



MECHANICAL PROPERTIES TESTS OF DELIGNIFIED AND DENSIFIED WOOD

Jörg Wehsener¹, Martina Bremer², Peer Haller¹

ABSTRACT: Wood modification has a long tradition. The target is in many cases to improve the mechanical properties, durability or other properties. In this study was investigated a combination of delignification and densification to enhance bending strength and ASE. Both wood treatments change properties in different ways depending on the conditions and the wood species. The primary objective of this treatment combination was the improvement of mechanical properties by partly extraction of lignin components. The treated structure retains cellulose and hemicellulose chains aligned. The densification perpendicular to the grain will reduce the cell lumina and compress the wood structure more than 70% together, afterwards. As a result, the cell walls become entangled and hydrogen bonds between adjacent cellulose nanofibers according [1].

The combined processes were applied to the green and dried poplar (*Populus nigra* L.). Firstly: delignification procedure was done with small samples in NaOH and Na₂SO₃ solution by more than 100°C, 130°C and 150°C for 7h. After washing and analyse wood components second step followed: densification. The wood was compressed perpendicular to the grain in different temperature levels (100°C, 130°C, 160°C) and holding times up to 80% of the original thickness. After the treatment, ASE, MoR and MoE were determined. The bending strength was mainly depending on the solution temperature and compression time and temperature. Bending strength has the optimum by around 90% lignin removal and 130°C pressing time over 24h. The bending strength will increase up to 450MPa compare to the densified reference of 250MPa and the untreated polar app. 65MPa. ASE was reduced by strong delignification and increase temperature.

KEYWORDS: tension, bending strength, recovery set, delignification, densification

1 INTRODUCTION

Investigations on modification processes have been done since the last decades. All types of processes (chemical, physical or biological) improve the material behavior in different ways. Thus, better material properties must be achieved. Samples of wood modification – like thermo hydro mechanical densification - was produced as LIGNOSTONE, STAYPAK or LIGNIFOL [8, 20]. Inoue et al. [6] and other authors tried to solve the recovery problem by superheated steam, [12] and [9] by heated oil, [10] in combination by both. Other treatment with chemical components and different types of resin impregnation (DMDHEU, melamine, phenol, furfuryl) were investigated by e.g. [19, 13, 21, 14, 11, 17] and many more. Beside of the published study from [4] using alkaline, densification and partial extraction of wood polymers will attract attention. The dependency of densification time and temperature to improve the

dimensional stability of are well known: first, decrease in the very hydrophilic hemicelluloses means a decrease in the water affinity. Second, the resulting furfurals bind to the lignin and provide additional crosslinking through this adhesion. In this way, the stresses that arose during compaction can be relieved by re-crosslinking the microfibrils of the cellulose. According to [10], these stresses are the main cause of recovery. Also the moisture influence reduced the glass transition temperature of lignin. It appears less tensions during densification process caused by wood plasticizing. Moisture also contributes to further hydrolysis of the polysaccharides, which supports hydrophobicity, but also leads to a shortening of cellulose chain length and an associated loss of strength. Results of those treatments show significant increase of mechanical properties and dimensional stability. In this point of view, success appear, but modification processes are dealing with small and thin wood samples.

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Poplar, as fast growing wood species has the potential to become high strength properties by chemical and mechanical modification. It is well-known that densification transverse to grain will improve the mechanical properties [18, 22, 15]. Delignification influence wood structure, dimensional stability and bond strength of cellulose [23, 25]. By using both, there is stronger increase of mechanical properties caused by breaking and reformation new hydrogen bonds [1, 16, 26]. A new approach is a partial delignification combined with a compression of the wood was described by [2, 3]. As a structure-forming polymer, lignin plays an important role in plants. It penetrates the cellulose fibers and stick them together whereby it contributes significantly to the strength properties. With partial removal of the lignin, a higher compression of the cellulose fibers is possible [24]. In addition, fewer cracks form in the cell wall during densification and the density and associated strength increase. According to [5] and [4] new intermolecular interactions between the cellulose chains are formed during compression, which further increases the strength. In addition, the dimensional stability is improved due to reduced water accessibility and other solvents. Another positive benefit of this material is the improved surface hardness [7]. These modified properties result in new applications potentials such as parts of music instruments, timber joints like wood nails or connection plats, machine parts or others.

2 MATERIAL AND PROCESS

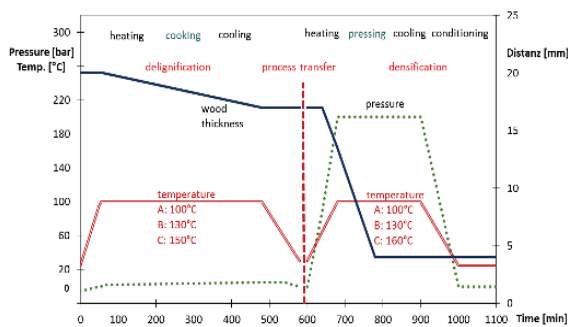


Figure 1: Process parameters during thermal delignification and thermo-mechanical densification (schematically)

2.1 MATERIAL

Green sawn and dried poplar wood (*Populus nigra* L.) was used for the investigations. The wood samples, with the dimensions of 200 mm (longitudinal) x 100 mm (tangential) and 20 mm (radial), were cut from green log with initial oven dry density of 0.44 g/cm³. The same log-material were used for all treatments, respectively, were used as untreated reference specimens. The initial moisture content (mc) of green specimens was 90%, 124% and 162% at the beginning of delignification process (temperature of 100°C, 130 °C and 150°C). The

dried one were treated at 9% moisture. Green and dried specimens were delignified in digester, washed and conditioned (20°C/65%RH) for two weeks. Afterwards, densification was applied in heating press. In each case, twin specimens were produced in order to be able to determine the changes in chemical composition caused by the delignification and to be able to proof hornification effects on delignification. In total, 12 specimens were modified by each process. Figure 1 shows both modification processes schematically:

2.2 DELIGNIFICATION

Delignification takes place in a 20 Litre autoclave with circulation pump (Figure 2). The delignification liquor contained sodium hydroxide (2.5 molar) and sodium bisulfite (0.4 molar). Wood specimens were placed in the digester with the help of a grid to ensure even circulation of the cooking liquor around all specimens. The temperature during delignification was varied whereby the delignification time was counted from reaching 100°C. Digestion time was seven hours for each. For removing the chemicals and dissolved wood components, several washing steps were carried out after the cooking process. At first, the specimens were washed inside the autoclave with hot water (80°C) two times. In the next step, the samples were placed in green water for at least 18h hours. This was followed by two short rinsing cycles with cold water. Finally, the samples were stored again in water for 2 days.

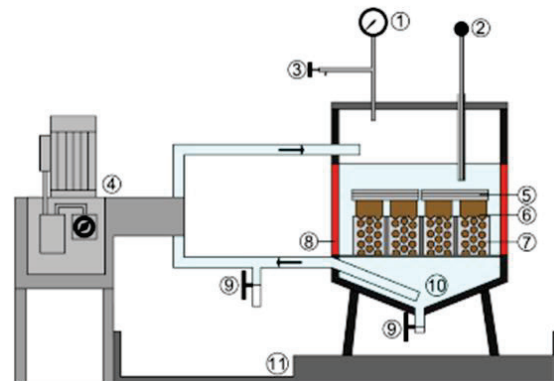


Figure 2: Digester equipment for delignification (1 pressure gauge with pressure relief valve, 2 temperature sensors, 3 valve, 4 circulation pump, 5 stainless steel perforated plates, 6 specimens, 7 stainless steel perforated grilles, 8 wall heating, 9 drain valve, 10 solution, 11 drip tray)

2.3 CHEMICAL CHARACTERIZATION

The delignified as well as the twin specimens were characterized for their chemical composition. For that, the specimens were dried at 103°C and milled by a cross beater mill using a 4 mm sieve. All contents relate to the dry matter of the specimens. Content of extracts were estimated by Soxhlet extraction with a mixture of ethanol and toluene (1:1 v/v) for 6 h. Since there is a mass loss

during delignification, the reference mass for the chemical analysis of the contents of the individual wood components changes. Therefore, the wet-chemically determined contents were corrected on the basis of the mass loss.

2.3.1 Cellulose

For determination of cellulose content about 1 g of the specimens were cooked three times under reflux in 25 % nitric acid in ethanol for a half hour each. After that the cellulose was separated by filtration using glass filter crucible of pore size 40 – 100 µm. The separated cellulose was cooked again in water for 1 h and washed acid free with water. Masses of cellulose were determined after drying at 103°C.

2.3.2 Holocellulose

About 0.3 g of specimens were used for estimation of holocellulose by delignification with sodium chlorite. For this, the specimens were shacked in a solution of 60 µL glacial acetic acid and 0.3 g sodium chlorite in 45 mL deionized water five times for 1 h at 70°C. After each hour glacial acetic acid as well as sodium chlorite was added again in the same amounts. Then the holocellulose was separated by filtration using glass filter crucible of pore size 16 – 40 µm and washed acid free with water. Masses of holocellulose were determined after drying at 103°C.

2.3.3 Hemicelluloses

Content of hemicelluloses results from the difference between holocellulose and cellulose.

2.3.4 Klason lignin

Content of Klason lignin was determined by dissolving of the Polysaccharides. About 0.5 g of specimens were mixed with 15 mL of 72 % sulphuric acid. This mixture was hold at room temperature for 2 h with occasional stirring. After that, the mixture was diluted with 560 mL of deionized water and cooked for 4 h under reflux. The resulting Klason lignin was separated by filtration using glass filter crucible of pore size < 16 µm and washed acid free with hot water. Masses of lignin were determined after drying at 103°C.

2.4 DENSIFICATION

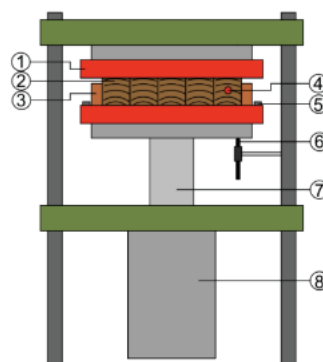
The specimens were conditioned in a climate chamber at 20°C and 90 % RH. At a wood moisture content (MC) of approx. 20 %, 12 boards of each species were thermo-mechanically densified transverse to the grain to a thickness of 4 mm. It was carried out as a thermo-mechanical pressure deformation in an open system (Figure 3). The process was applied in three steps: 1) heating for 60 min by using temperature of 100,130 and 160°C, 2) densifying during next 120 min by maximum pressure of 40 N/mm² followed by a second phase of thermosetting for 4 h, 20 h or 24 h, and 3) cooling to 40°C within 60 min.

During the densification process, MC was reduced. Metal spacers were used to limit the degree of densification but there was no limitation in width. Volumetric density

measurements were performed subsequently on densified specimens. After conditioning, specimens were cut parallel to fibre in original thickness for mechanical tests. Compression set *C* was evaluated as.

$$C = \frac{R_0 - R_c}{R_0} \quad (1)$$

where *R*₀ is the initial thickness and *R*_c is the thickness of



compressed specimens. The compression set was 80 % caused by inhomogeneous cross section of delignified specimens.

Figure 3: Hot press (1 hot plate, 2 specimens, 3 fixer, 4 temperature sensors, 5 metal spacer, 6 transducers, 7 plunger, 8 hydraulic cylinder)

2.4.1 Three-point bending test

The modulus of rupture (*MoR*) was determined in a three-point bending test according to DIN 52186 (1978) with a span of 120 mm parallel to the grain and 3 replicate (*n*) specimens of 140 mm (longitudinal) x 10 mm x 4 mm (radial) for each test modification parameter. The direction of densification was equal to the direction of loading. Test speed was 6 mm per minute.

2.4.2 Determination of dimensional stability (ASE)

Based on the DIN 52184 modified material was stored in normal conditions for 10 days. Specimens of 10 mm (longitudinal) x 20 mm x 4 mm (*n* = 3) were tested with five cycles of oven drying at 103 ± 2°C for 18 h. This was followed by a soaking phase by means of water vacuum-pressure impregnation (50 kPa and 15 min followed by 800 kPa and 20 min) with subsequent water storage for 24 h to determine the change of volume due to the treatment. ASE was calculated as

$$ASE = \frac{a_{untreated} - a_{treated}}{a_{untreated}} \quad (2)$$

where *a*_{untreated} is the maximal swelling before and *a*_{treated} is the maximal swelling after treatment.

Additional were determined compression-set-recovery *C_R* according Equation (3), which

express permanent thickness after swelling. It was calculated as

$$C_R = \frac{R_{C,wet} - R_c}{R_0 - R_c} \quad (3)$$

where $R_{C,wet}$ is the thickness of dried compressed specimens after swelling.

3 RESULTS AND DISCUSSION

3.1 PROCESSES

Washing was done two times 15 min and repeated after 18 h water storage. Depending on treatment temperature, surface colour changed from white to dark brown. This may be explained by contact of black liquid, caused by lignin. Green and dried specimens were soft after processing and distorted after drying. Particularly with regard to the dried poplar specimens showed drying cracks and loose fibers (reduced bonds due to removed lignin). After conditioning, densification, the determination of chemical components took place. Mean density of green and dried specimens after treatment are shown in Table 1. As expected, the poplar density was multiplied by densification and once again more by delignification.

In generally, on dried specimens were measured higher density than on the green one, significantly. The density of green sawn specimens was slightly affected at delignification temperature of 100°C and 130°C, but degreased of 150°C significantly. Higher process temperatures result in more defibration and degradation of the cell composite.

Table 1: Oven-dry density of poplar (reference=0.44g/cm³), only densified poplar (1.22g/cm³) specimens after delignification and densification (treatment temperature/treatment time)

Delignification temperature (°C)	Densified, delignified 100°C/4h (g/cm ³)	Densified, delignified 130°C/4h (g/cm ³)	Densified, delignified 160°C/4h (g/cm ³)
100 green	1.46	1.39	1.41
100 dried	1.50	1.51	1.49
130 green	1.44	1.36	1.44
130 dried	1.55	1.51	1.53
150 green	1.09	0.93	0.98
150 dried	1.38	1.41	1.34

3.2 CHEMICAL COMPOSITION

It can already be seen from the measured loss of mass due to delignification that there is a significant difference between the green and pre-dried wood. While the mass loss of the green specimens is independent of the temperature, in the dry specimens the mass loss increases with rising delignification temperature. A comparison of mass loss with the summarized changes in the contents of the wood components, which were determined by wet-

chemical methods, shows the same trends. In the case of green specimens the absolute value of measured loss is significantly smaller than the summarized changes in contents of wood components. This suggests that depolymerizations occur. The resulting low molecular weight (oligomeric) products cannot be determined with the wet chemical methods. For the technical-dried specimens, the values of the measured mass losses and the sum of changes of wood components are closer together. Beside this, the mass loss at delignification temperature of 150°C is much higher for the dried specimens. If one considers the change in the individual wood components (Table 2), the different behaviour of the green compared to the technical dried specimens can also be seen here. With the exception of cellulose, in the dried specimens the proportion of all components decreases with increasing temperature. The lignin is then completely removed. Extracts and hemicelluloses are only about 80% present after delignification at 150°C. The change in the cellulose content shows a minimum at 130°C, which is caused by the binding of reactive degradation products of lignin or hemicelluloses to cellulose. At 150°C, the degradation of hemicelluloses and lignin leads to less reactive substances with condensed structures and low content of functional groups.

Table 2: Changes in contents of wood components

Sample	T _{Delignification} (°C)	Change in contents (% (w/w))				
		EXTRACTIVES	LIGNIN	CELLULOSE	HEMI-CELLULOSE	ASH
green	100	15	-19	6	-68	169
	130	1	-44	13	-62	87
	150	-14	-91	47	-68	202
dry	100	-21	-5	-15	-42	1265
	130	-42	-54	-7	-52	372
	150	-81	-99	-20	-77	287

For green specimens the change of lignin content correlates also with the temperature. In contrast, the degradation of hemicelluloses seems to be almost independent of temperature. The increase of cellulose content can be explained with the binding of reactive degradation products. Change in extractive content varies with temperature. Indeed, the content of extractives in green specimens is in general less. Digestion chemicals that have not been removed completely cause the sharp increase in the ash content for all specimens. These results show that the accessibility of the digestion chemicals is better in technical dried wood than in green wood.

3.3 DENSIFIED WOOD

Delignified specimens were app. 4 mm thick after densification. The average density after the treatment varied from 0.9 g/cm³ to 1.5 g/cm³, depending of different delignification. Deformation of cross section does not influence compression quality result in very tightly

packed, stiff and hard material. Only highly delignified specimens show loose fibres on the surface.

3.4 BENDING STRENGTH

Bending strength was determined by 3-point bending test. MoR of undensified poplar was according *Wehsener et al. (2018)* approx. 62 MPa by mean density of 0.36 g/cm³ and on 50 % densified specimens MoR of 107 MPa (0.69 g/cm³) was achieved. The MoR of tested only densified reference (approx 75%) increased to 234 MPa by density of 1.219 kg/m³.

Results of MoR from green cut and dried poplar (4 h and 20/24 h densification process) were summarized in Figure 4. Densification of delignified poplar lead to increased MoR depending on treatment temperature and time.

Delignified specimens by 100°C (lignin content 19%) show high MoR (440 MPa) after long densification process (24 h, 130°C), as well as after short process of 4 hours and 160°C (426 MPa). There was not remarkable influence of using green sawn or dried poplar on bending strength after delignification at temperatures of 100°C or 130°C.

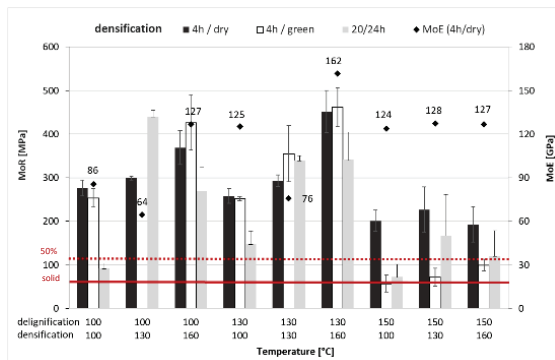


Figure 4: Bending strength of poplar (red), 50% densified (red dotted) and 80% densified and delignified by 100°C, 130°C and 150°C; lignin content by delignification temperature was 19% (100°C), 13% (130°C) and 2% (150°C)

Delignification temperature of 130°C and 4 h on 160°C densified shows also high MoR of 462 MPa (dry) and 452 MPa (green). Specimens treated by 150°C, MoR decrease all about, but twice on green sawn specimens at the same time.

MoE of 150°C delignified specimens sustained at the high level close to 120 GPa, but shows also indifferent behaviour on lower temperature. In general, it can be seen, the MoR values are higher on lower temperature and longer pressing time (130°C, 24h) even on short time and high temperature (160°C, 4h). For obtaining optimum results, the delignification temperature should be on mild temperatures (100°C or 130°C) for partial delignification (reduced lignin between 20 and 60%) and high temperatures for densification (130°C; 160°C). The treatment process achieve maximal bending strength on less than 50% reduced hemicellulose and elevated densification temperature.

The delignification reduces density and hence characteristics like mechanical properties are affected. The measured bending strength are indifferent in regards to the expected results. Figure 5 shows the MoR results of the untreated, densified, and delignified and densified specimens (densification: 4 h) in relationship to the density. High delignified green sawn wood (2% lignin) show similar results close to 100 MPa, but very high density differences (0.44 to 1.4 g/cm³). Dried and high modified wood achieved MoR twice time compared to green sawn specimens. Specimens low delignified (13% and 19% lignin) tend to high density up to 1.5 g/cm³ and MoR (up to 250 MPa). All specimens show maximum MoR independent of the initial moisture content. Only 80% densified specimens double MoR nearly and density compared to 50% densification. Values of around 200 MPa were measured.

Material of 2% lignin remaining are less compact and dense than mild delignified wood. Specimens failed interlaminar and show defibration. High delignification reduced bonding of cellulose fibers, caused by lignin removing. Bending tests show MoE around 120 GPa, but low MoR on 100 MPa by green sawn specimens. In opposite to the high treated material (2% lignin) the mild delignified material (13% and 19% lignin) shows high elasticity and no brittle failure. Material has compact and dense structure across the section. Bending strength increased up to 440MPa and the specimens failed very ductile. Green sawn specimens failed by shear and on several specimens fiber splitting appeared.

Considering the densification time it can be stated that long densification time of 24 hours combined by mild delignification leads to high mechanical properties. In the tests, the MoR showed similar tendency and increased when densification time chosen for 4 hours and densification temperature was high.

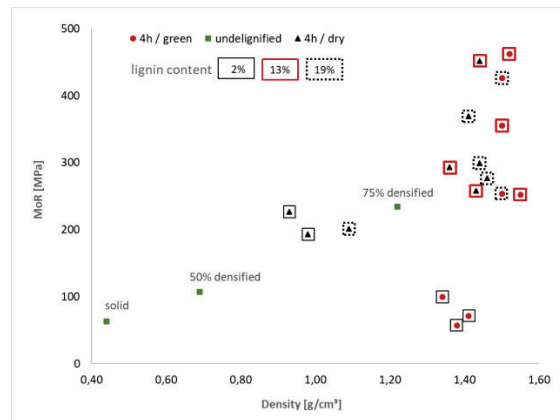


Figure 5: MoR in comparison to density modified on green and dried poplar with reduced lignin content and densification ratio

3.5 ANTI-SWELLING EFFICIENCY (ASE)

During five cycles of swelling and shrinking the recovery-set of the modified material was reduced. As previously reported by [15], recovery-set of densified wood is decreasing with increasing intensity of a thermal

modification. In this study, solid, densified and densified combined with delignified modification processes were tested. Highly delignified specimens showed different layers by loose fibres. In general, swelling is reduced by increased temperature and compression time compared to only densified specimens. Influence of different lignin content was not detectable. Swelling of green sawn specimens was smaller than dried one. The results showed similar behaviour of maximum swelling and recovery, basically. Specimens (lignin 13%) densified by 100°C and 20 hours and also densified by 130°C and 4 hours, recovery decrease to 62.4% and 59.4%. The treatment (19% lignin content) reduces recovery less than 50% (Table 3). The material obtained from the combined process of thermo-mechanical densification and delignification was, thus, more dimensionally stable.

Table 3: ASE and C_R of modified specimens (*-failed)

Lignin (%)	Densification time (h)	Densification temperature (°C)	ASE	C_R	
19	green	24	100	0.49	0.15
	dried	4	100	0.27	0.48
	dried	24	130	0.31	0.49
	dried	4	160	0.32	0.40
13	dried	24	160	0.45	0.24
	green	20	100	0.62	0.9
	dried	4	100	0.38	0.31
	dried	4	130	0.22	0.50
	dried	4	160	0.59	0.23
	dried	20	100	0.29	0.37
2	dried	24	130	0.38	0.41
	dried	24	160	0.47	0.27
	green	4	100	0.23	0.35
	dried	4	130	0.33	0.36
	dried	4	160	0.23	0.31
	dried	20	100	0.26	0.36
2	dried	20	130	0.5	-*
	dried	20	160	0.34	0.24

4 CONCLUSIONS

The results obtained in this study confirmed the potential of the combined treatment to improve material properties e.g. bending strength and ASE. It showed also the absolute requirement to determine the optimum between process temperature and time of delignification and densification. The rate of delignification depends strongly on temperature. At 100°C, lignin degradation only takes place to a small extent. At 150°C, on the other hand, lignin is almost completely degraded. The degradation of the hemicellulose does not show this strong temperature influence. Strong delignified material shows less compact structure than mild delignified. Mechanical properties increase by extended densification and high temperatures. Improved bending strength was gained by partly removed lignin and temperature of 130°C on both processes. The bending strength increase up to 450 MPa compared to the

densified reference of 250 MPa and the untreated poplar of 65 MPa. The densification time had the strongest influence parameter on the dimensional stability. High delignification rate as well as decrease of polar polysaccharides reduces ASE. However, there was no clear correlation to bending strength and the density.

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