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I-PAN

INNOVATIVE POPLAR LOW DENSITY STRUCTURAL PANEL

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D3.4 Report on validation of the developed resins in lab scale LSB

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Document information

Abstract

In the framework of I-PAN, CHIMAR develops a technology for producing an innovative resin suitable for bonding poplar strands and recycled wood to form light weight OSB (LSB) panels for structural use.

The R&D objectives for the innovative resin system are:

- Curing temperature lower than that of the conventional resin systems by up to 10%.
- Compensation of strand moisture contents higher than 3.5%.
- To provide panels with formaldehyde emissions satisfying the most stringent European Standards as per EN 13986.

This report details the work performed by CHIMAR in the synthesis and characterisation of resins and their subsequent evaluation in the production of lab scale LSB.

Keywords

Poplar, wood, OSB, wood-based panels, engineered wood, binder, adhesive, resin, formaldehyde

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* Abbreviations of editor/contributor name

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LIST OF ABBREVIATIONS AND DEFINITIONS

Table 1 - List of abbreviations

OSB	Oriented Strand Board
LSB	Lightweight Strand Board
MUF	Melamine Urea Formaldehyde
MUPF	Melamine Urea Phenol Formaldehyde
PF	Phenol Formaldehyde
IB	Internal Bond strength or tensile strength
TS	24h Thickness swelling
MOR	Modulus of Rupture or bending strength
MOR-B	Bond Durability - Modulus of Rupture after 2h boiling
V100	IB after 2h boiling and drying
Perforator	Formaldehyde content as per EN 120
ANOVA	Analysis of Variance
MR	Molar Ratio of Resins
DSC	Differential Scanning Calorimetry

1 INTRODUCTION

I-PAN project aims at providing novel and environmentally friendly solutions in the field of engineered wood boards (or wood-based panels) and particularly in Oriented Strand Boards (OSB) manufacturing process with the target to reach the higher level functional characteristics of Lightweight Strand Boards (LSB). OSB/LSB is a type of wood-based panel consisting of strands of wood pressed together in layers and bonded with a synthetic resin. The wood species used in OSB manufacture include both softwood and hardwood. The resin types typically used include Phenol formaldehyde (PF), Melamine fortified Urea Formaldehyde (MUF) or isocyanate (pMDI). In the I-PAN project a breakthrough lightweight OSB panel will be developed, comprising 50% of its volume of recycled wood and for the remaining 50% of poplar wood by using the upper part of the poplar tree that is commonly underused. To this purpose, a novel OSB manufacturing process will be developed and an innovative binder system will be formulated thus allowing the reduction of energy requirements during the drying and pressing process of OSB manufacture, the minimization of VOC emissions and the reduction of the overall production cost.

In the framework of I-PAN, CHIMAR develops a technology for producing an innovative resin suitable for bonding poplar strands and recycled wood to form light weight OSB panels for structural use. The innovative resin is based on formaldehyde and will be of the aminoplastic (urea-formaldehyde, urea-melamine-formaldehyde/melamine-urea-formaldehyde, melamine-formaldehyde), or phenolic (phenol-formaldehyde) type or a combination of these types and it will be combined with a suitable cross-linking agent and possibly with a suitable formaldehyde catcher.

The R&D objectives for the innovative resin system are:

- Curing temperature lower than that of the conventional resin systems by up to 10%.
- Compensation of strand moisture contents higher than 3.5%.
- To provide panels with formaldehyde emissions satisfying the most stringent European Standards as per EN 13986.

In what follows, the work performed by CHIMAR comprising the synthesis and characterisation of resins and their subsequent evaluation in the production of lab scale LSB is described. Since this work was based on the findings of previous deliverables D3.2 and D3.3, a summary of them is first given.

2 SUMMARY OF THE WORK PERFORMED IN D3.2 AND D3.3

In the framework of D3.2, CHIMAR work included the development of suitable resin precursors, their application in preliminary resin synthesis and their subsequent evaluation in the production of lab scale LSB. At first both, the lab resin synthesis process and OSB lab preparation were calibrated. Both, strands from Bulgarian spruce and poplar strands received from IMAL/IBL, were used to produce the light weight OSB. The resin precursors were based on urea, formaldehyde, melamine and phenol. Suitable formulations of MUF and MUPF type resins were developed and samples of them were produced at CHIMAR lab and tested. The binder systems included special hardeners and crosslinkers to enhance the performance of the main binder and allow the reduction of the press temperature. Prior to panel production the strands were dried with target final moisture content around 3-4%. Panels with reduced density were prepared and their mechanical strength, water resistance and emission potential were tested. The effect of viscosity of the binder resin was evaluated. Optimization was performed to adjust the properties of the panels as well as their formaldehyde emission potential. The results indicated that it is feasible to manufacture at laboratory scale light weight boards (density level of ~500kg/m^3) with poplar strands of I-214 clone supplied by the I-PAN partners, which meet the standard requirements for OSB/3 grade as well as the E1 formaldehyde class. The press temperature was reduced by more than 10% without increasing the press cycle thus meeting the target of lower energy demand.

In the framework of D3.3, CHIMAR continued with the verification of the results obtained in Task 3.2, via extended lab trials. The effect of reduced board density (in the range of 450-500kg/m^3) on the overall performance of the LSB was studied. The lower the density of the panels is, the worse the panel properties are. The deterioration of the board properties, due to the reduced density of the LSB, was compensated by increased resin loading. The optimum resin loading can only be determined during a pilot or an industrial scale trial. A PF-resin system was developed and its use in lab scale LSB production was examined too. The results indicated that it is feasible to produce at laboratory scale light weight OSB-type boards meeting the standard requirements for OSB/3 grade as well as the E1 formaldehyde class using poplar strands of I-214 clone supplied by the I-PAN partners and a binder system of PF resin with special hardener. The press temperature and the press cycle for the PF-LSB were the same as those for the MUPF-bonded LSB. The use of specially developed hardener is essential to obtain PF-bonded LSB with properties meeting the standard requirements.

3 CONSIDERATIONS AND METHODS

The experimental study carried out by CHIMAR and reported herein included the validation of the resins developed in the previous Tasks 3.2 and 3.3 in the production of lab scale LSB. The LSB boards were prepared out of poplar strands made from a new fast-growing poplar tree species namely I-214 clone, which is grown in several European countries. Additionally the upper part of the tree log and the tree branches were mainly used for the preparation of the strands. The strands were supplied by the partners IBL, IMAL.

It should be pointed out that the laboratory strand boards prepared have actually random-orientated strands rather than oriented. Therefore, further to the typical test methods applied as per EN 300 - IB, TS, MOR and V100, the ***bond durability*** test (MOR-B), according to Canadian standard CSA 0437-93 on OSB and Waferboard, was also performed. The said CSA contains, further to the O-1 and O-2 grades, also the R-1 grade i.e. Random strand board that is more related to the laboratory panels produced at CHIMAR lab.

Furthermore, the optimization of the application of the PF resin system was studied. DSC analysis was also performed on both the MUPF and the PF resins used for the production of poplar LSB. The analysis was done on both straight resin samples and resin systems as applied in OSB panel production.

The main target of the work that is presented herein is to obtain panels with the desired properties/performance (OSB/3) and low formaldehyde emission levels (E1 class).

4 EXPERIMENTAL STUDY

The raw materials used were:

- Poplar strands delivered by IBL/IMAL
- Resins of MUPF or PF type produced in CHIMAR laboratory using conventional raw materials
- Hardeners, conventional and special type, prepared in CHIMAR lab
- Cross-linking agents, prepared in CHIMAR lab
- Formaldehyde catchers/scavengers prepared in CHIMAR lab
- Paraffin wax from a Greek producer.

The first objective addressed was the optimization of the addition level of the special hardener, which was used to enhance the PF resin curing during laboratory LSB panel production. As reported earlier, the use of a special hardener is essential for mechanical properties improvement and optimization of its level of addition was required, since the higher addition level had negative effect on the LSB properties. Furthermore, the glue factor of the PF resin was reduced as compared to previous trials so as to better simulate the industrial scale production.

Thus, the lab LSB production was performed by applying PF resin systems with either K_2CO_3 hardener at one level as reference, or proprietary CHIMAR hardener H5545 at two different levels, which, however, were lower than the level used in the previous trials. The properties of the PF resin produced in CHIMAR lab are depicted on the next table.

Table 2: *Resin properties*

Resin type	PF for OSB
Solids, 2g 2h 120°C, %	49.86
Viscosity, 25°C, cP	240
pH, 25°C	11.18
Free formaldehyde, %	0.11
Alkalinity, %	4.71
Gel time, 100°C, min	12

The production details and settings of the one layer strand boards produced with random orientation of the poplar strands are shown in the next table.

Table 3: *OSB Production details*

Board Dimensions, cm	43 x 43 x 1.6	
Target Density, kg/m^3	500	
Target board moisture, %	6.5	
Press Factor, s/mm	12	
Press Temperature, ^{o}C	180	
Wood Source	Poplar IBL/IMAL	
Resin Factor, % solid on dry wood	6.0	
Paraffin Level, % solid on dry wood	1.0	
Hardener Type	K_2CO_3	H5545
Hardener Level, % solid on solid resin	3.0	2.0 and 4.0
Target mat moisture, %	10.5	
Strands initial moisture, %	5.5-6.5	

The panels obtained were subjected to the full range of tests – IB, TS, V100, MOR, MOR-B and perforator test. The average testing values are shown in the next table.

Table 4: *OSB Testing results - set 1*

Press temperature, ^{o}C	180		
Press factor, s/mm	12		
Target density, kg/m^3	500		
Hardener type / level, %	K_2CO_3 / 3.0	H5545 / 2.0	H5545/ 4.0
Thickness, mm	16.12	16.08	16.04
Density, kg/m^3	502	498	487
IB, Mpa	0.28	0.30	0.36
V100, Mpa	0.11	0.12	0.14
TS, %	24.52	22.20	18.81
MOR, Mpa	13.03	13.99	14.20
MOR-B, Mpa	3.24	3.69	4.34
Perforator, mg/100g	2.35	2.01	1.98

For statistical reasons and in order to verify the results obtained, the same LSB production was repeated and the results of this series of trials are summarised in the next table.

Table 5: *OSB Testing results - set 2*

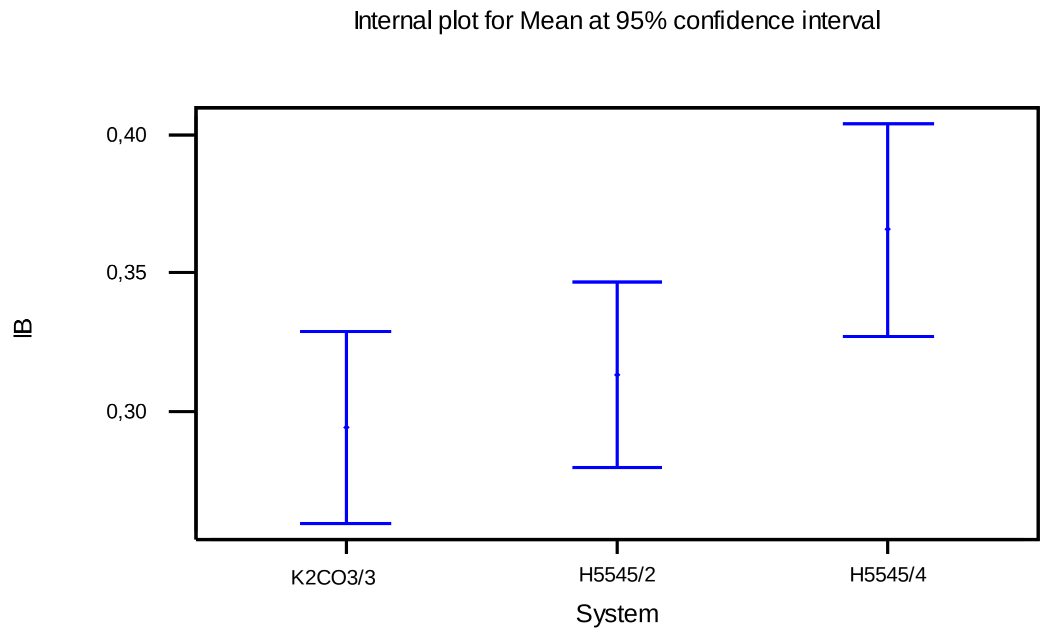
Press temperature, °C	180		
Press factor, s/mm	12		
Target density, kg/m^3	500		
Hardener type / level, %	K_2CO_3 / 3.0	H5545 / 2.0	H5545/ 4.0
Thickness, mm	16.09	16.05	16.02
Density, kg/m^3	485	492	490
IB, Mpa	0.30	0.32	0.37
V100, Mpa	0.14	0.15	0.17
TS, %	19.09	20.28	19.36
MOR, Mpa	14.32	16.10	16.34
MOR-B, Mpa	3.94	4.14	4.60
Perforator, mg/100g	1.75	1.87	1.80

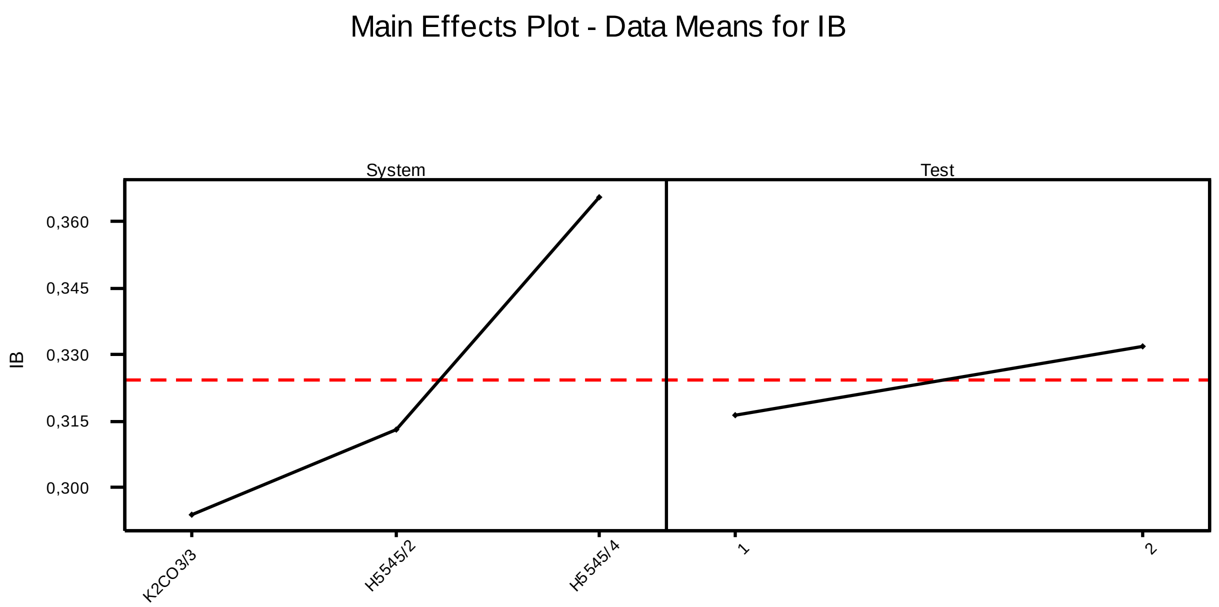
The testing values obtained from both series of tests were subjected to Analysis of Variance (ANOVA) and the results are the following:

```
Analysis of Variance for IB, using Adjusted SS for Tests

Source         DF     Seq SS     Adj SS     Adj MS      F      P
System          2   0,033039   0,033039   0,016519   4,86  0,015
Test            1   0,002178   0,002178   0,002178   0,64  0,430
System*Test     2   0,000172   0,000172   0,000086   0,03  0,975
Error          30   0,101900   0,101900   0,003397
Total          35   0,137289
```

The ANOVA shows that only the system (hardener/level) **is significant** factor at 95% confidence interval for the IB of the produced LSB, and there is no interaction between the number of test and the system.


Internal plot for Mean at 95% confidence interval
0,40
0,35
0,30
IB
K2CO3/3
H5545/2
H5545/4
System


Main Effects Plot - Data Means for IB
System
Test
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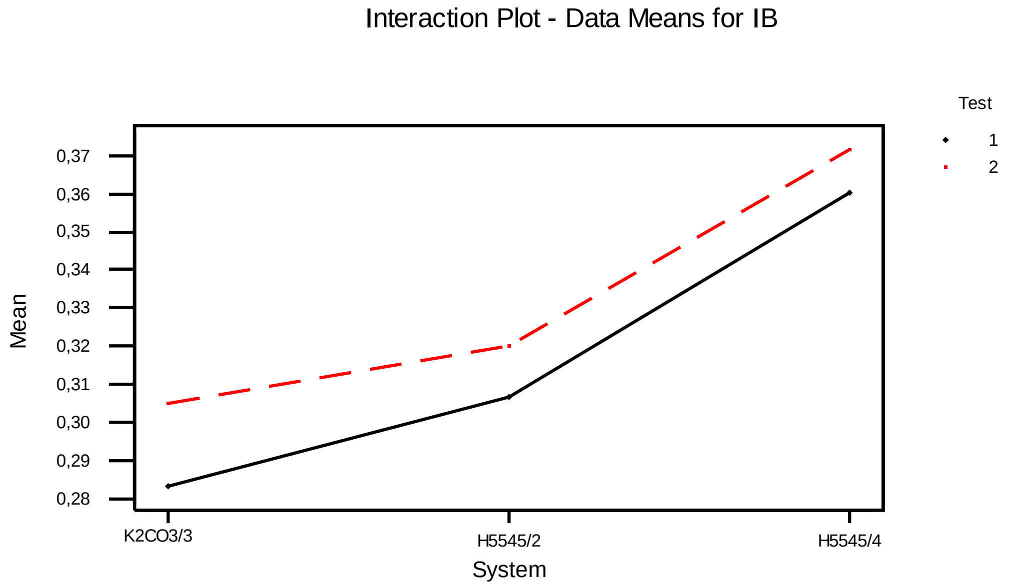
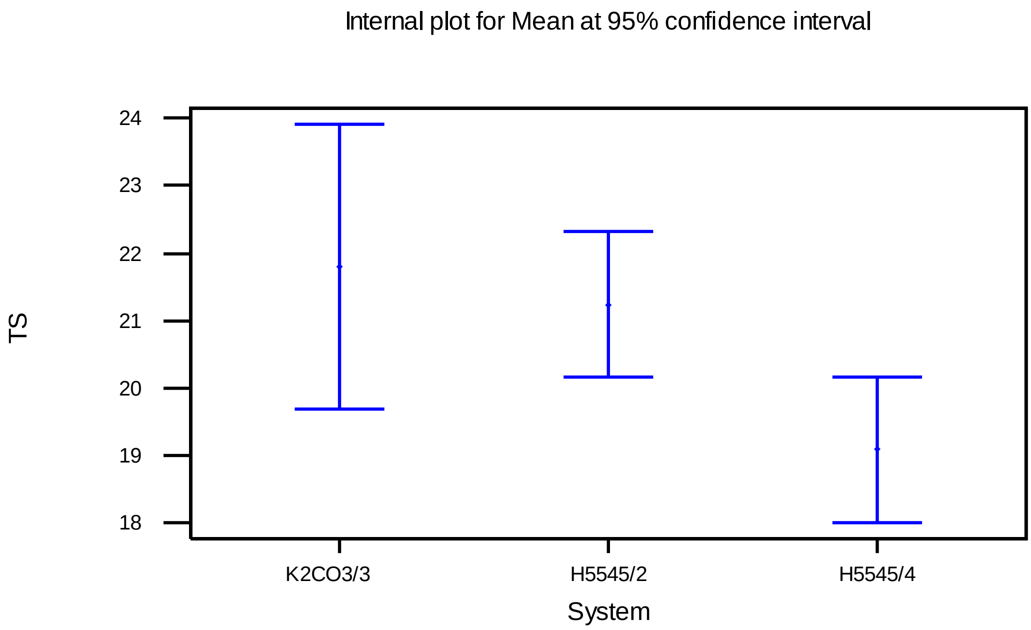
0,345
0,330
0,315
0,300
IB
K2CO3/3
H5545/2
H5545/4
1
2

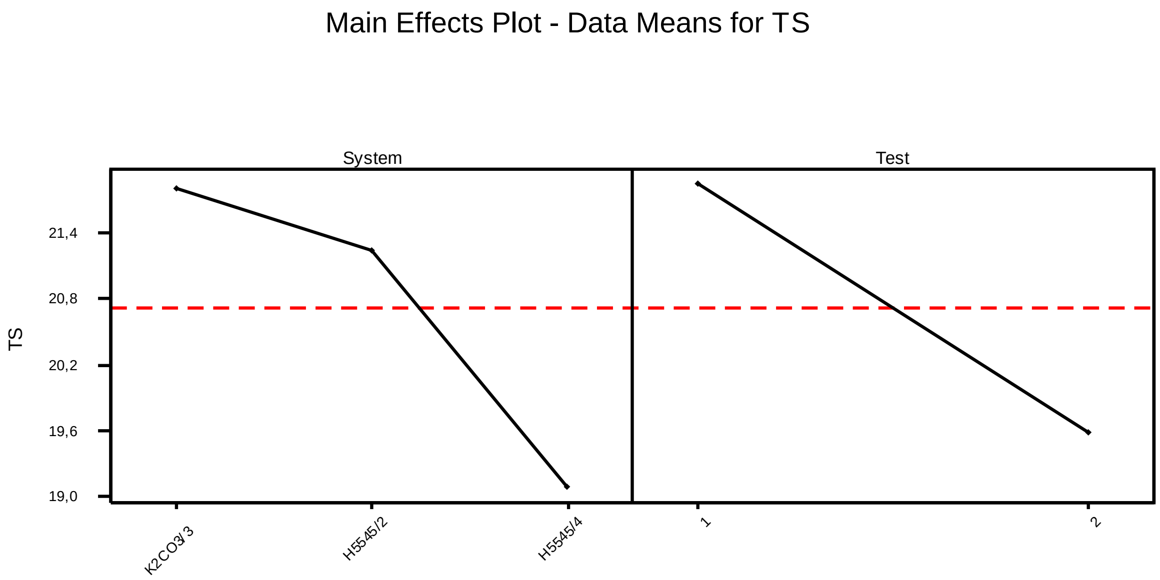


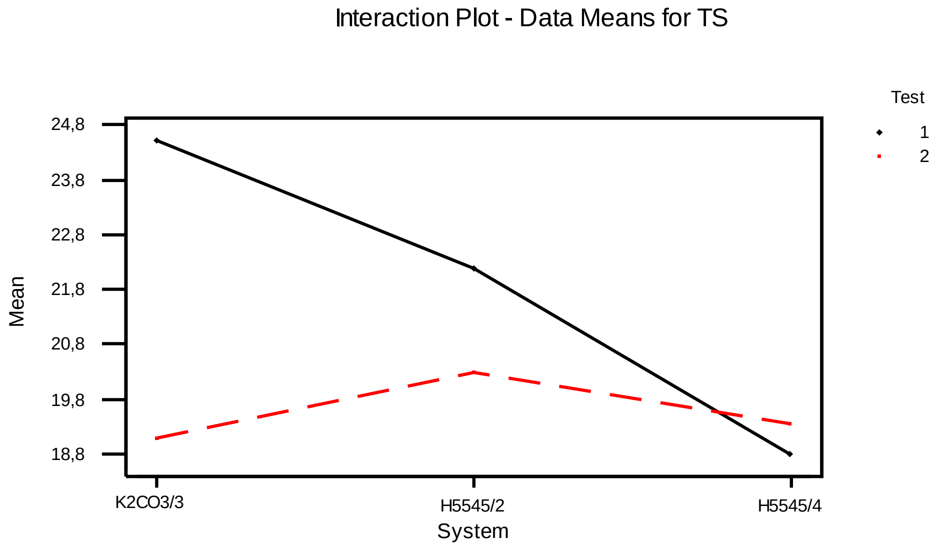
Analysis of Variance for TS, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
System	2	49,415	49,415	24,707	8,84	0,001
Test	1	46,353	46,353	46,353	16,58	0,000
System*Test	2	54,305	54,305	27,152	9,71	0,001
Error	30	83,891	83,891	2,796		
Total	35	233,964				

For the 24h swelling (TS) of the panels both factors examined, the system and the number of test, **are significant** at 95% confidence interval and there is interaction between them, P<0.050.





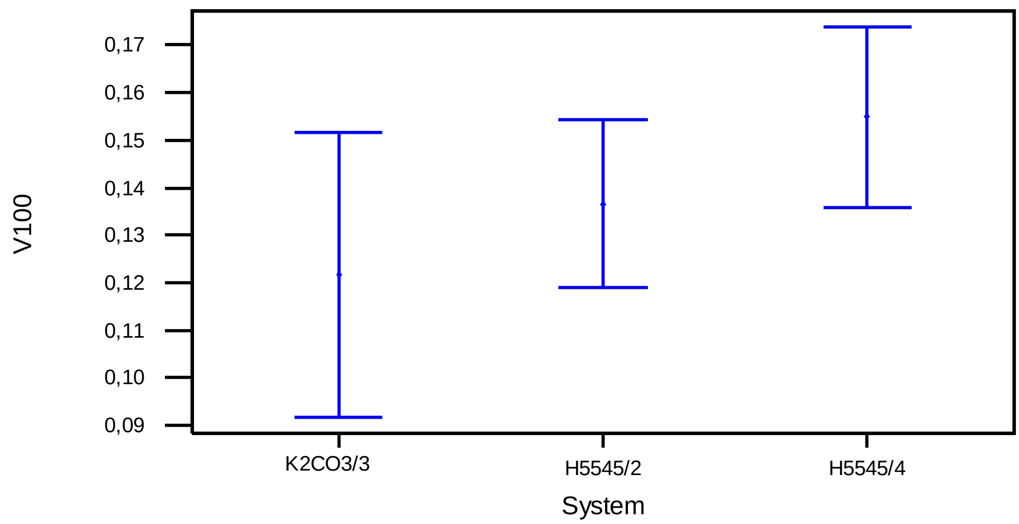


Analysis of Variance for V100, using Adjusted SS for Tests

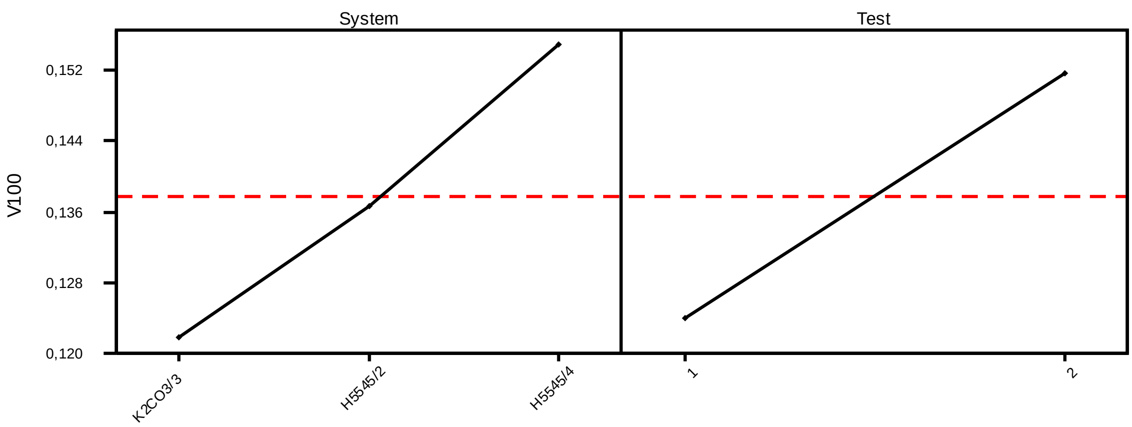
Source	DF	Seq SS	Adj SS	Adj MS	F	P
System	2	0,006689	0,006689	0,003344	2,77	0,079
Test	1	0,006944	0,006944	0,006944	5,75	**0,023**
System*Test	2	0,000156	0,000156	0,000078	0,06	0,938
Error	30	0,036233	0,036233	0,001208		
Total	35	0,050022				

At 95% confidence interval only the number of test **is significant** factor for the V100 of the panels tested and there is no interaction between the system tested and the number of test.

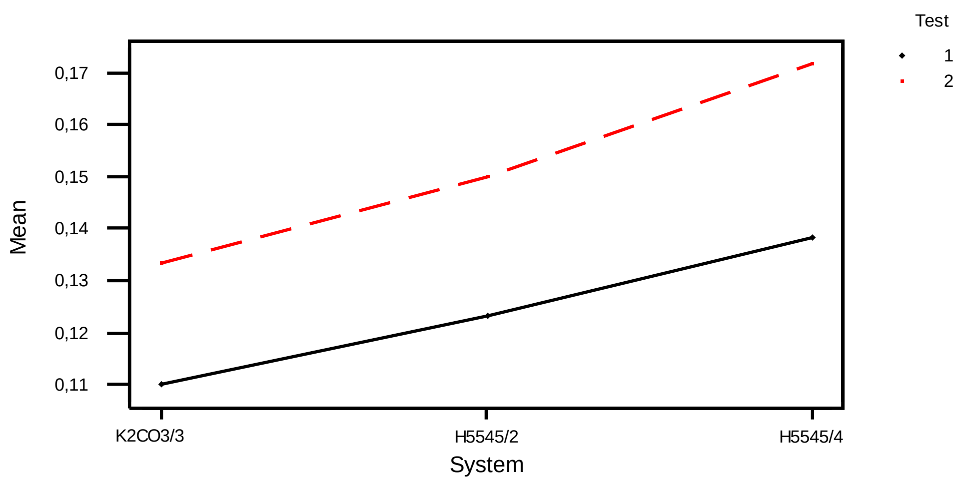
Internal plot for Mean at 95% confidence interval



Main Effects Plot - Data Means for V100



Interaction Plot - Data Means for V100

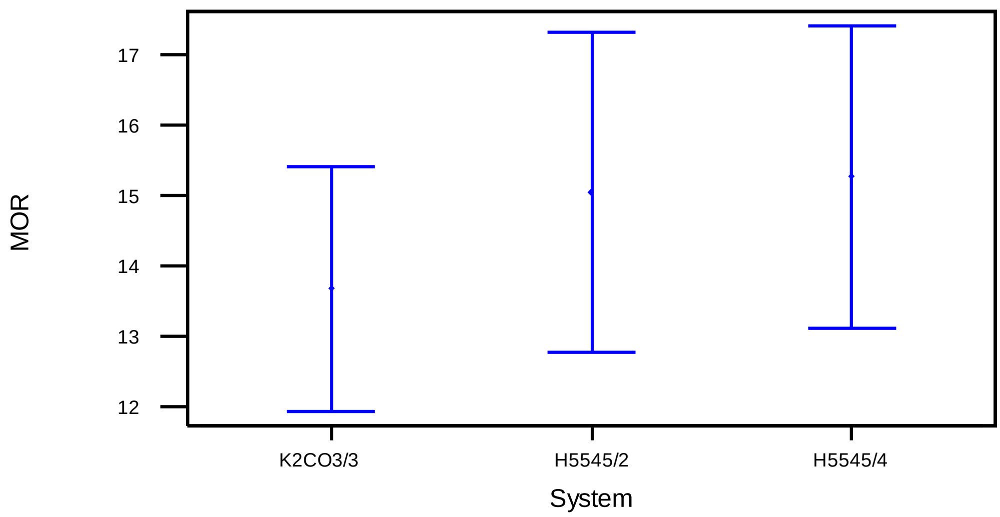


Analysis of Variance for MOR, using Adjusted SS for Tests

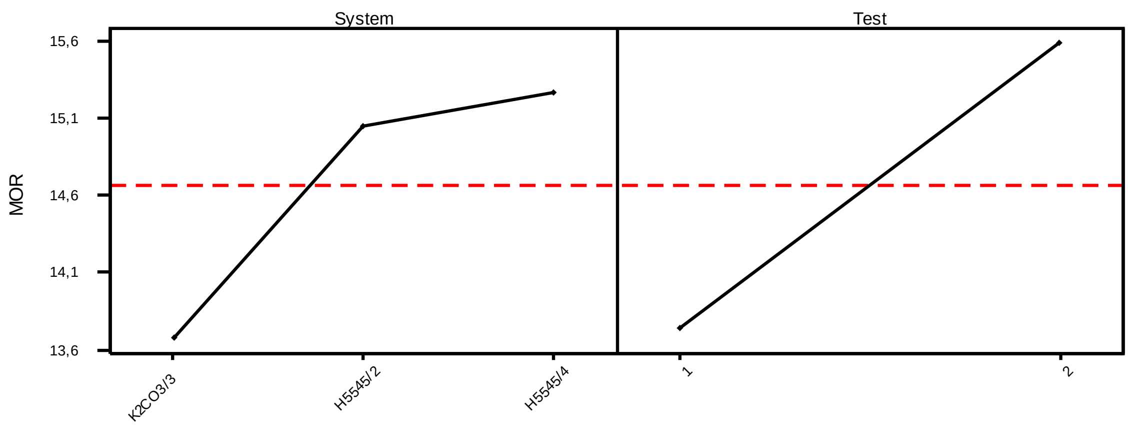
Source	DF	Seq SS	Adj SS	Adj MS	F	P
System	2	5,9621	5,9621	2,9810	3,95	0,081
Test	1	10,2490	10,2490	10,2490	13,57	**0,010**
System*Test	2	0,4595	0,4595	0,2297	0,30	0,748
Error	6	4,5315	4,5315	0,7553		
Total	11	21,2021				

ANOVA performed on the MOR values shows that only the number of test **is significant** at 95% confidence interval and there is no interaction between the system used and the test number, P<0.050.

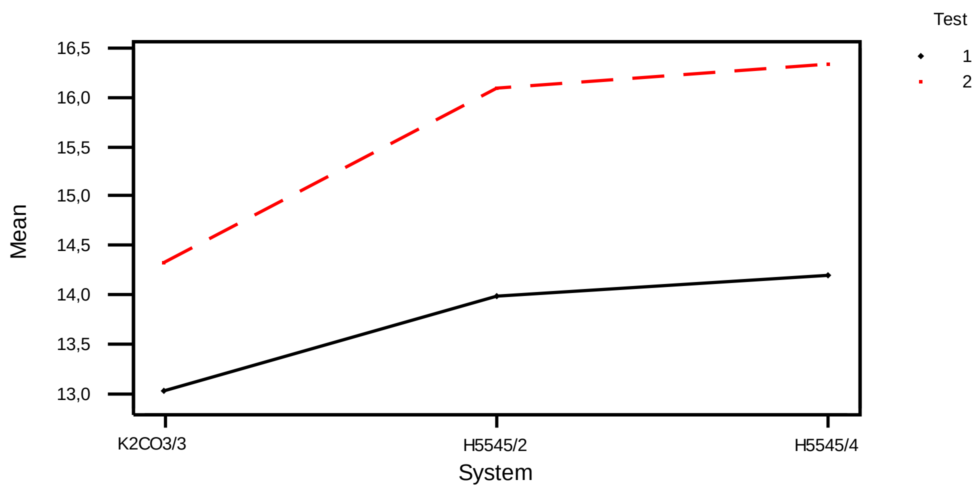
Internal plot for Mean at 95% confidence interval



Main Effects Plot - Data Means for MOR



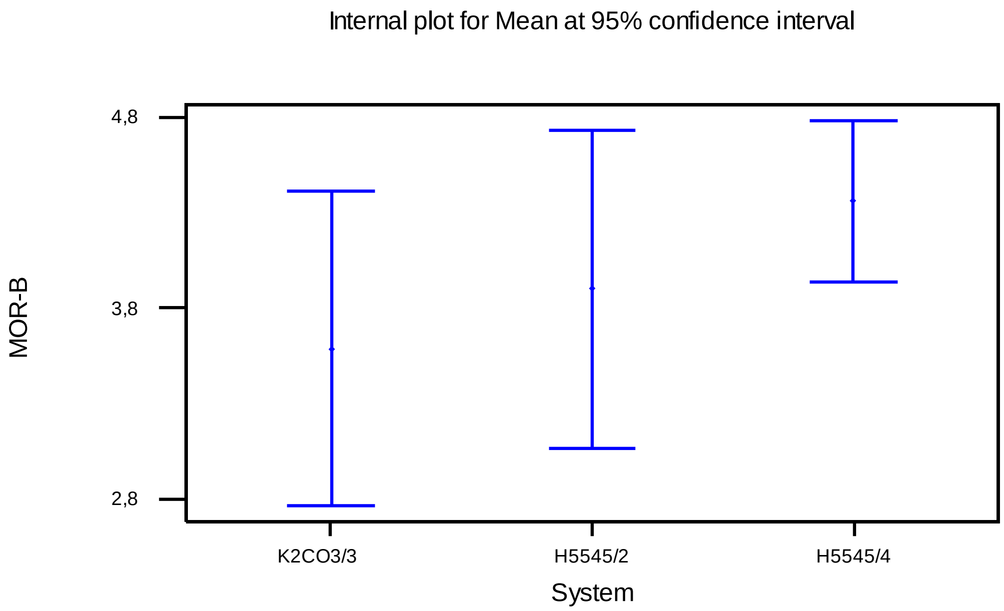
Interaction Plot - Data Means for MOR

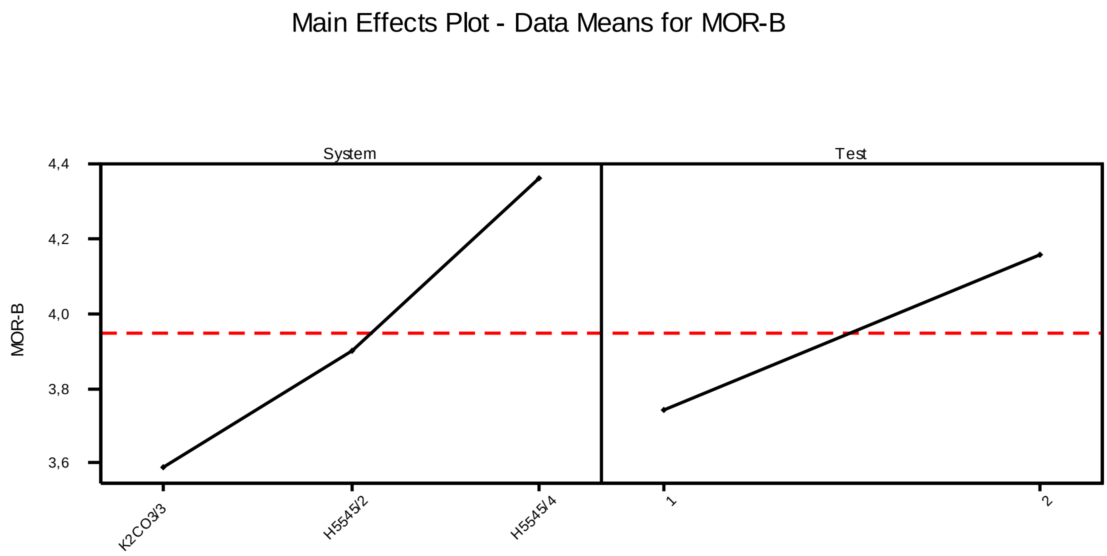


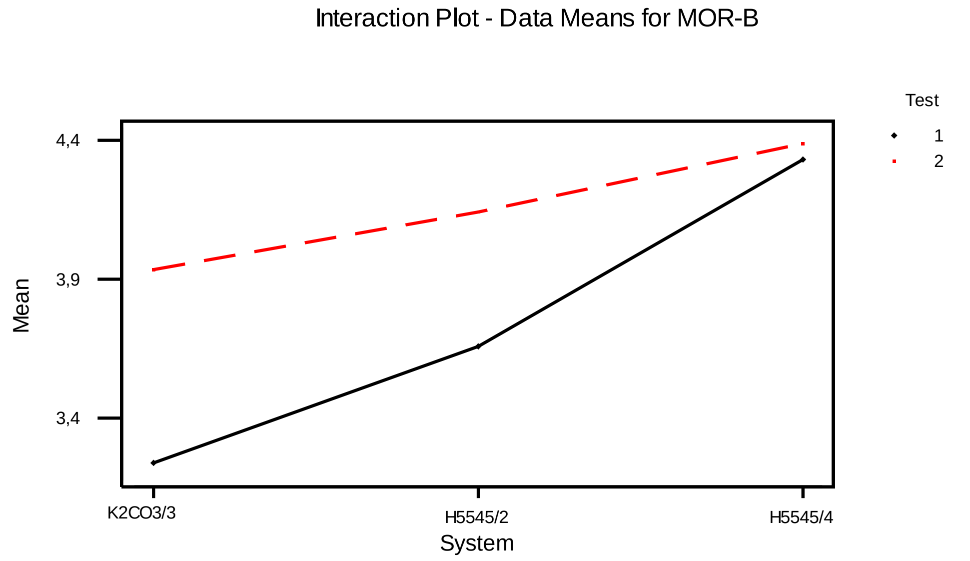
Analysis of Variance for MOR-B, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
System	2	1,2235	1,2235	0,6118	3,25	0,111
Test	1	0,5084	0,5084	0,5084	2,70	0,151
System*Test	2	0,2150	0,2150	0,1075	0,57	0,593
Error	6	1,1296	1,1296	0,1883		
Total	11	3,0765				

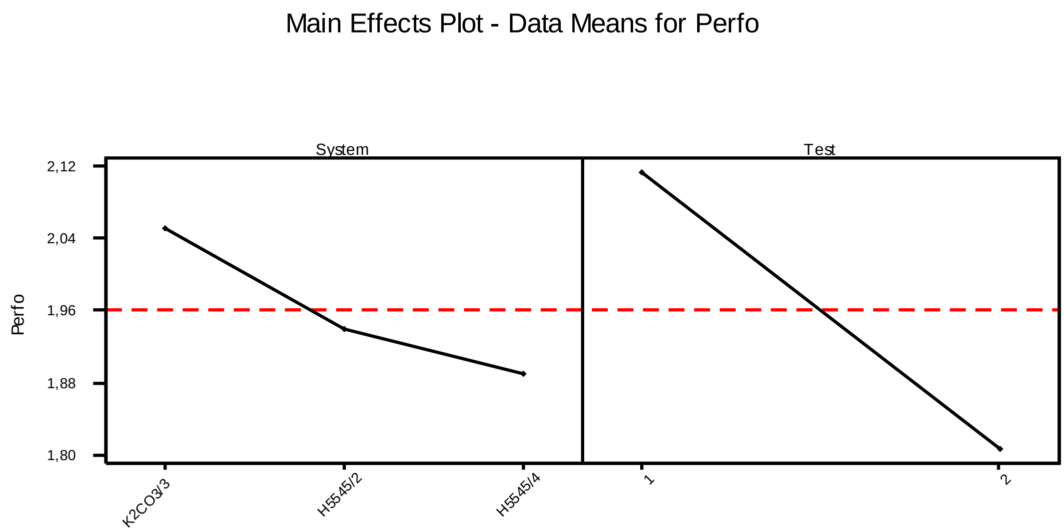
None of the factors examined is significant for the MOR-B values of the LSB panels at 95% confidence interval and there is no interaction between them.


Internal plot for Mean at 95% confidence interval
4,8
3,8
2,8
MOR-B
K2CO3/3
H5545/2
H5545/4
System


Main Effects Plot - Data Means for MOR-B
System
Test
4,4
4,2
4,0
3,8
3,6
MOR-B
K2CO3/3
H5545/2
H5545/4
1
2



The formaldehyde content values were not sufficient for statistical analysis, therefore only the main effect plot was drawn.



The testing results and the ANOVA performed on them indicate that there is statistically significant difference only for internal bond and thickness swelling of the LBS panels, however only when a special hardener was used at 4.0% level dry on dry resin, panel properties meeting the standard requirements were obtained. Generally speaking, the panels produced during the second series of trials showed better results.

Therefore, in the following trials the system of above PF resin together with hardener H5545 at 4% addition level was applied in the production of light weight OSB panels. The effect of reduced panel density in the range of 450-500kg/m^3 on the properties of the panels, while keeping the glue factor constant was first examined. As just mentioned the same type PF resin used in the previous trials was prepared and used for the lab panel production. The panel production details and settings are summarised in the next table.

Table 6: *OSB Production details*

Board Dimensions, cm	43 x 43 x 1.6
Target Density, kg/m^3	500, 475, 450
Target board moisture, %	6.5
Press Factor, s/mm	12
Press Temperature, ^{o}C	180^{o}C
Wood Source	Poplar IBL/IMAL
Strands moisture, %	6.0-7.0
Resin Factor, % solid on dry wood	6.0
Paraffin Level, % solid on dry wood	1.0
Hardener Type and Level, % solid on solid resin	H5545 / 4.0
Target mat moisture, %	10.5

Two sets of panels were produced using the above mentioned settings and the panel properties were measured and are summarised in the two following tables.

Table 7: *OSB Testing results - set 1*

Target density, kg/m^3	500	475	450
Thickness, mm	16.08	16.03	16.01
Density, kg/m^3	502	468	453
IB, MPa	0.39	0.31	0.20
V100, MPa	0.14	0.11	0.07
TS, %	21.20	18.96	16.87
MOR, MPa	12.36	13.20	10.66
MOR-B, MPa	4.23	3.03	3.65
Perforator, mg/100g	1.85	2.11	2.98

Table 8: *OSB Testing results - set 2*

Target density, kg/m^3	500	475	450
Thickness, mm	16.12	16.04	16.10
Density, kg/m^3	503	474	460
IB, MPa	0.40	0.27	0.23
V100, MPa	0.15	0.14	0.12
TS, %	24.15	21.60	19.04
MOR, MPa	13.62	12.43	11.31
MOR-B, MPa	4.33	3.33	2.74
Perforator, mg/100g	1.65	2.31	1.97

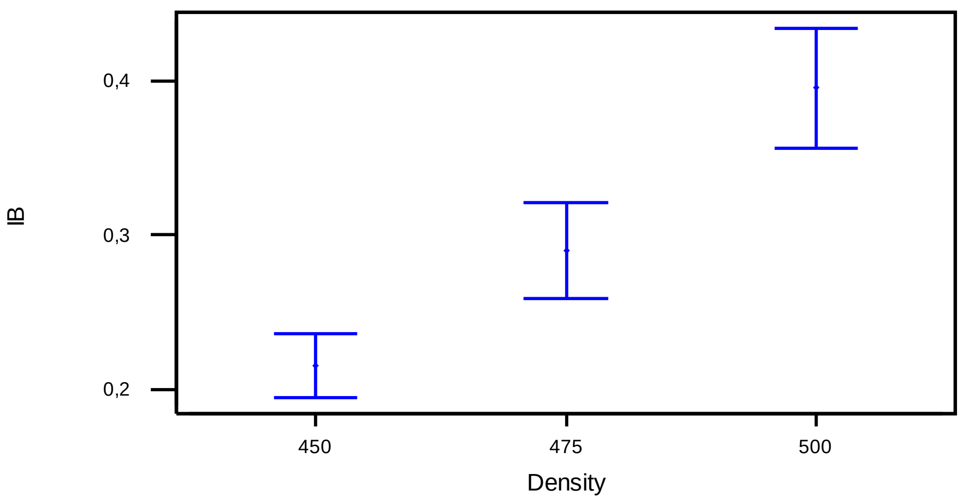
The results obtained from the ANOVA that was performed on the panel testing values are the following:

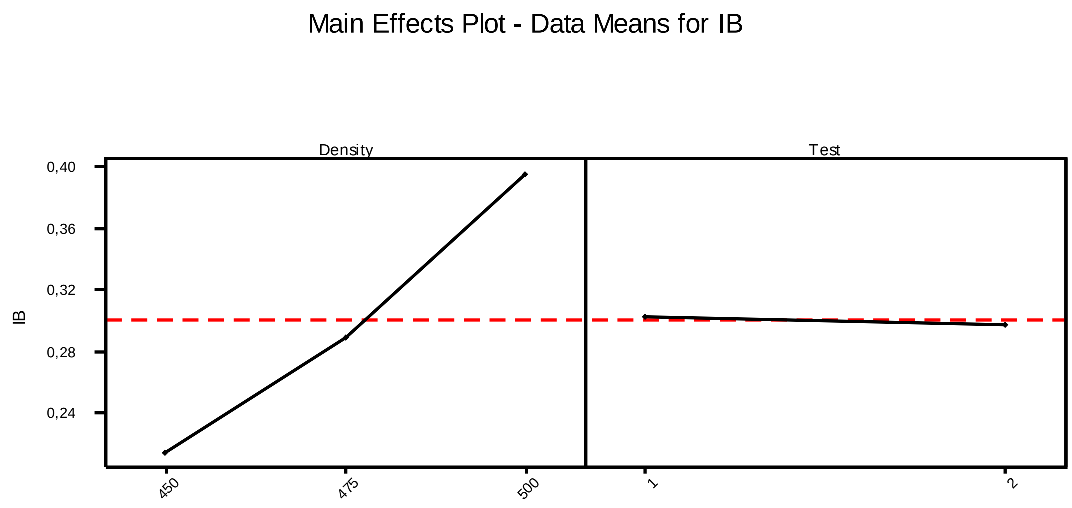
Analysis of Variance for IB, using Adjusted SS for Tests

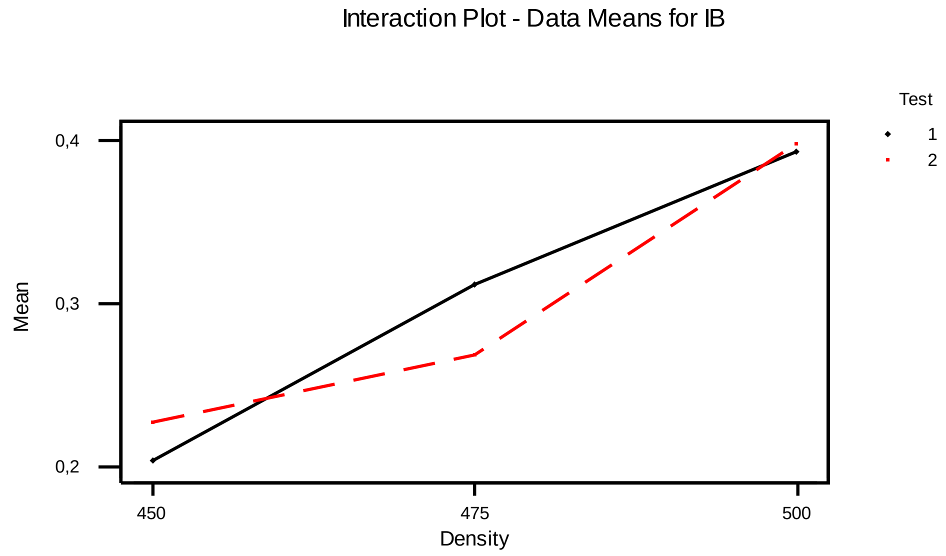
Source	DF	Seq SS	Adj SS	Adj MS	F	P
Density	2	0,198106	0,198106	0,099053	41,02	**0,000**
Test	1	0,000225	0,000225	0,000225	0,09	0,762
Density*Test	2	0,007117	0,007117	0,003558	1,47	0,245
Error	30	0,072450	0,072450	0,002415		
Total	35	0,277897				

For the IB of the LBS panels produced, only the density **is significant** factor at 95% confidence interval, P<0.050, and there is no interaction with the number of test.

Internal plot for Mean at 95% confidence interval



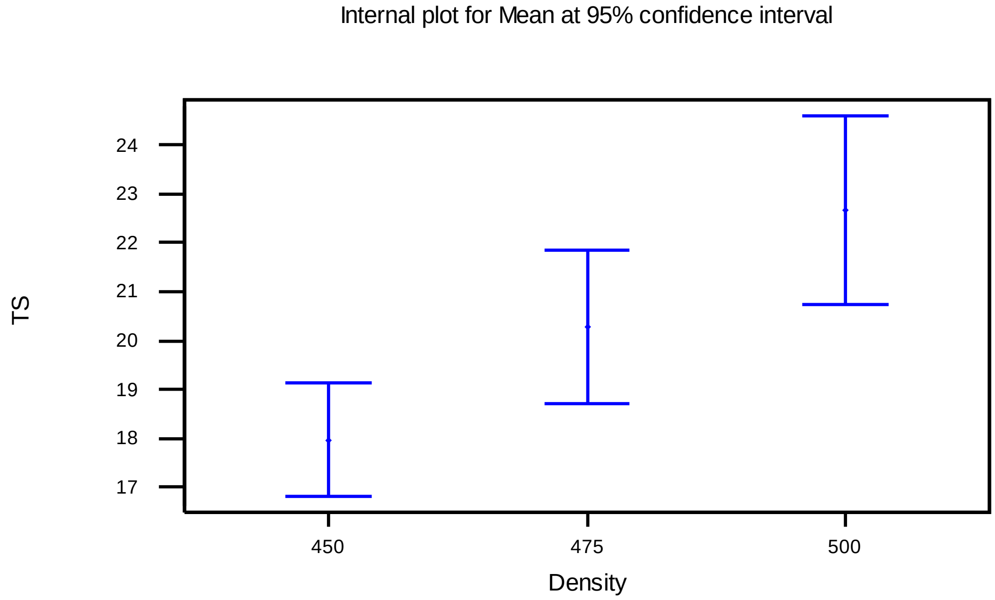


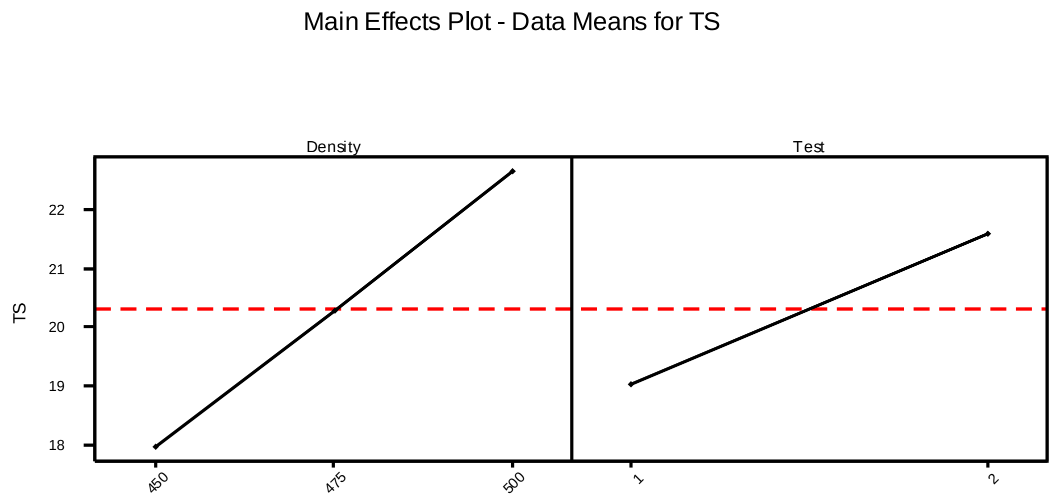


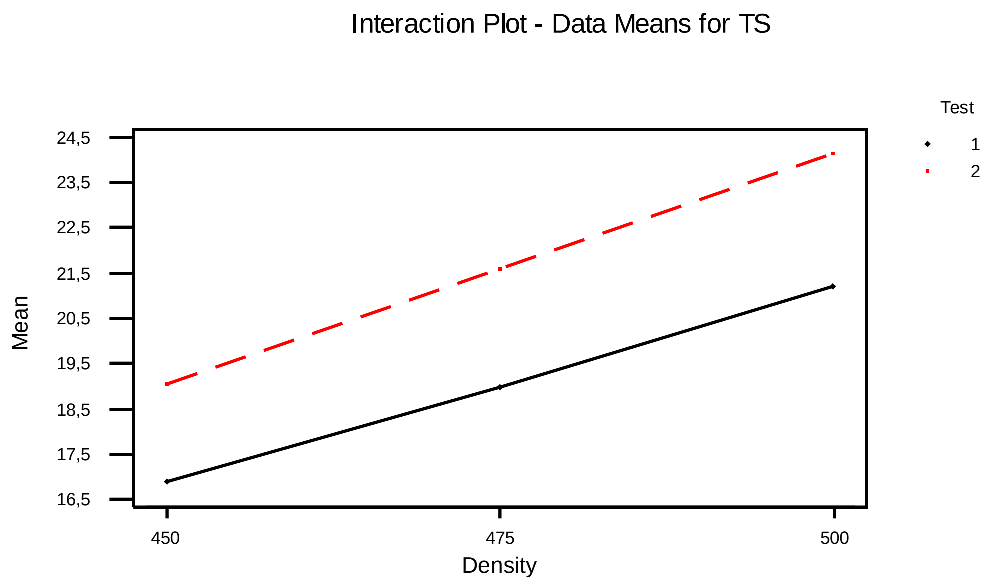
Analysis of Variance for TS, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Density	2	133,774	133,774	66,887	13,68	**0,000**
Test	1	60,140	60,140	60,140	12,30	**0,001**
Density*Test	2	0,910	0,910	0,455	0,09	0,911
Error	30	146,673	146,673	4,889		
Total	35	341,497				

Both factors examined, the density of panels and the number of test, **are significant** at 95% confidence interval for the TS, but there is no interaction between them.

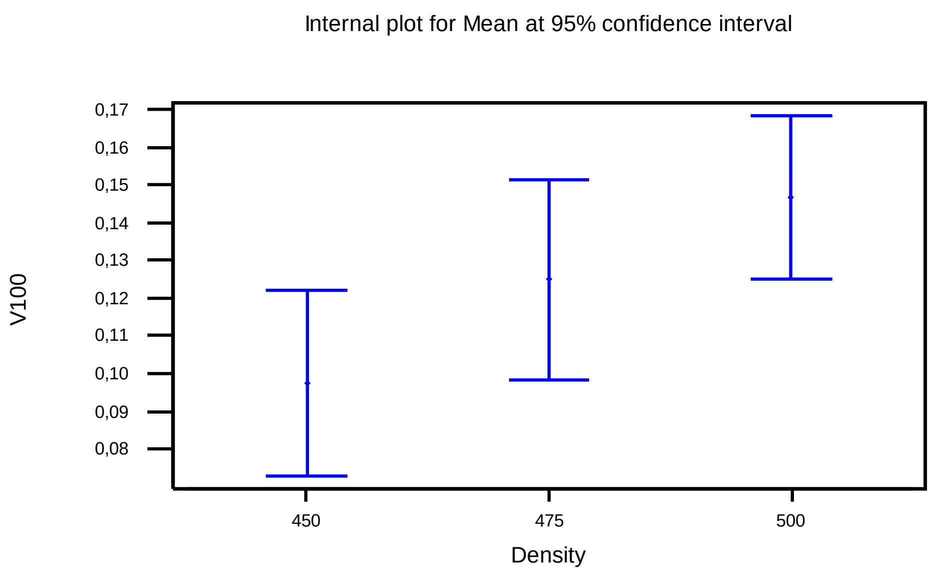

Internal plot for Mean at 95% confidence interval
TS
24
23
22
21
20
19
18
17
450
475
500
Density

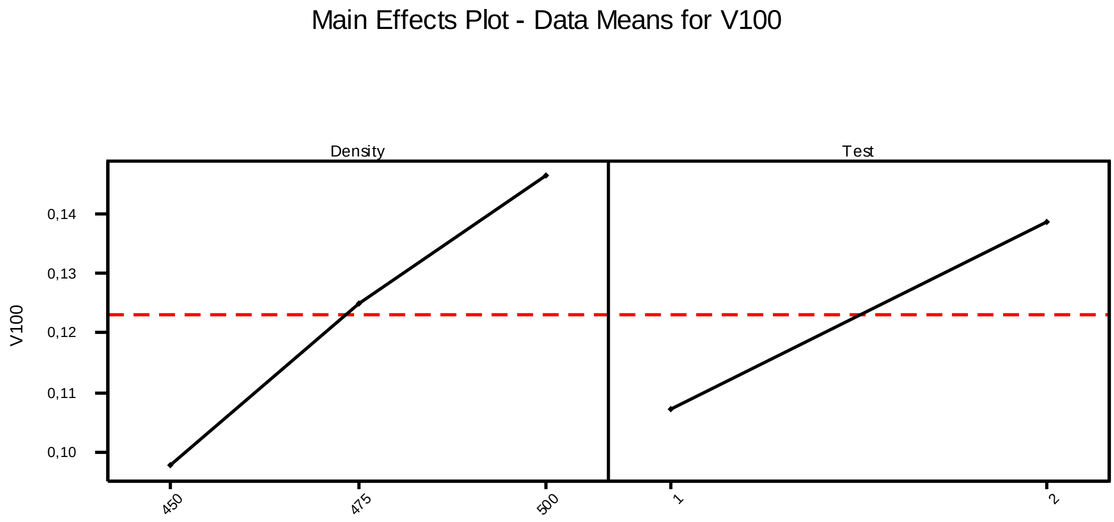

Main Effects Plot - Data Means for TS
Density
Test
TS
22
21
20
19
18
450
475
500
1
2

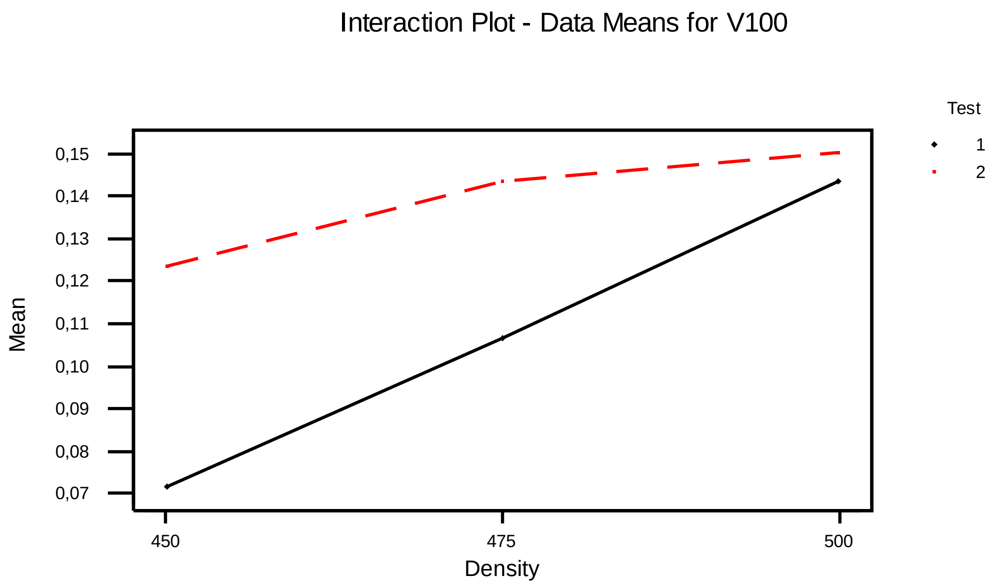


Analysis of Variance for V100, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Density	2	0,014572	0,014572	0,007286	6,04	**0,006**
Test	1	0,009025	0,009025	0,009025	7,48	**0,010**
Density*Test	2	0,003150	0,003150	0,001575	1,30	0,286
Error	30	0,036217	0,036217	0,001207		
Total	35	0,062964				





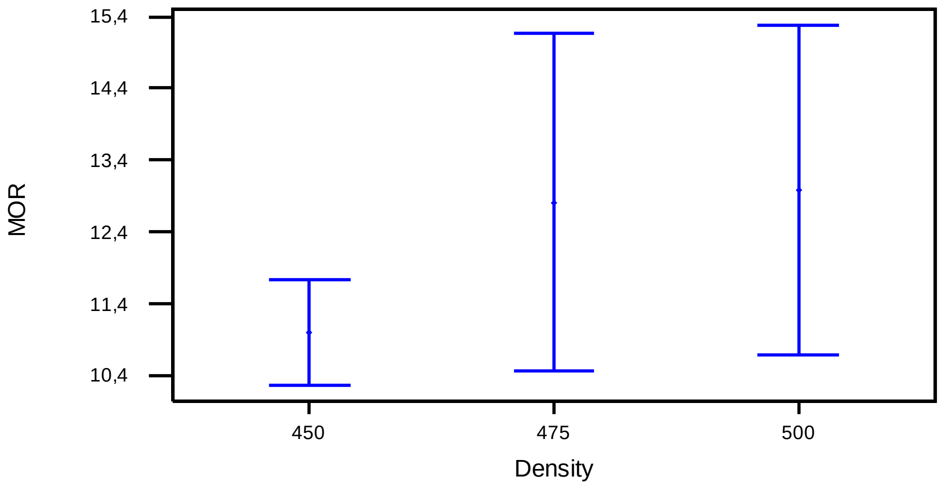


Analysis of Variance for MOR, using Adjusted SS for Tests

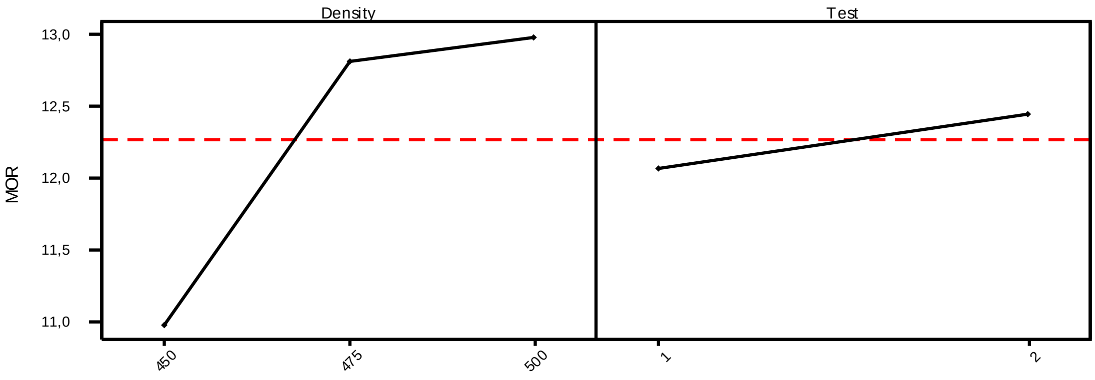
Source	DF	Seq SS	Adj SS	Adj MS	F	P
Density	2	9,877	9,877	4,939	2,68	0,147
Test	1	0,426	0,426	0,426	0,23	0,648
Density*Test	2	2,158	2,158	1,079	0,59	0,585
Error	6	11,046	11,046	1,841		
Total	11	23,507				

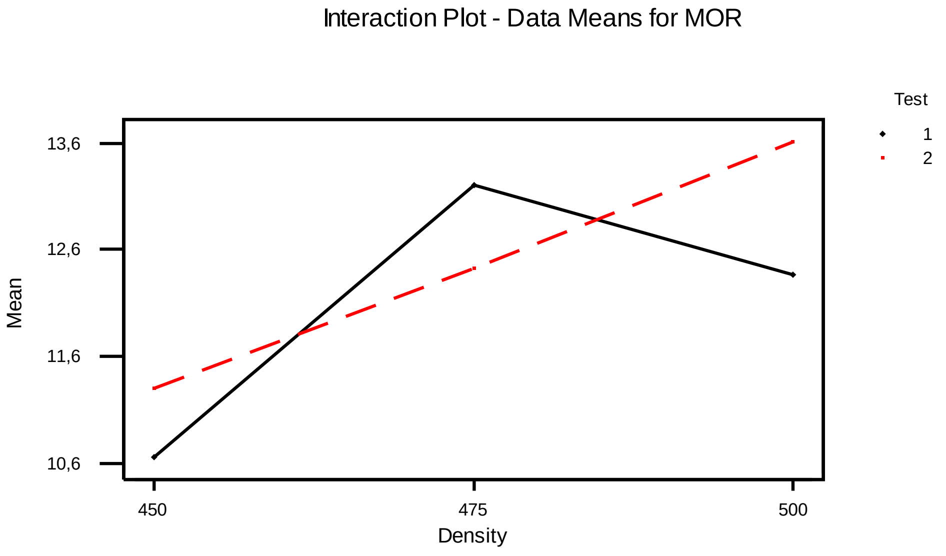
None of the factors examined is significant at 95% confidence interval of the MOR and there is no interaction between them.

Internal plot for Mean at 95% confidence interval



Main Effects Plot - Data Means for MOR

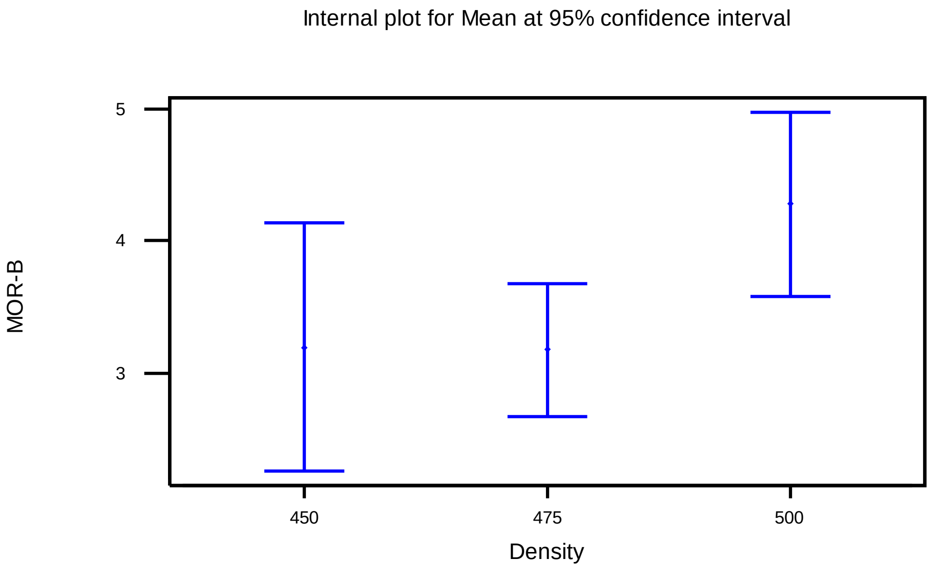


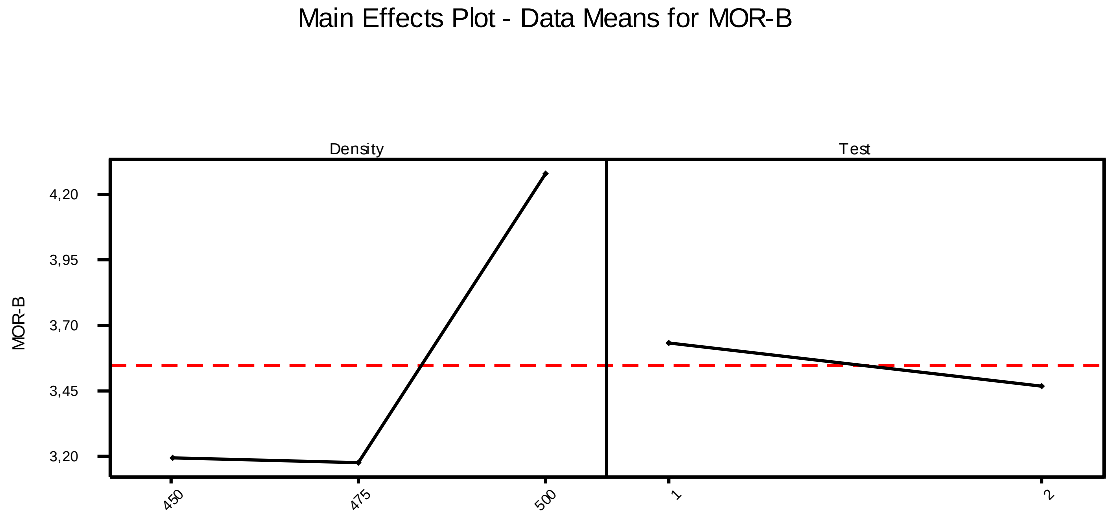


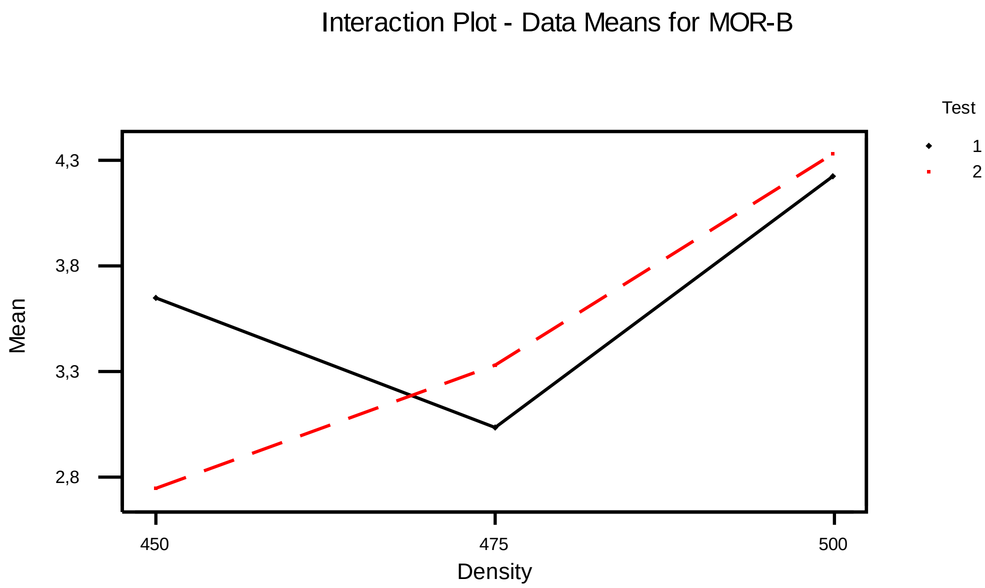
Analysis of Variance for MOR-B, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Density	2	3,1833	3,1833	1,5916	9,35	**0,014**
Test	1	0,0850	0,0850	0,0850	0,50	0,506
Density*Test	2	0,8321	0,8321	0,4160	2,44	0,167
Error	6	1,0210	1,0210	0,1702		
Total	11	5,1213				

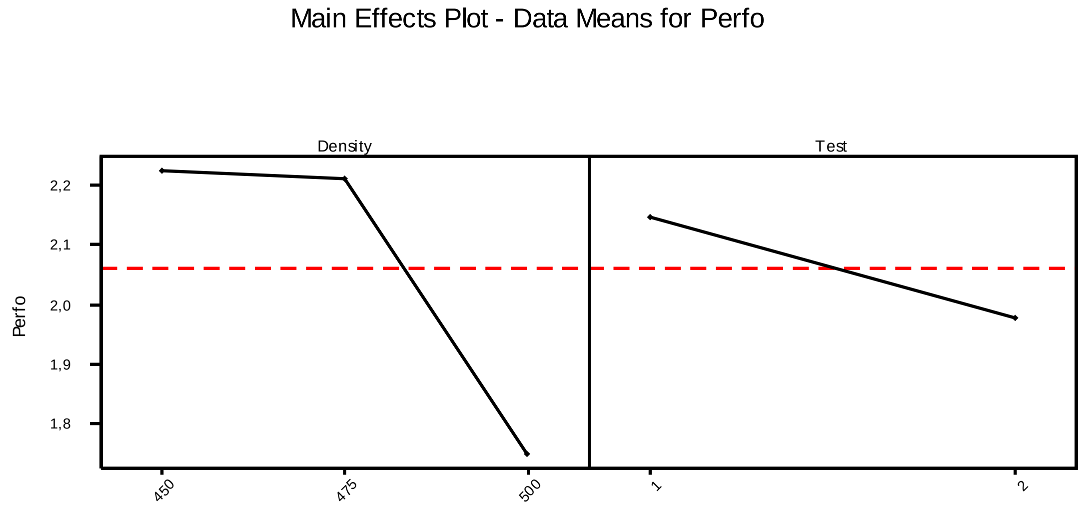
Only density of panels **is significant** factor at 95% confidence interval for the MOR-B and there is no interaction with the number of trial.







As the formaldehyde content values of the panels measured were not sufficient for statistical analysis, only the main effect plot was drawn.



The results obtained and the statistical analysis (ANOVA) performed on them indicate that the mechanical properties of the LSB panels worsen when the density is reduced. The lower the density of panels is, the worse the properties become. In order to retain the properties of the LSB panels, the resin loading factor was stepwise increased in the next trials. The H5545 hardener was added to the PF resin at 4.0% dry on dry level, while the panel production conditions were the same as in the previous trials. The LSB lab production details are shown in the next table.

Table 9: *OSB Production details*

Board Dimensions, cm	43 x 43 x 1.6		
Target Density, kg/m^3	500	475	450
Resin Factor, % solid on dry wood	6	7	8
Target board moisture, %	6.5		
Press Factor, s/mm	12		
Press Temperature, °C	180°C		
Wood Source	Poplar IBL/IMAL		
Strands moisture, %	6.0-7.0		
Paraffin Level, % solid on dry wood	1.0		
Hardener Type and Level, % solid on solid resin	H5545 / 4.0		
Target mat moisture, %	10.5-11.5		

Two sets of panels were again produced, their properties were determined and the testing results are summarised in the tables below.

Table 10: *OSB Testing results - set 1*

Target density, kg/m³	500	475	450
Resin factor, % dry/dry wood	6.0	7.0	8.0
Thickness, mm	16.10	16.07	16.13
Density, kg/m³	507	473	454
IB, MPa	0.36	0.35	0.30
V100, MPa	0.18	0.19	0.16
TS, %	20.19	19.18	21.96
MOR, MPa	9.67	11.17	13.04
MOR-B, MPa	3.83	3.95	5.16
Perforator, mg/100g	2.40	2.71	2.86

Table 11: *OSB Testing results – set 2*

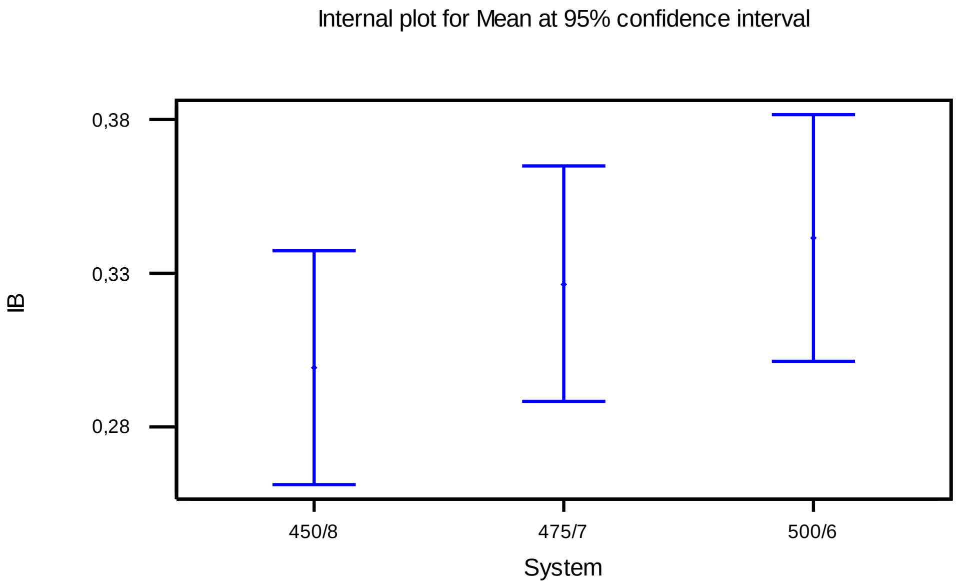
Target density, kg/m³	500	475	450
Resin factor, % dry/dry wood	6.0	7.0	8.0
Thickness, mm	16.11	16.18	16.24
Density, kg/m³	500	480	454
IB, MPa	0.32	0.31	0.30
V100, MPa	0.15	0.13	0.17
TS, %	17.79	20.39	21.40
MOR, MPa	12.02	13.71	11.52
MOR-B, MPa	3.76	2.67	3.16
Perforator, mg/100g	2.56	2.13	2.41

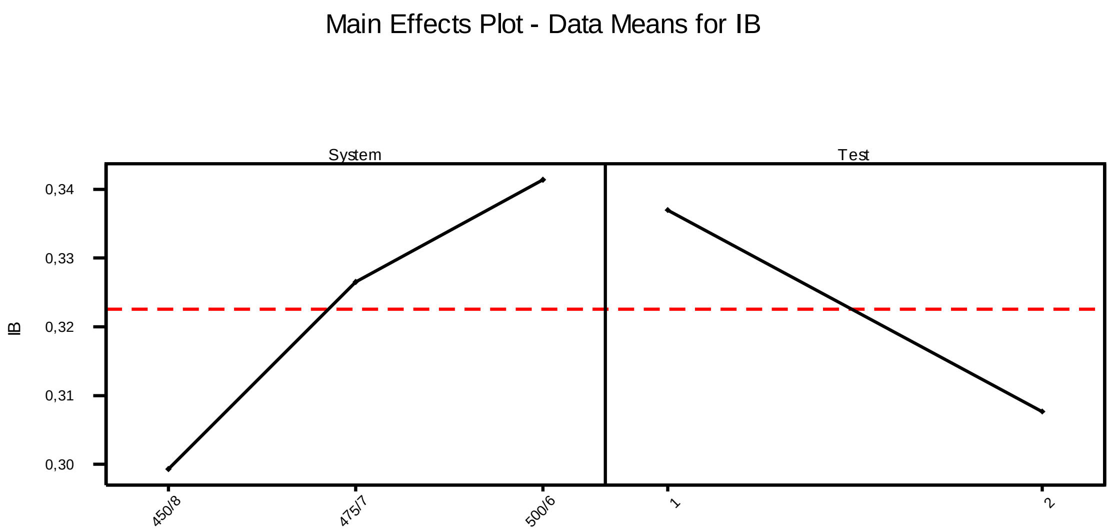
The results from the ANOVA performed on the above property values are as follows.

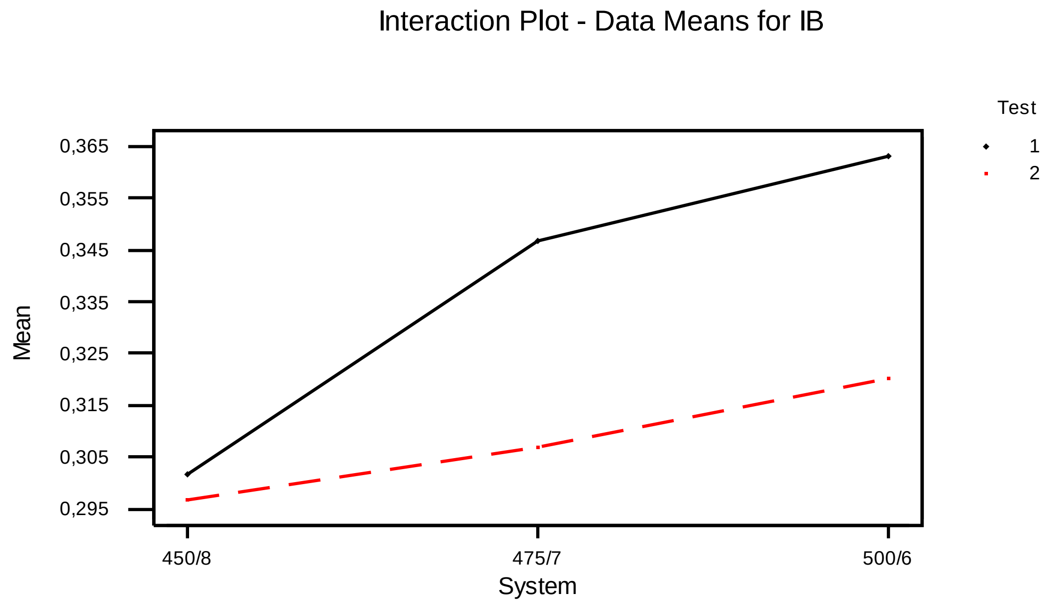
```
Analysis of Variance for IB, using Adjusted SS for Tests

Source        DF     Seq SS      Adj SS      Adj MS       F      P
System         2   0,011150   0,011150   0,005575    1,48  0,243
Test           1   0,007803   0,007803   0,007803    2,07  0,160
System*Test    2   0,002706   0,002706   0,001353    0,36  0,701
Error         30   0,112817   0,112817   0,003761
Total         35   0,134475
```

None of the factors examined is significant for the IB of the panels at 95% confidence interval and there is no interaction between them.

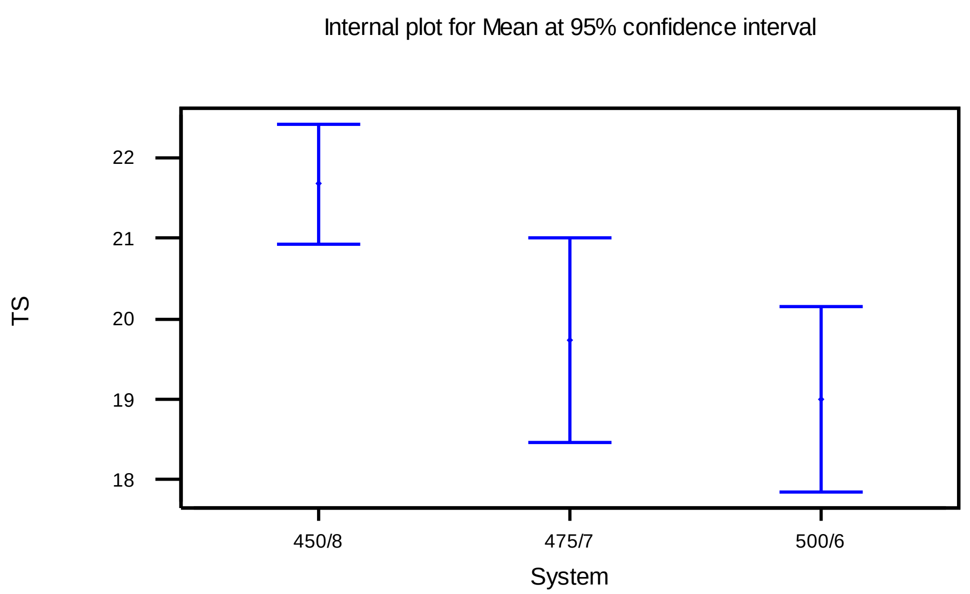

Internal plot for Mean at 95% confidence interval
0,38
0,33
0,28
IB
450/8
475/7
500/6
System

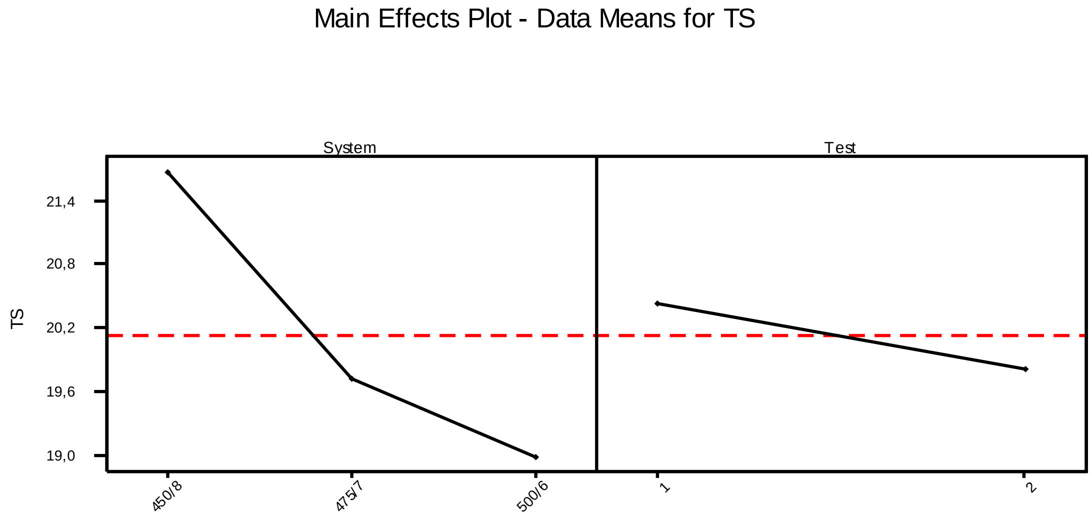

Main Effects Plot - Data Means for IB
System
Test
0,34
0,33
0,32
0,31
0,30
IB
450/8
475/7
500/6
1
2

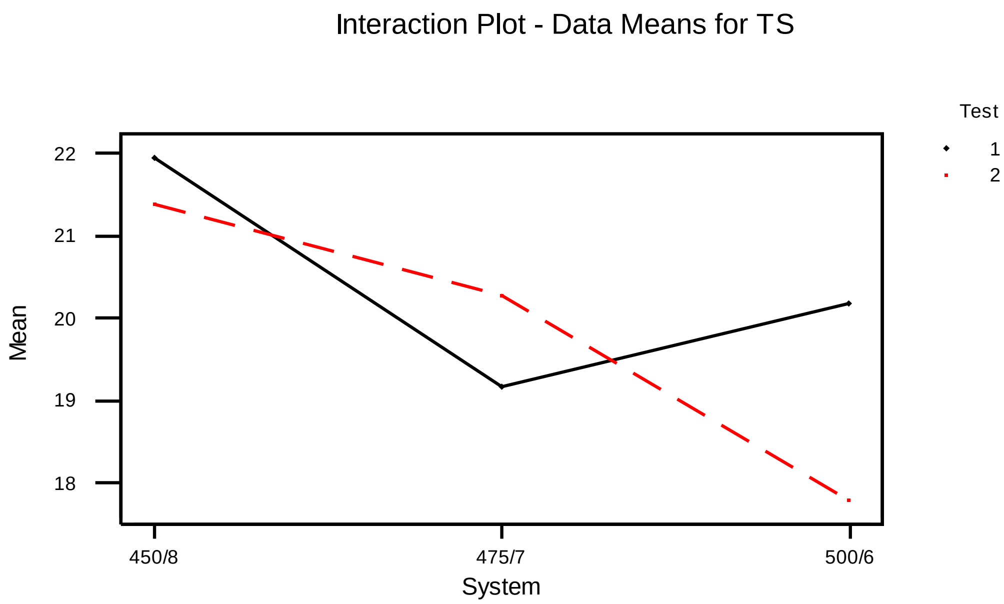


Analysis of Variance for TS, using Adjusted SS for Tests						
Source	DF	Seq SS	Adj SS	Adj MS	F	P
System	2	46,167	46,167	23,084	9,34	0,001
Test	1	3,435	3,435	3,435	1,39	0,248
System*Test	2	18,442	18,442	9,221	3,73	0,036
Error	30	74,145	74,145	2,472		
Total	35	142,189				

The system used for the panels production **is significant** factor at 95% confidence interval for the TS and there is interaction between system and number of trial, P<0.050.



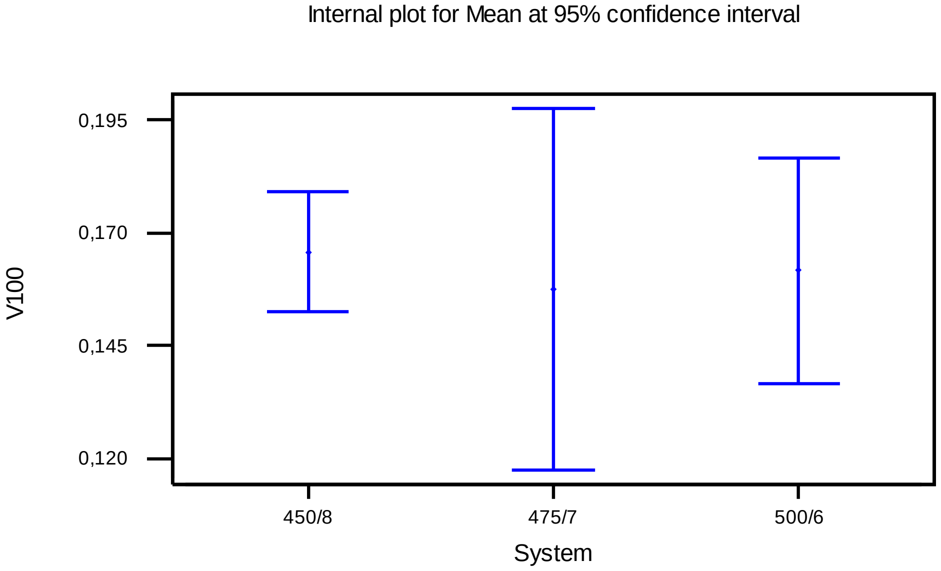


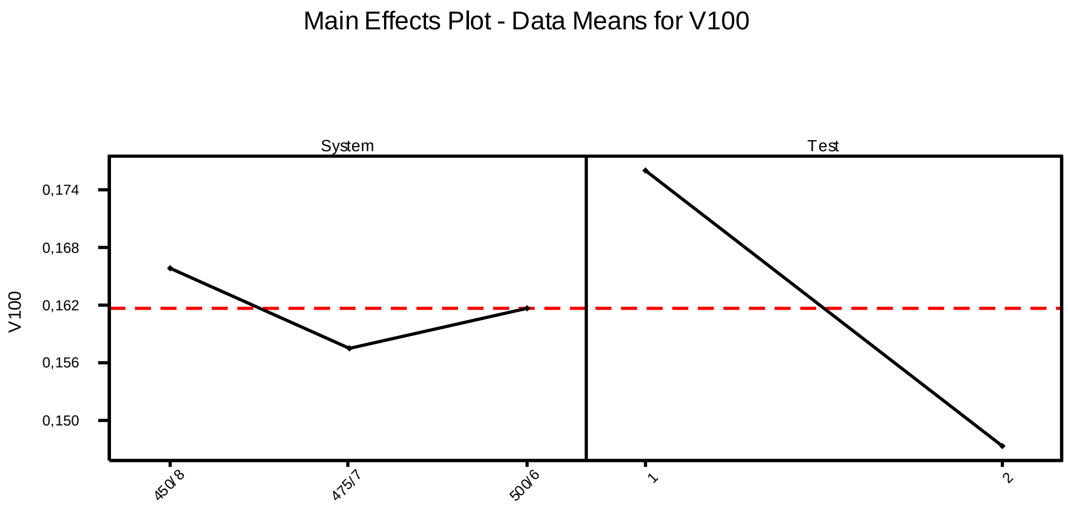


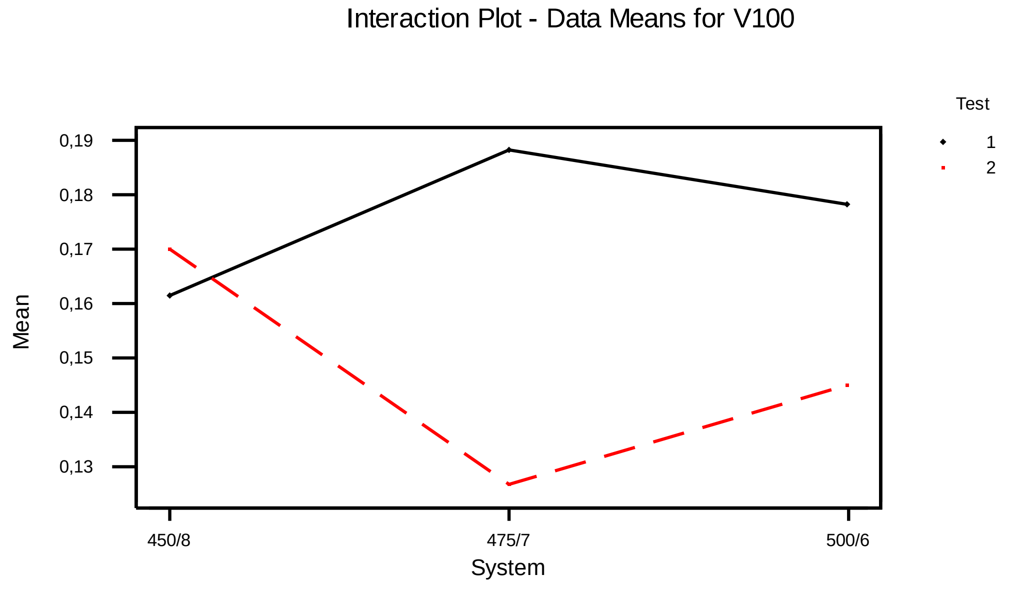
Analysis of Variance for V100, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
System	2	0,000417	0,000417	0,000208	0,12	0,886
Test	1	0,007511	0,007511	0,007511	4,39	**0,045**
System*Test	2	0,007439	0,007439	0,003719	2,17	0,131
Error	30	0,051333	0,051333	0,001711		
Total	35	0,066700				

Only the number of test **is significant** at 95% confidence interval for the V100 values of the LSB panels and there is no interaction between the factors examined.


Internal plot for Mean at 95% confidence interval
0,195
0,170
0,145
0,120
V100
450/8
475/7
500/6
System

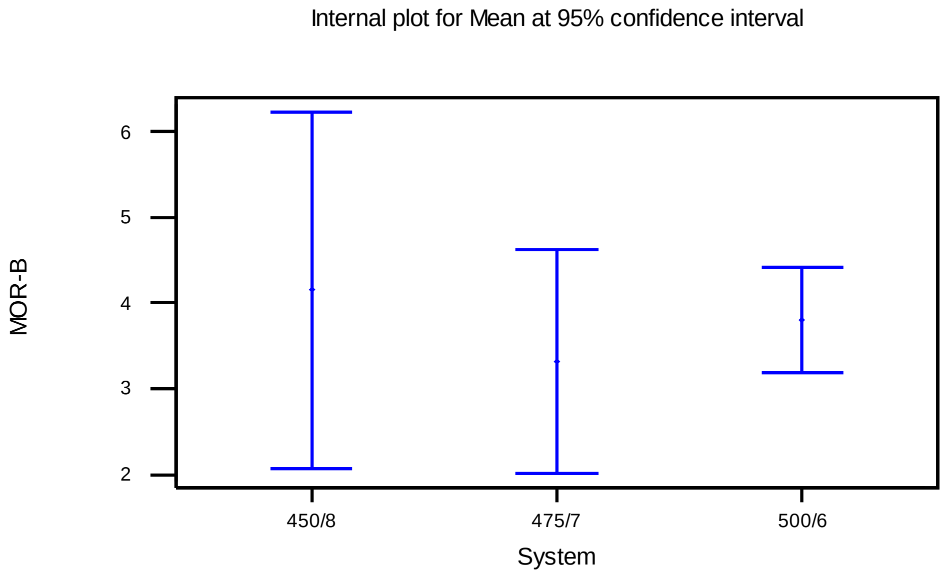

Main Effects Plot - Data Means for V100
System
Test
0,174
0,168
0,162
0,156
0,150
V100
450/8
475/7
500/6
1
2

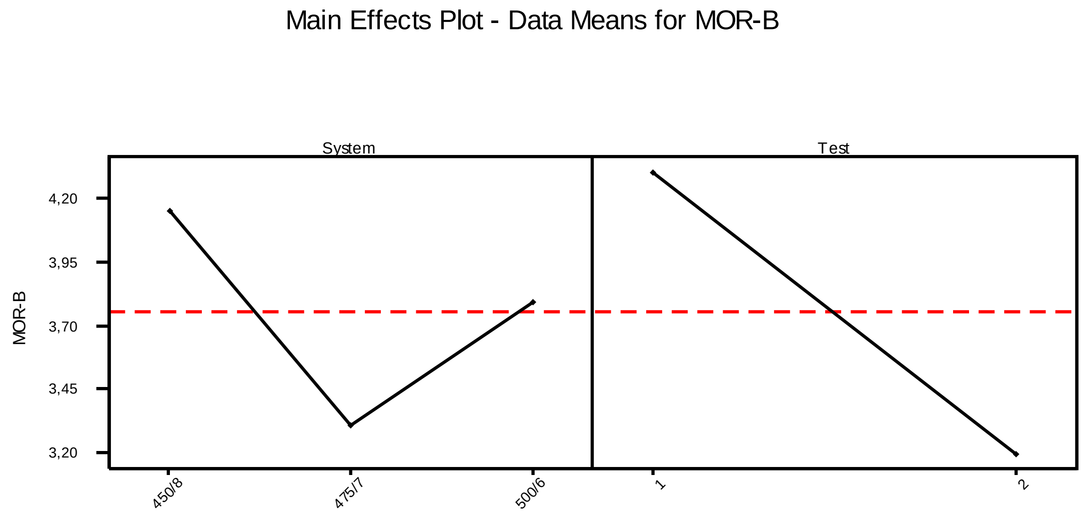


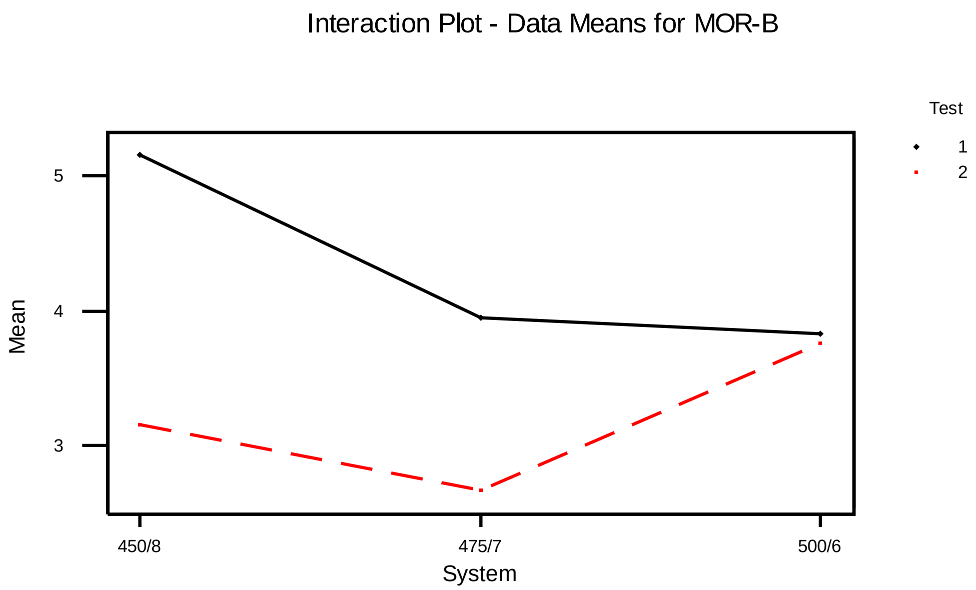
Analysis of Variance for MOR-B, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
System	2	1,4473	1,4473	0,7237	2,17	0,195
Test	1	3,7297	3,7297	3,7297	11,19	**0,016**
System*Test	2	1,9009	1,9009	0,9504	2,85	0,135
Error	6	1,9998	1,9998	0,3333		
Total	11	9,0776				

Only the number of test **is significant** at 95% confidence interval for the MOR-B values of the LSB panels and there is no interaction between the factors examined.

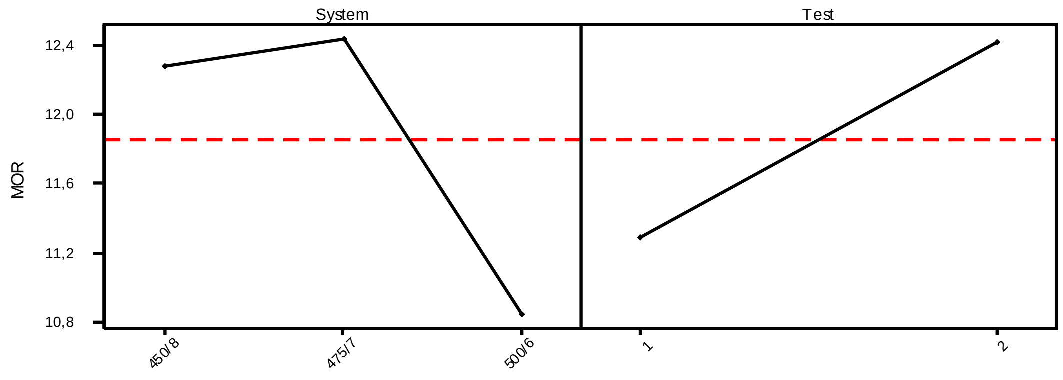




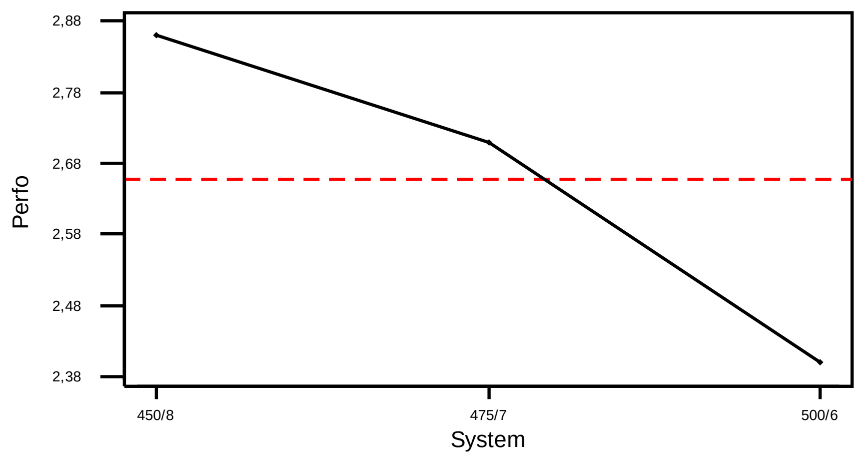


As both the MOR and formaldehyde content values of the panels were not sufficient for ANOVA, only the main effect plots were drawn and are given below.

Main Effects Plot - Data Means for MOR



Main Effects Plot - Data Means for Perfo



The testing results and the ANOVA performed indicate that the increased resin loading can compensate the decrease of the mechanical properties of the LSB panels when the density is reduced from 500 to 450kg/m^3. Evidently the thickness swelling property is affected too, however, only during a pilot or industrial trial either the resin loading or the hardener level could be optimised.

In the framework of WP3, the curing performance of the resins synthesized was studied either alone or in mixtures with additives by means of a Shimadzu DSC-50 Differential Scanning Calorimetry (DSC) apparatus. For the DSC measurements, a sample of approximately 10 mg was placed each time in a special aluminium cell, which was then sealed. Measurements were carried out in a nitrogen atmosphere. The samples were kept at 30°C for 5 min and then heated up to 220°C at a rate of 10°C/min. The special aluminium cells used are capable of withstanding pressures up to 30 bars. Since the sample is placed in a closed cell, water evaporation is suppressed and therefore, the endotherm peak in the DSC scan obtained is absent. Both resin types, MUPF and PF, were used in this study.

The following systems of MUPF resin were examined:

- MUPF – 1, plain resin
- MUPF – 2, resin with 3.5% $(NH_4)_2SO_4$
- MUPF – 3, resin with 3.5% hardener HR2
- MUPF – 4, resin with 3.5% hardener HR2 and crosslinker CR1

The DSC thermograms of the MUPF resin systems are presented in Figure 1.

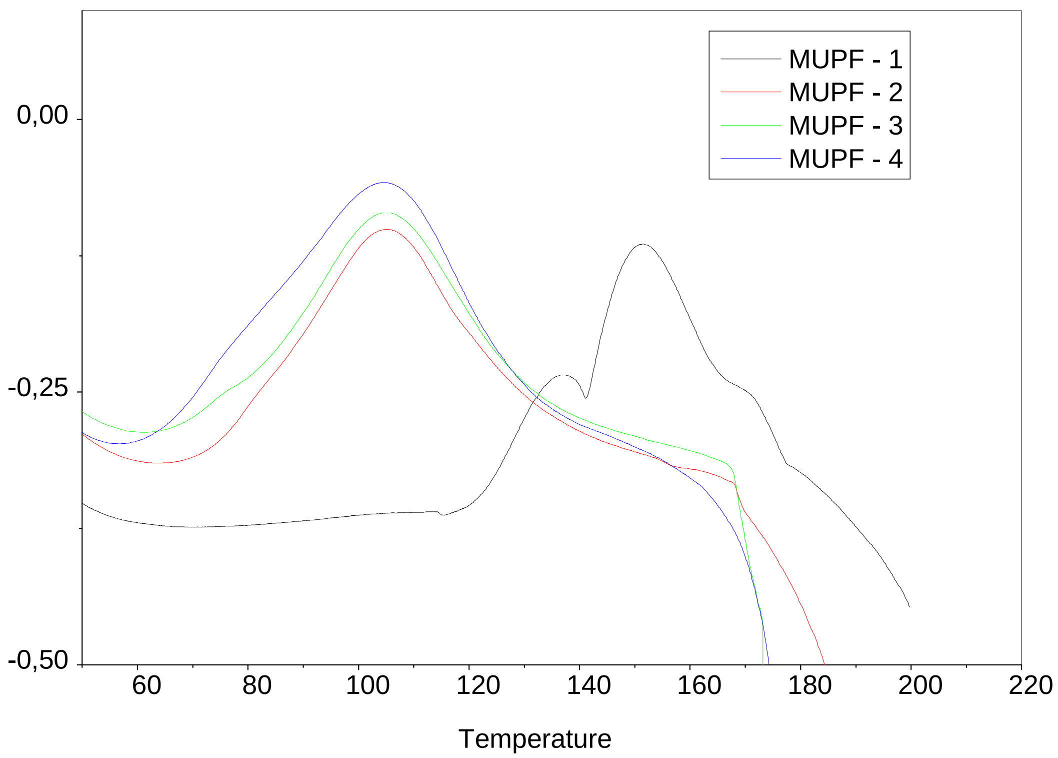


Figure 1: *DSC scans of MUPF resin systems*

The details of the DSC peaks of the studied systems, as summarised in the table below, indicate that the MUPF resin alone has a very slow curing reaction since all peak characteristics (onset, maximum, endset) are shifted to very high temperatures. On the other hand, the additives $(NH_4)_2SO_4$, HR2 and (HR2+CR1) have severely influenced all the peak characteristics, indicating that the resin systems became more reactive. Furthermore, in the reverse order the scans of the systems show a larger positive (exothermic) peak, which is due to the polymerisation reaction of the resin.

Table 12: *DSC results of MUPF resin systems*

System	T_{ONSET} (°C)	T_{MAX} (°C)	T_{ENDSET} (°C)	Specific heat of reaction (J/g)
MUPF - 1	141.9	156.9	196.82	65.89
MUPF - 2	75.19	105.05	131.34	45.94
MUPF - 3	77.92	105.09	131.46	41.96
MUPF - 4	64.5	103.3	130.59	56.87

The specific heat of reaction of the systems varies and there is no clear dependence on the additives used. A higher heat of reaction of a resin suggests that the crosslinking density is higher compared to a resin with a lower heat of reaction. This result is further substantiated by the % conversion versus temperature graph (Figure 2).

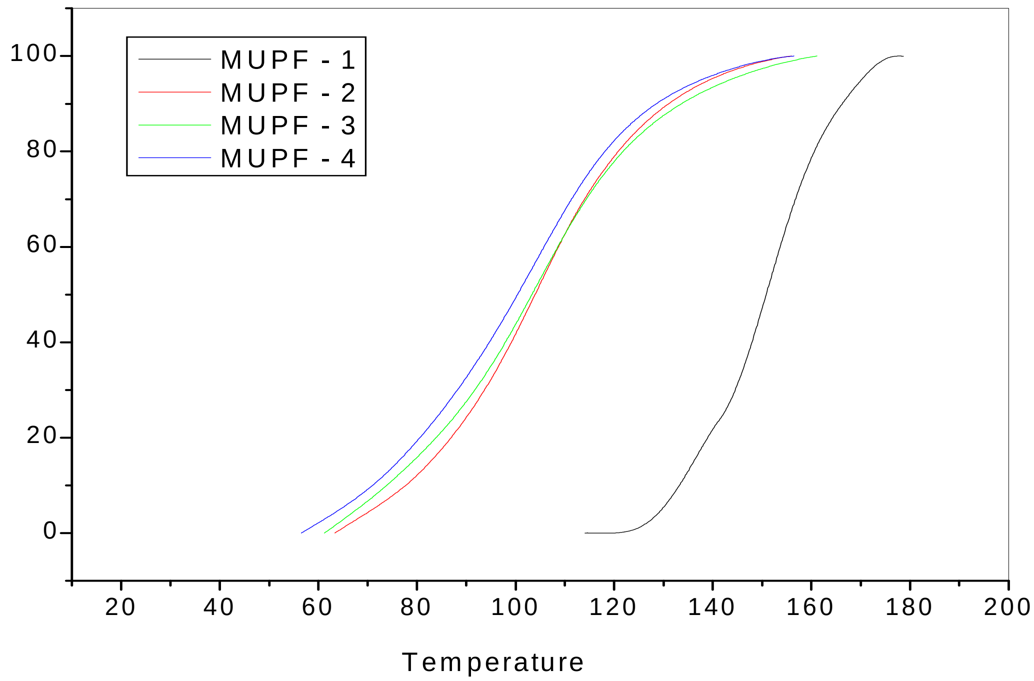


Figure 2: *% conversion as a function of temperature of MUPF resin systems*

The % conversion (C), regarded as a measure of the extent of crosslinking, is defined as:

$C(T) = 100 \times (A_T/A_0)$

where A_T is the peak area up to a temperature T and A_0 is the total peak area (i.e. total heat of reaction).

In Figure 2, it is easy to identify the less reactive system, i.e. MUPF alone, while all the rest three resin systems have a significant % conversion at a temperature of 100-110^{o}C, a temperature which is reached in the core of a panel during the hot pressing stage of the panel production. However, at around 150^{o}C all systems have the same crosslinking density, which before this point differs and at a certain point one or another resin is faster. In any case the system MUPF-4 has higher % conversion. It can then be concluded that the system MUPF–4 will have the best performance keeping the hot pressing settings constant. This is confirmed by the mechanical properties of the panels produced with this system (D3.3 report).

The same study (determination and processing of the thermal analysis data) was performed for the PF resin. The systems examined are as follows:

- PF – 1 plain resin
- PF – 2 resin with 3% K_2CO_3
- PF – 3 resin with 2% H5545
- PF – 4 resin with 4% H5545

The DSC thermograms of the PF resin systems are presented in Figure 3 and the respective DSC peak results are given in Table 13.

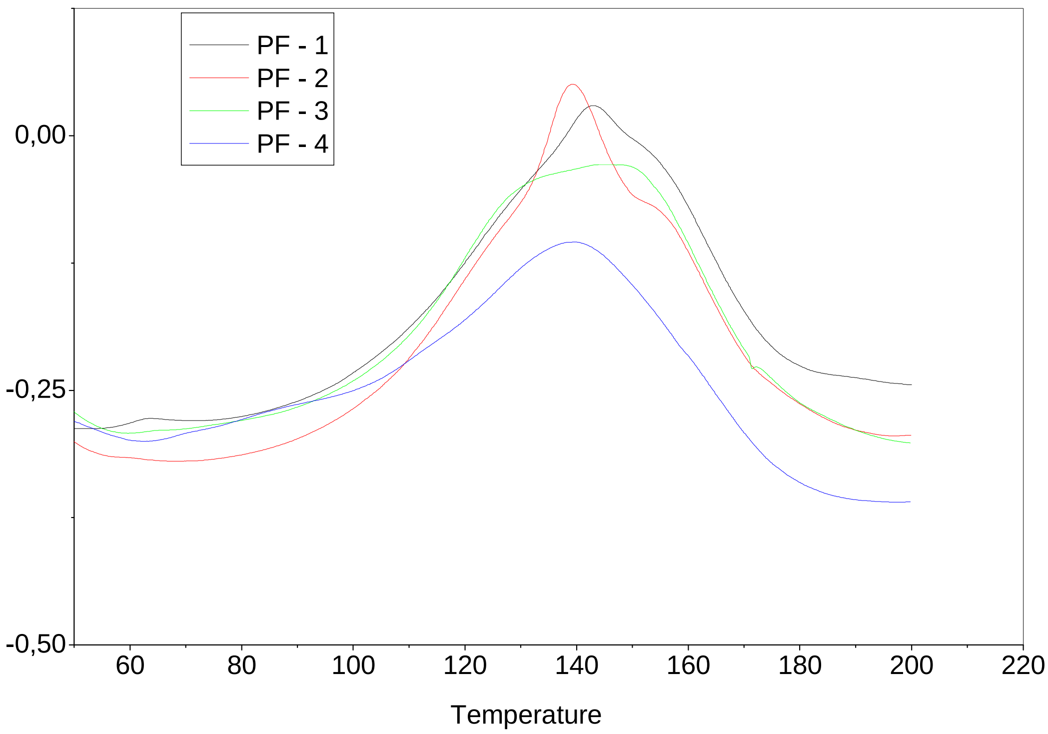


Figure 3: *DSC scans of PF resin systems*

Table 13: *DSC results of PF resin systems*

System	T_{ONSET} (°C)	T_{MAX} (°C)	T_{ENDSET} (°C)	Specific heat of reaction (J/g)
PF - 1	104.82	143.03	175.34	76.81
PF - 2	115.09	139.26	165.82	95.73
PF - 3	100.23	145.18	196.52	96.39
PF - 4	103.60	139.7	176.51	57.22

In this case it is evident that the PF resin alone has similar curing rate with the systems with hardeners, but generally the PF systems are all less reactive than the MUPF ones.

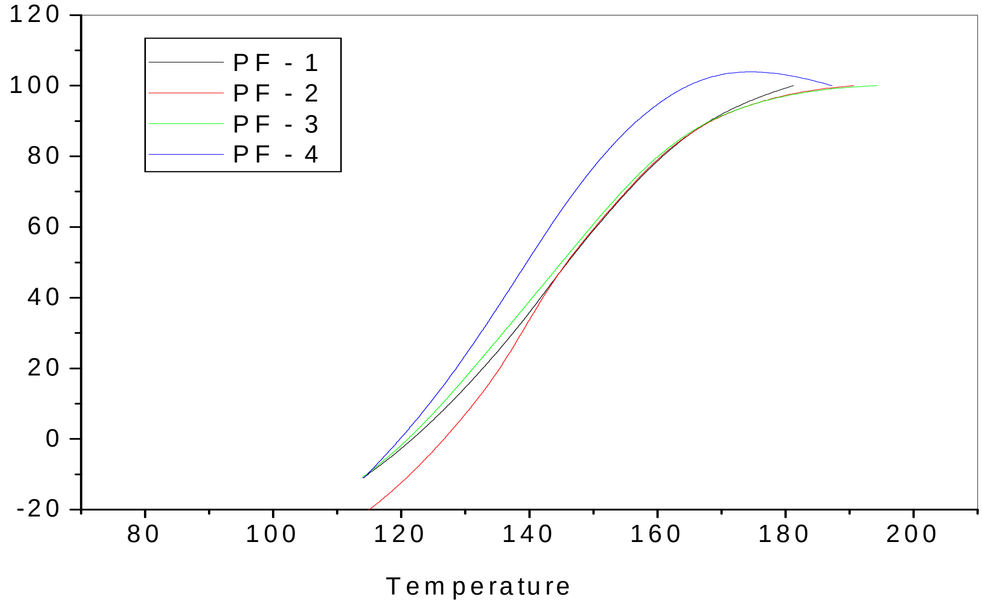


Figure 4: *% conversion as a function of temperature of PF resin systems*

At lower temperatures, both PF systems with H5545 show higher % conversion (Figure 4), which depends on the hardener level. Thus, at higher temperatures the PF-4 system with the highest amount of H5545 is clearly differentiated from the rest three systems, which are similar to each other. The mechanical properties of the PF-bonded LSB panels support these finding as well.

5 CONCLUSIONS/RECOMMENDATIONS

The results of the study performed in the framework of WP3 revealed the following:

- It is feasible to produce at laboratory scale light weight OSB-type boards meeting the standard requirements for OSB/3 grade as well as the E1 formaldehyde class using poplar strands of I-214 clone supplied by the I-PAN partners and a binder system of PF resin with special additives. This result was obtained with the MUPF resin system too (D3.3.). Both the developed PF and MUPF resin systems met the I-PAN objective of poplar LSB panels with formaldehyde emissions satisfying the most stringent European Standards as per EN 13986.
- As in the case of MUPF resin, when producing the PF-bonded LSB the press temperature was reduced by more than 10% without increasing the press cycle, thus meeting the I-PAN objective of curing temperature lower than that of the conventional resin systems by up to 10% and consequently lower energy demand.
- The developed PF and MUPF resin systems met the I-PAN objective of compensation of strand moisture contents ≥ 3.5%.
- The level of the special hardener H5545 used along with the PF resin in the production of laboratory light weight OSB panels was optimized.
- The effect of the reduced panel density of the LSB manufactured with PF resin on the panel properties was investigated. It was found, that the lower the density of the panels, the worse the panel properties are. Increased resin loading can compensate the decrease in panel properties. The optimum resin loading can only be determined during a pilot or an industrial scale trial.
- The curing performance of both MUPF and PF resin alone or with additives was studied via DSC thermal analysis. The DSC data and its processing confirmed that the use of additives improve the curing and the crosslinking density of the resins.