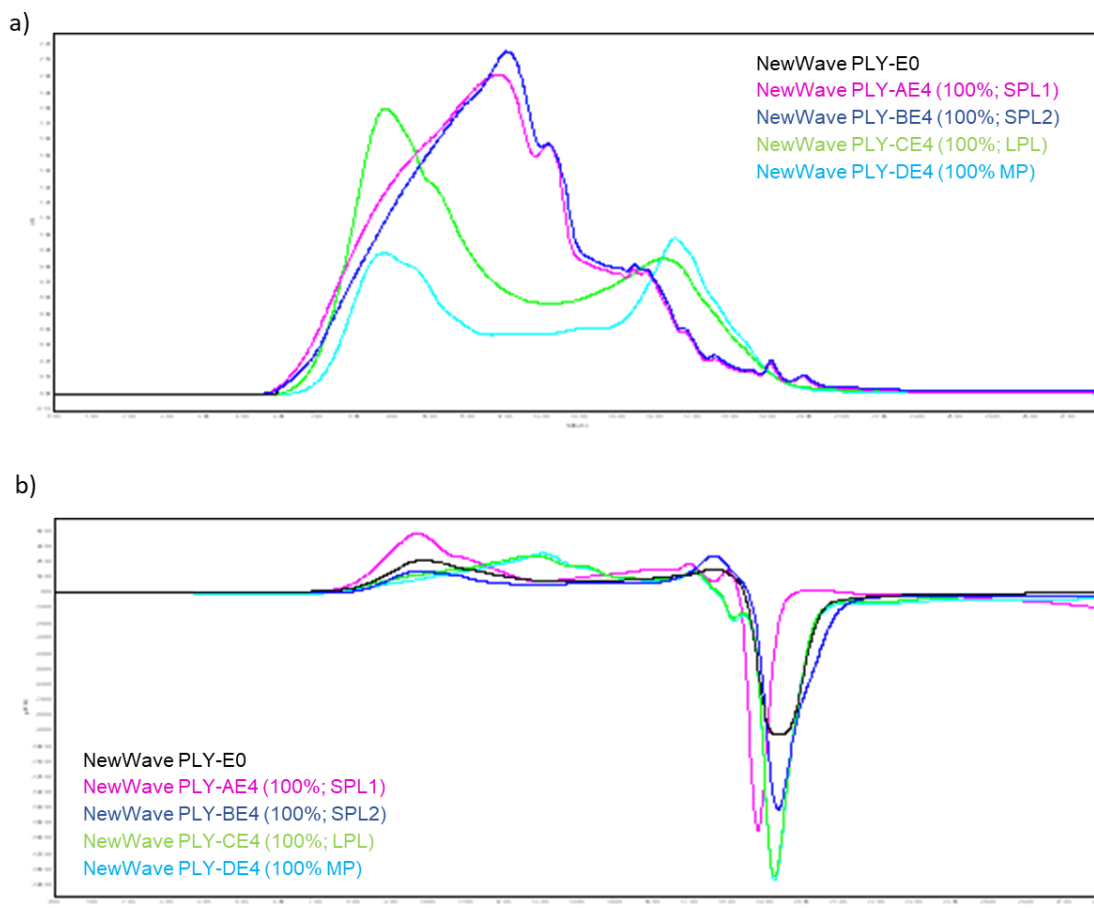


Figure 12. Molecular weight distribution in a) UV detector and b) IR detector of reference resin (E0), BTG NW SPL1 lignin resin replacing 100% (AE4), BTG NW SPL2 lignin resin replacing 100% (BE4), BTG NW LPL lignin resin replacing 100% (C43), BTG NW MP lignin resin replacing 100% (DE4).

Table 5. Results of molecular weights of reference resin (E0), BTG NW SPL1 lignin resin replacing 100% (AE4), BTG NW SPL2 lignin resin replacing 100% (BE4), BTG NW LPL lignin resin replacing 100% (C43), BTG NW MP lignin resin replacing 100% (DE4).

Sample	Mn (Da)	Mw (Da)	PDI
NewWave PLY-E0 (0%)	<b>2866</b>	<b>463071</b>	<b>161,58</b>
NewWave PLY-AE4 (SPL1; 100%)	<b>2407</b>	<b>440329</b>	<b>312,92</b>
NewWave PLY-BE4 (SPL2; 100%)	<b>2083</b>	<b>245617</b>	<b>226,70</b>
NewWave PLY-CE4 (LPL; 100%)	<b>1390</b>	<b>178907</b>	<b>138,39</b>
NewWave PLY-DE4 (MP; 100%)	<b>1107</b>	<b>142718</b>	<b>124,32</b>

## Thermal properties

### Thermogravimetric analysis (TGA)

The thermogravimetric analysis (TGA) technique allows to analyze how the properties of a material (mass) changes as a function of temperature and, therefore, allows to determine the decomposition temperatures of the different resins, as well as the residues that remains at the end of the thermal process in a controlled atmosphere. A temperature ramp to study the behavior of the samples against temperature was carried out. In the TGA graphs it can be observed that there are no significant differences between the different plywood resins. In most of the samples it was observed that the degradation at 900 °C was not complete, maintaining between 15-20% of its initial weight.

### Differential scanning calorimetry (DSC)

Differential scanning calorimetry (DSC) is a thermo-analytical technique that measures the temperature difference between a sample and an internal reference as a function of time and temperature. The observed temperature difference translates into a heat flow. This allows for measuring endothermic and exothermic transitions depending on said temperature.

The developed plywood resins will later be used in the production of plywood boards. Therefore, it is necessary to know the reactivity of the samples at temperatures between 60 °C and 200 °C. For this purpose, a ramp is made at 20°C min.

▪ Resins with 25% of substitution

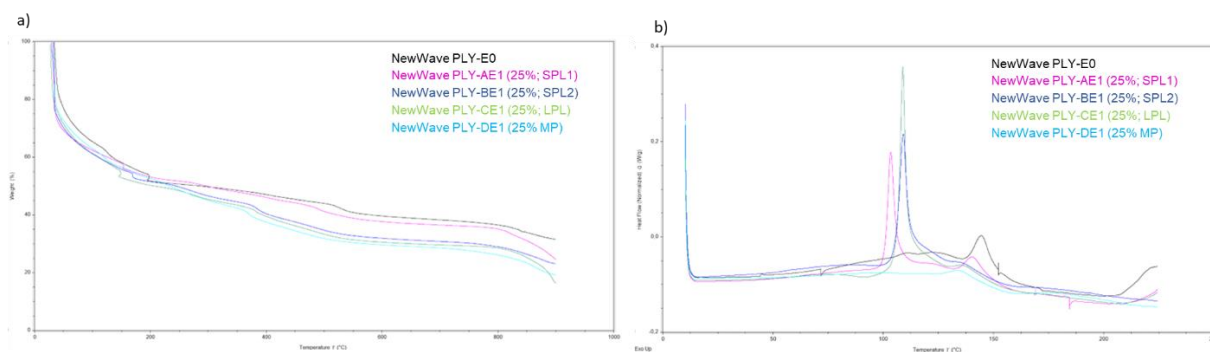


Figure 13. a) TGA and b) DSC of reference resin (E0), lignin BTG NW SPL1 resin replacing 25% (AE1), lignin BTG NW SPL2 resin replacing 25% (BE1), lignin BTG NW LPL resin replacing 25% (CE1) and lignin BTG MP resin replacing 25% (DE1).

Table 6. Results of enthalpy of reference resin (E0), lignin BTG NW SPL1 resin replacing 25% (AE1), lignin BTG NW SPL2 resin replacing 25% (BE1), lignin BTG NW LPL resin replacing 25% (CE1) and lignin BTG MP resin replacing 25% (DE1).

Sample	T <sup>a</sup> (°C)	Enthalpy (J/g)
NewWave PLY-E0 (0%)	144	146,41
NewWave PLY-AE1 (SPL1; 25%)	101	141,33
NewWave PLY-BE1 (SPL2; 25%)	110	113,45
NewWave PLY-CE1 (LPL; 25%)	107	121,94
NewWave PLY-DE1 (MP; 25%)	128	62,45

Resins with 50% of substitution

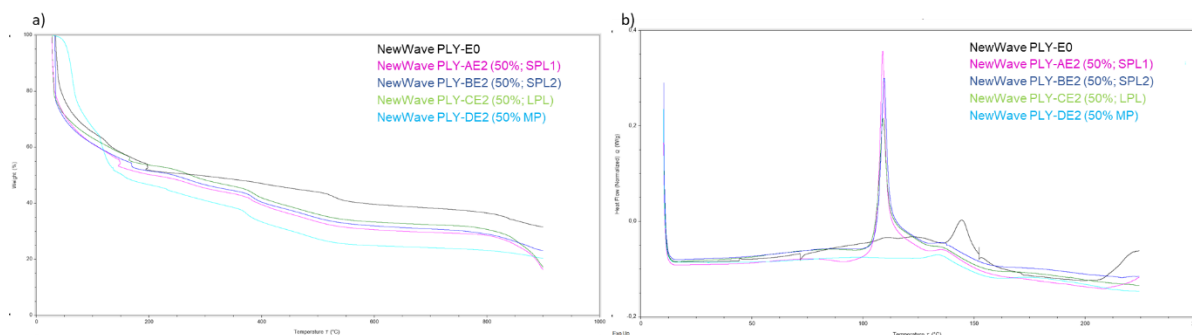


Figure 14. a) TGA and b) DSC of reference resin (E0), lignin BTG NW SPL1 resin replacing 50% (AE2), lignin BTG NW SPL2 resin replacing 50% (BE2), lignin BTG NW LPL resin replacing 50% (CE2) and lignin BTG MP resin replacing 50% (DE2).

Table 7. Results of enthalpy of reference resin (E0), lignin BTG NW SPL1 resin replacing 50% (AE2), lignin BTG NW SPL2 resin replacing 50% (BE2), lignin BTG NW LPL resin replacing 50% (CE2) and lignin BTG MP resin replacing 50% (DE2).

Sample	T <sup>a</sup> (°C)	Enthalpy (J/g)
NewWave PLY-E0 (0%)	144	146,41
NewWave PLY-AE2 (SPL1; 50%)	108	121,32
NewWave PLY-BE2 (SPL2; 50%)	109	137,04
NewWave PLY-CE2 (LPL; 50%)	107	108,95
NewWave PLY-DE2 (MP; 50%)	129	59,15

Resins with 75% of substitution

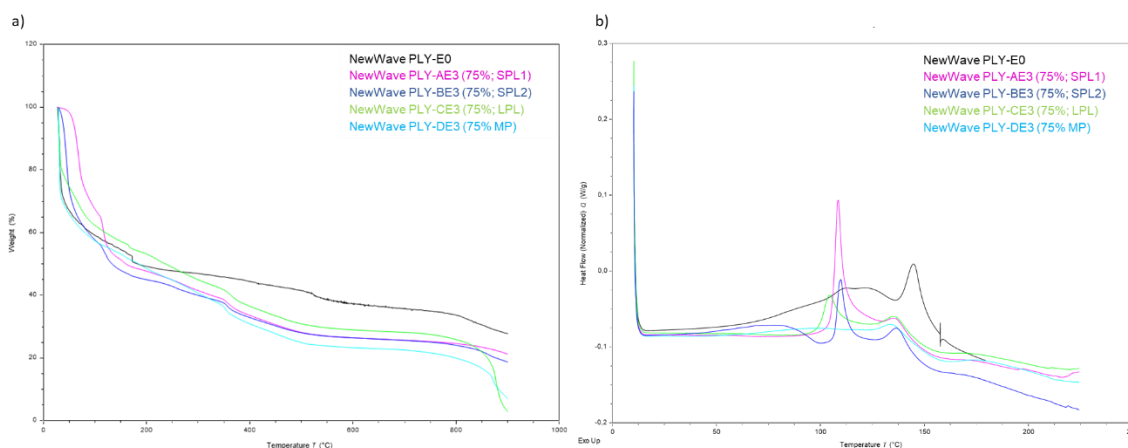


Figure 15. a) TGA and b) DSC of reference resin (E0), lignin BTG NW SPL1 resin replacing 75% (AE3), lignin BTG NW SPL2 resin replacing 75% (BE3), lignin BTG NW LPL resin replacing 75% (CE3) and lignin BTG MP resin replacing 75% (DE3).

Table 8. Results of enthalpy of reference resin (E0), lignin BTG NW SPL1 resin replacing 75% (AE3), lignin BTG NW SPL2 resin replacing 75% (BE3), lignin BTG NW LPL resin replacing 75% (CE3) and lignin BTG MP resin replacing 75% (DE3).

Sample	T <sup>a</sup> (°C)	Enthalpy (J/g)
NewWave PLY-E0 (0%)	144	146,41
NewWave PLY-AE3 (SPL1; 75%)*	108	77,52
NewWave PLY-BE3 (SPL2; 75%)*	111	115,94
NewWave PLY-CE3 (LPL; 75%)*	104	50,95
NewWave PLY-DE3 (MP; 75%)*	133	58,42

Resins with 100% of substitution

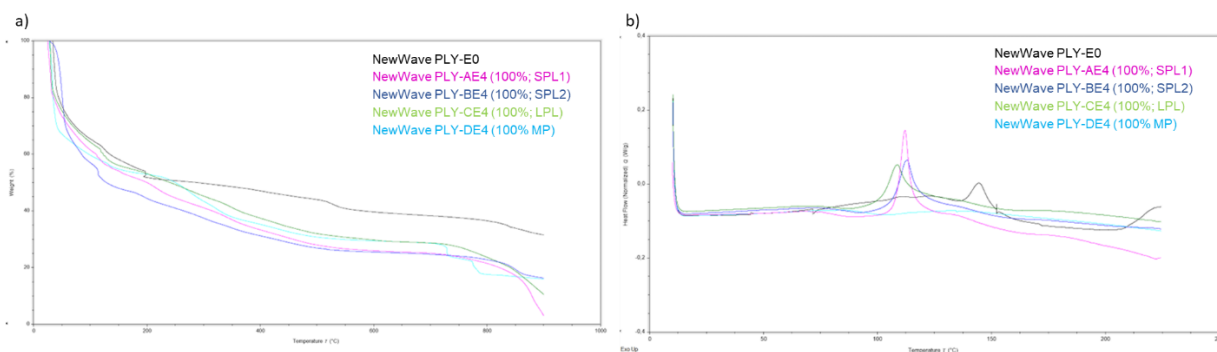


Figure 16. a) TGA and b) DSC of reference resin (E0), lignin BTG NW SPL1 resin replacing 100% (AE4), lignin BTG NW SPL2 resin replacing 100% (BE4), lignin BTG NW LPL resin replacing 100% (CE4) and lignin BTG MP resin replacing 100% (DE4).

Table 9. Results of enthalpy of reference resin (E0), lignin BTG NW SPL1 resin replacing 100% (AE4), lignin BTG NW SPL2 resin replacing 100% (BE4), lignin BTG NW LPL resin replacing 100% (CE4) and lignin BTG MP resin replacing 100% (DE4).

Sample	T <sup>a</sup> (°C)	Enthalpy (J/g)
NewWave PLY-E0 (0%)	144	146,41
NewWave PLY-AE4 (SPL1; 100%)	110	68,35
NewWave PLY-BE4 (SPL2; 100%)	112	109,72
NewWave PLY-CE4 (LPL; 100%)	106	49,47
NewWave PLY-DE4 (MP; 100%)	132	57,98

### Curing ramp (Rheometer)

Rheological properties were measured to analyze the stress-strain relationship and to understand the flow/deformation properties of the different resins. To evaluate the modulus generation capacity, a curing ramp was carried out in a rheometer at 20<sup>a</sup> C/min. The results show that in most cases, resins using LPL and MP lignins begin to gain modulus later than resins using SPL1 and SPL2 lignins including the reference indicating a later onset of curing. Considering the percentage used, the trend observed is that by increasing the substitution percentage, the resins begin to cure later because resins with a larger amount of lignin do not gain much module, and this starts later.

- Resins with 25% of substitution

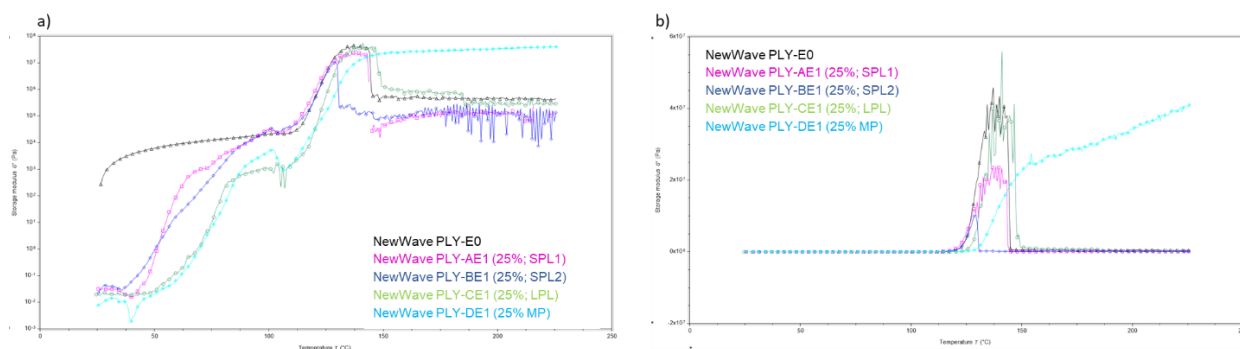


Figure 17. a) Rheometer and b) rheometer with logarithmic scale of reference resin (E0), lignin BTG NW SPL1 resin replacing 25% (AE1), lignin BTG NW SPL2 resin replacing 25% (BE1), lignin BTG NW LPL resin replacing 25% (CE1) and lignin BTG MP resin replacing 25% (DE1).

- Resins with 50% of substitution

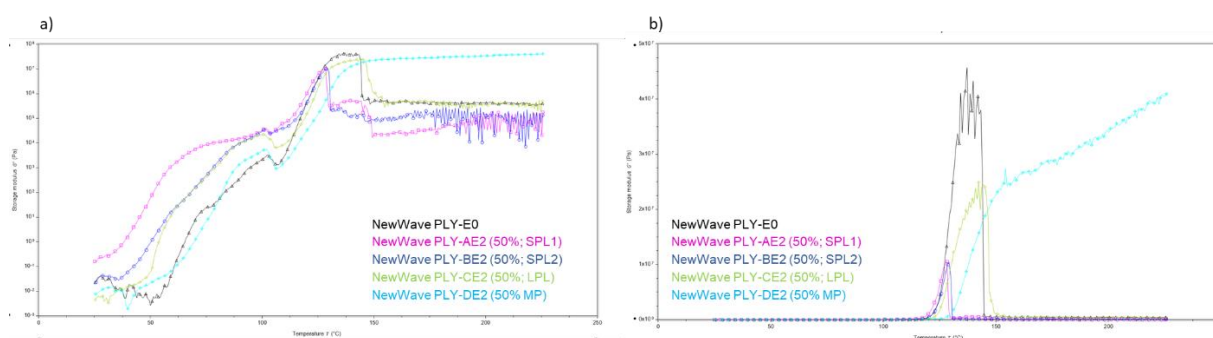


Figure 18. a) Rheometer and b) rheometer with logarithmic scale of reference resin (E0), lignin BTG NW SPL1 resin replacing 50% (AE2), lignin BTG NW SPL2 resin replacing 50% (BE2), lignin BTG NW LPL resin replacing 50% (CE2) and lignin BTG MP resin replacing 50% (DE2).

■ Resins with 75% of substitution

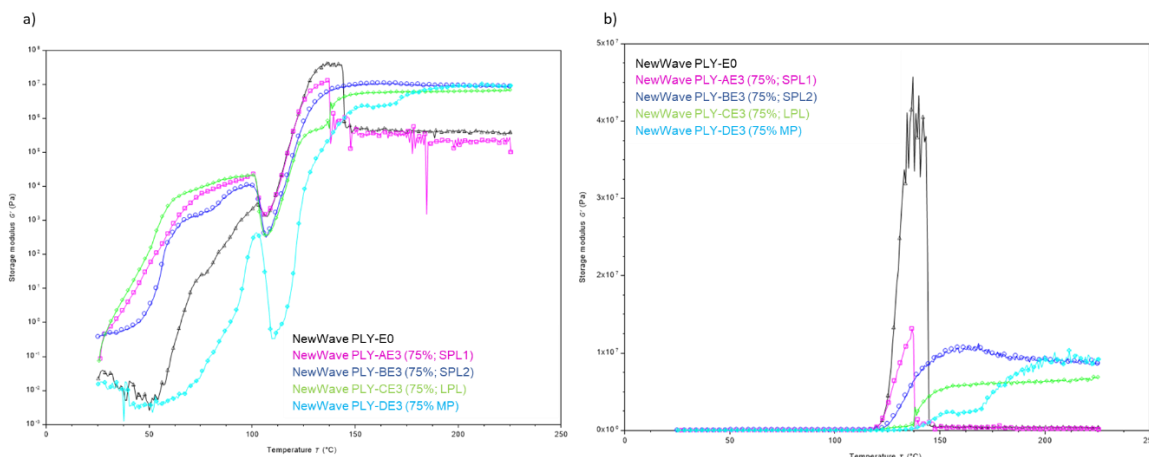


Figure 19. a) Rheometer and b) rheometer with logarithmic scale of reference resin (E0), lignin BTG NW SPL1 resin replacing 50% (AE2), lignin BTG NW SPL2 resin replacing 50% (BE2), lignin BTG NW LPL resin replacing 50% (CE2) and lignin BTG MP resin replacing 50% (DE2).

■ Resins with 100% of substitution

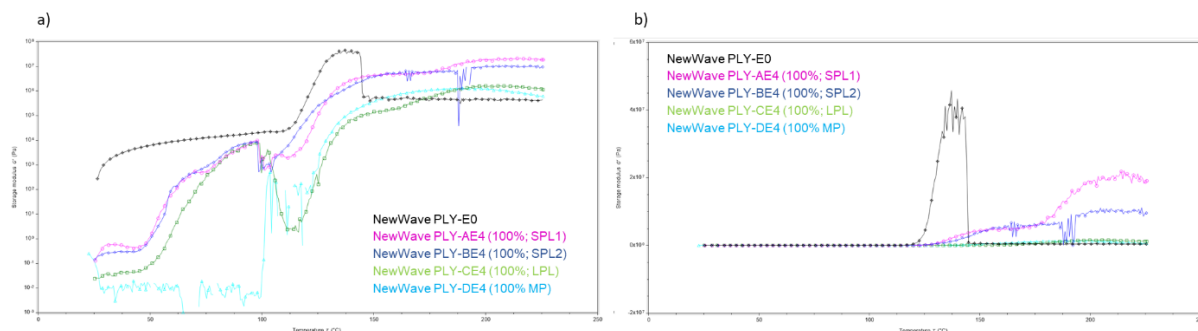


Figure 20. a) Rheometer and b) rheometer with logarithmic scale of reference resin (E0), lignin BTG NW SPL1 resin replacing 100% (AE4), lignin BTG NW SPL2 resin replacing 100% (BE4), lignin BTG NW LPL resin replacing 100% (CE4) and lignin BTG MP resin replacing 100% (DE4).

## MDF resins

During the period M12-M24, studies have started on the development of MDF resins varying the degree of phenol substitution by 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% of phenol with the different lignin's in the final MDF resin. Subsequently, a complete characterization was carried out.

### Characterization of MDF resins

In the table below, the physical-chemical properties of the MDF resins with 25% substitution applying the different lignin's are shown. The results show that there are no significant differences between the samples. However, a high content of free formaldehyde was observed in all MDF resins. Therefore, a smaller amount of formaldehyde will be used in the standard recipe in an endeavor to reduce the free formaldehyde content. For each resin, all analysis were performed in trifold, in the table below the average values for each property are given.

Table 10. Characterization of MDF resins [Ht (Hardening time); SC (solid content); FF (free formaldehyde); FP (Free phenol)]

	Lignin	Replaced	$\eta$ , cP (25°C)	$\rho$ , g/cm <sup>3</sup> (25°C)	pH (25°C)	H t, min	SC, %	Miscibility	FF, %	FP, %
$\bar{x}$ (E0)		0%	267	1,198	10,55	19	49,8	1000	0.12	0,0
$\bar{x}$ (AE1)	BTG NW SPL1	25%	380	1,201	10,68	13,2	49,8	1000	1.30	0,0
$\bar{x}$ (BE1)	BTG NW SPL2	25%	298	1,250	10,5	15,1	50,9	1000	1.31	0,0
$\bar{x}$ (CE1)	BTG NW MP	25%	280	1,248	10,8	15,7	51,0	1000	1.33	0,0
$\bar{x}$ (DE1)	BTG NW LPL	25%	258	1,197	10,56	14,8	51,0	1000	0.68	0,0

### Gel Permeation Chromatography (GPC)

Gel permeation chromatography (GPC) analyzes were performed to evaluate the molecular weight of each resin. As in the case of plywood resins and considering the earlier performed GPC study of each lignin which indicated a weight for the SPL1 and SPL2 lignin in a range of 2700 to 3000 Da, 900 Da for LPL lignin and 450 Da for the MP lignin., the molecular weight of the MDF resins slightly decreased according to the type of lignin used following the next order SPL1 > SPL2 > LPL > MP. The repeatability between the different batches was also confirmed, obtaining very similar values.

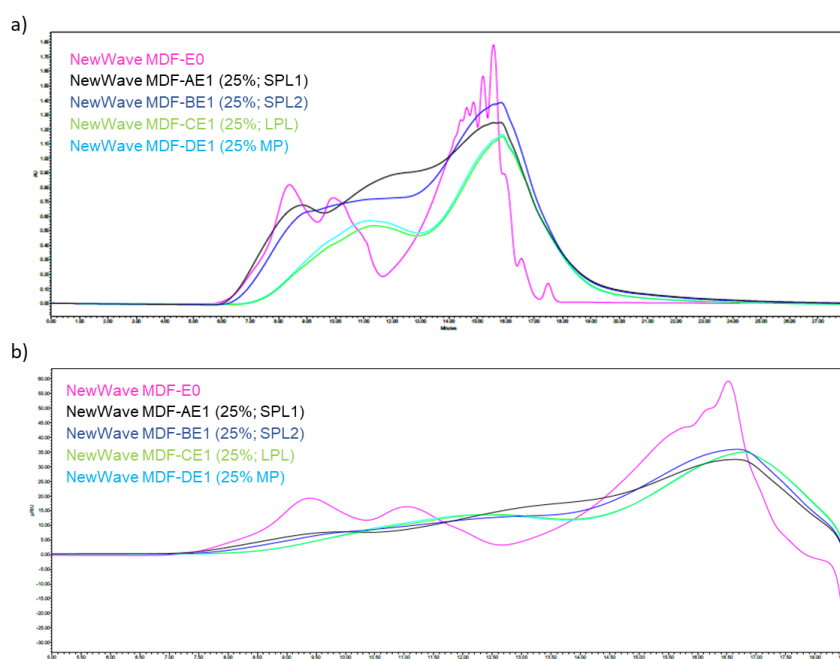


Figure 21. Molecular weight distribution in a) UV detector and b) IR detector of reference resin (E0), BTG NW SPL1 lignin resin replacing 25% (AE1), BTG NW SPL2 lignin resin replacing 25% (BE1), BTG NW LPL lignin resin replacing 25% (CE1), BTG NW MP lignin resin replacing 25% (DE1).

Table 11. Results of molecular weights of reference resin (E0), lignin BTG NW SPL1 resin replacing 25% (AE1), lignin BTG NW SPL2 resin replacing 25% (BE1), lignin BTG NW LPL resin replacing 25% (CE1) and lignin BTG MP resin replacing 25% (DE1).

Sample	Mn (Da)	Mw (Da)	PDI
NewWave MDF-E0 (0%)	2371	311462	54.55
NewWave MDF-AE1 (SPL1; 25%)	1607	166139	93.05
NewWave MDF -BE1 (SPL2; 25%)	1468	106449	140.03
NewWave MDF CE1 (LPL; 25%)	1317	38940	51.74

NewWave MDF DE1 (MP; 25%)	1331	42723	60.69
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## Thermal properties

### Thermogravimetric analysis (TGA)

A temperature ramp to study the behavior of the samples against temperature was carried out. In the TGA graphs there are no significant differences between the different MDF resins. In most of the samples it was observed that the degradation at 900 °C was not complete, maintaining between 15-20% of its initial weight.

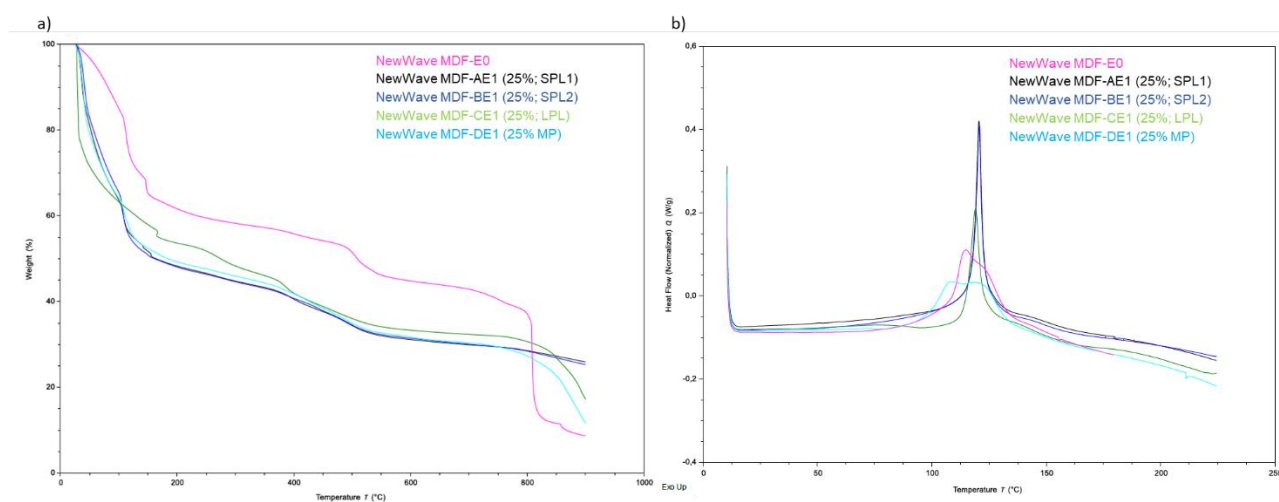


Figure 22. a) TGA and b) DSC of reference resin (E0), lignin BTG NW SPL1 resin replacing 25% (AE1), lignin BTG NW SPL2 resin replacing 25% (BE1), lignin BTG NW LPL resin replacing 25% (CE1) and lignin BTG MP resin replacing 25% (DE1).

### Differential scanning calorimetry (DSC)

The developed MDF resins will later be used in the production of MDF boards. Therefore, it is necessary to know the reactivity of the samples at temperatures between 60 °C and 200 °C.

The table below shows the temperature (curing) and enthalpy results of each MDF resin manufactured. It can be seen that the temperature for all the resins is between 114-120 °C while the temperature of the reference resin is at 114°C. Only the resin containing the MP lignin with a phenol substitution of 25% (DE1) shows a lower temperature (109°C) and the lowest enthalpy value.

*Table 12. Results of enthalpy of reference resin (E0), lignin BTG NW SPL1 resin replacing 25% (AE1), lignin BTG NW SPL2 resin replacing 25% (BE1), lignin BTG NW LPL resin replacing 25% (CE1) and lignin BTG MP resin replacing 25% (DE1).*

Sample	T <sup>a</sup> (°C)	Enthalpy (J/g)
NewWave MDF-E0 (0%)	<b>115</b>	<b>186.06</b>
NewWave MDF-AE1 (SPL1; 25%)	<b>120</b>	<b>138.98</b>
NewWave MDF -BE1 (SPL2; 25%)	<b>121</b>	<b>185.07</b>
NewWave MDF CE1 (LPL; 25%)	<b>119</b>	<b>146.14</b>
NewWave MDF DE1 (MP; 25%)	<b>109</b>	<b>127.98</b>

### Curing ramp (Rheometer)

Rheological properties were measured to analyse the stress-strain relationship and to understand the flow/deformation properties of the different resins. To evaluate the modulus generation capacity, a curing ramp was carried out in a rheometer at 20<sup>a</sup> C/min. The results indicate that both the MDF resin using SPL1 and SPL2 lignins begin to cure before the reference resin and that the resins using SPL2 lignin presents the highest modulus value. On the other hand, the resins using LPL and MP lignins barely gain module.

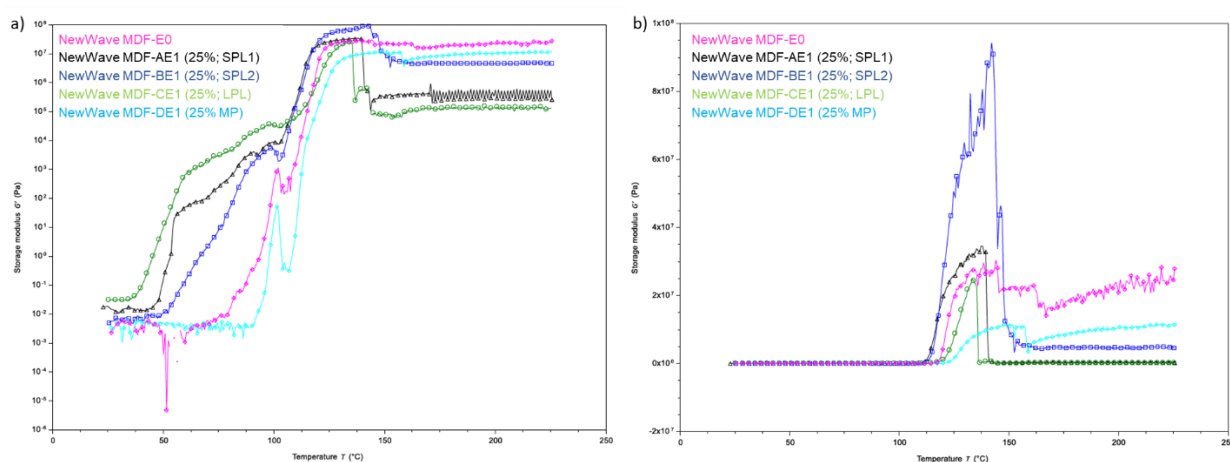


Figure 23. a) Rheometer and b) rheometer with logarithmic scale of reference resin (E0), lignin BTG NW SPL1 resin replacing 25% (AE1), lignin BTG NW SPL2 resin replacing 25% (BE1), lignin BTG NW LPL resin replacing 25% (CE1) and lignin BTG MP resin replacing 25% (DE1).

## Conclusions

D5.2 is the second of two deliverables describing the development of new plywood resins. In Task “5.1 Lignin characterization”, four lignin samples were received from BTG and have been analyzed. Parameters such as the water content, density, viscosity, pH, % of phenol, elemental analysis (C, H, N), GPC & metal content have been analyzed. Furthermore, to be able to understand the reactivity of the samples, thermal degradation, calorimetry scanning differential and curing ramp analyses were performed as well. Noteworthy is that during the analysis and testing, the samples showed to be very homogeneous, which is not always the case with lignins and lignin derived materials.

In Task “5.2 Resin screening test”, a recipe was developed, and a 100 % phenol-formaldehyde resin was synthesized to be used as a blank to compare with resins with incorporated lignin to replace the phenol. The different lignin samples provided by BTG were tested as phenol substitutes in amounts/percentages of 25%, 50% & 75%. All the resin synthesis reactions could be carried out without problems in the polymerization step, except for resins with 75% phenol substitution by the solid lignin samples (SPL). At 75% substitution, the solid lignin samples generate a too high viscosity in the reactor, preventing polymerization. The synthesized resins have been analyzed to obtain information required for evaluation in the following tasks.

Regarding the Task “5.3 Resin application tests and upscaling”, plywood resins were produced replacing phenol by the different lignin’s in amounts of 25%, 50%, 75% and even 100%. D5.2 shows the complete characterization of all the resins generated regarding density, viscosity, pH, % of phenol and formaldehyde, thermal degradation, calorimetry scanning differential and curing ramp analyses. Moreover, D5.2 gives the first results of the development of MDF resins with a phenol substitution of 25% using the four different lignin’s incl. a complete characterization. The use of the developed resins for the manufacture of both plywood and MDF boards incl. the procedure and characterization are described in D5.3 that corresponds to the Task “5.4: Development and characterization of MDF, plywood and CLT panels.”

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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