Highly-Densified Panels from Hyun Aspen (*Populus alba* × *glandulosa*) Wood with Different Delignification Levels Using Alkaline Hydrogen Peroxide Pretreatment

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Defect-free, highly-densified panels without springback were prepared from Hyun aspen (*Populus alba* × *glandulosa*) wood with different delignification levels by alkaline hydrogen peroxide (AHP) pretreatment. The AHP concentrations were adjusted to 3%, 7%, 10%, while the temperature (100 °C), time (3 h), pH (11), liquor ratio (6.25), and stabilizer (0.5% EDTA) remained constant. The delignification of Hyun aspen wood panels was indirectly quantified by analyzing the UV absorbance of the spent liquor after AHP treatment, wherein delignification ranged from 12% to 58% depending on the AHP concentration. To achieve highly-densified panels, the densification conditions, such as pressure, temperature, and time, were examined. Panels subjected to cold-pressing (25 °C) at 30 MPa for 1 h, followed by hot-pressing (105 °C) at 30 MPa for 6 h, then immediately removed after heating, resulted in durable, highly-densified panels with no springback or cracks.

DOI: 10.15376/biores.20.1.1600-1613

Keywords: Hyun aspen; Alkaline hydrogen peroxide; Delignification; Densification; Panels

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INTRODUCTION

Hyun aspen (*Populus alba* \times *glandulosa*), commonly known as hybrid poplar, is an interspecies cross between white poplar (P. alba) and Korean poplar (P. glandulosa). Developed in 1965 at the Institute of Forest Genetics in Suwon, Korea, the hybrid was intended to rapidly reforest barren hills and denuded mountains (Yun et al. 2008). Out of 330 crosses of various *Populus* species, the hybrid was found to exhibit exceptional growth on poor, acidic slopes (Hyun and Cho 1963; Hyun et al. 1967). The hybrid is also recognized for its adaptability to diverse soil conditions (Son et al. 1981), resilience to salt and drought stresses (Yoon et al. 2014), and superior growth rate. Moreover, its tendency to form clonal colonies through an interconnected root system enables the hybrid to regenerate and persist over prolonged periods. However, its economic value for timber has been limited due to inferior wood characteristics, such as low dry matter yield and low specific gravity (Noh et al. 1981), weak tensile strength, and susceptibility to twisting. Despite being one of Korea's major non-native plantation species, Hyun aspen has mainly been used for lower-value applications, such as matchsticks, fuelwood (Noh 1982), and roughage feeds for livestock (Kang et al. 1990). Although economic utilization remains challenging, there is a growing need to explore more valuable uses for the hybrid. A potential utilization for low-quality wood, such as Hyun aspen, is in the production of wood

panels through bulk densification. Introducing a delignification step prior to densification has been proposed as a means for improving fiber bonding and enhancing the mechanical properties of the panels (Sánchez et al. 2018). By partially reducing the lignin content, delignification allows the remaining cellulose fibers to bond more effectively, facilitating easier densification during compression. Several delignification methods utilize sodium hydroxide, sodium sulfite, sodium chlorite (Li et al. 2016; Yang et al. 2019), hydrogen peroxide (Frey et al. 2018; Li et al. 2019; He et al. 2020; Liang et al. 2020), or mixtures of these reagents (Song et al. 2017, 2018; Han et al. 2019; Li et al. 2020). Notably, partial delignification of wood via treatment of sodium sulfite and sodium hydroxide, followed by hot-pressing, resulted in high-performance panels with excellent strength, toughness, and ballistic resistance (Song et al. 2018). Their study showed that a delignification level of 45% yielded the highest strength and density. In comparison, alkaline hydrogen peroxide (AHP) delignification is effective at reducing lignin while preserving the fiber integrity of wood. This makes it a promising method for producing stronger, denser, and more durable panels through the combined process of delignification and densification. Moreover, AHP offers practical advantages, as it is simpler to work with, can be performed under atmospheric conditions, easier to manage in terms of effluent treatment, and more environmentally friendly, leaving no toxic by-products.

Natural wood is a renewable resource with high strength-to-weight ratio and exceptional versatility, making it a preferred material in construction and furniture. However, natural wood has inherent limitations such as water absorption, susceptibility to deformation, and vulnerability to environmental factors. The densification of wood addresses these drawbacks by improving its strength, hardness, surface abrasion resistance, and dimensional stability (Cabral *et al.* 2022). Wood can be densified such that it achieves a density of up to 1.5 g/cm³ (Rautkari *et al.* 2011). In recent years, research has focused on optimizing densification techniques and exploring environmentally friendly pretreatment methods to further enhance wood's performance without compromising sustainability.

The purpose of this study was to prepare highly-densified panels from Hyun aspen wood through AHP delignification followed by densification. Various delignification levels and their corresponding densification conditions were examined to determine their effects on the characteristics and density of the prepared panels. This study is a continuation of the work on AHP delignification of three low-value lignocellulosic biomass under atmospheric conditions for high-performance wood materials (Mun and Mun 2024).

EXPERIMENTAL

Materials

Hyun aspen (*Populus alba* × *glandulosa*) wood was sourced from the foothills of Yangyari in Wanju, Korea. The wood was processed and cut into flitches, from which panels with dimensions of 100 mm (longitudinal, *L*, parallel to the grain orientation) ×100 mm (radial, R) × 10 mm (tangential, T) were prepared. Part of the wood panels was ground and then sieved to prepare the woodmeal. A 40 to 80 mesh was used in this experiment.

Sodium hydroxide (93%), disodium ethylenediaminetetraacetic acid (99.5%, Na₂EDTA), and sodium chlorite (78%) were purchased from Duksan Pure Chemicals (Ansan, Korea). Hydrogen peroxide (34.5% by titration) was purchased from Samchun Chemicals (Pyeongtaek, Korea).

Methods

Delignification of Hyun aspen woodmeal

A 2.0 g of oven-dried (o.d.) woodmeal was placed in a 500-mL glass bottle. Extractives were not removed from the woodmeal before AHP delignification. A specified volume of 10% AHP solution, based on the liquor ratio, was added to the woodmeal. EDTA, 0.5% based on the weight of woodmeal, was also added to stabilize hydrogen peroxide. The mixture was subjected to delignification under the conditions outlined in Table 1, using a shaking water bath. After treatment, the residue was filtered through a 1G4 glass filter and washed with 150 mL hot distilled deionized water. The residue was then dried in a convection oven at 105 °C for 24 h, and the residual yield was determined accordingly.

Table 1. Delignificatior	n Conditions	for	Woodmea	
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Parameter	Values	Parameter	Values		
Temperature (°C)	27, 70, 100	AHP concentration (%)	10		
Time (h)	1-27	Liquor ratio	6.25, 12.5		
pH	11.0±0.1	Stabilizer, EDTA (g)	0.01		
Shaking (rpm)	140				

Determination of lignin from the residue and filtrate

The residue was allowed to reach equilibrium moisture content and was then used for the determination of acid-insoluble lignin (TAPPI 222-om11 2011) and acid-soluble lignin (TAPPI UM 250 1991). The acid-soluble lignin content was measured at a wavelength of 210 nm, using an absorption coefficient of 105 L/g·cm. The total lignin (on residue) and total lignin (on wood) were obtained using Eqs. 1 and 2, respectively.

 $Total \ lignin \ (on \ residue, \ \%) = Acid-insoluble \ lignin + Acid-soluble \ lignin \ (1)$

 $Total \ lignin \ (on \ wood, \ \%) = Total \ lignin \ (on \ residue) \times Residual \ yield$ (2)

To estimate the dissolved lignin in the filtrate, the spent liquor and washings were combined. The total UV absorbance of the combined liquor was measured at 280 nm with appropriate dilution. The lignin removal or delignification (*DL*) was obtained using Eq. 3,

$$DL(\%) = (L_o - L_t) \times 100 / L_o$$
(3)

where L_o is the original lignin content (on wood, %) and L_t is the total lignin content (on wood, %). The L_o was for wood panels was determined as 24.10% (Mun and Mun 2024).

Determination of carbohydrates from the residue

The carbohydrate content of residues from selected delignification conditions was determined. Holocellulose and α -cellulose contents were measured according to the method described by Wise *et al.* (1946). Hemicellulose content was calculated as the difference between the holocellulose and α -cellulose contents.

Delignification of Hyun aspen wood panels

Ten wood panels (415 g, o.d.) were placed in a 10-L plastic container, and approximately 2.6 L AHP solution with 2.1 g EDTA was added. The panels were soaked in the solution at 25 °C for 24 h. The panels were transferred to a 10-L round-bottom reactor, as well as the used AHP liquor. Delignification of the panels was carried out under

the conditions shown in Table 2. Following treatment, the AHP-treated panels were removed from the liquor and soaked into 5 L water in a 10-L plastic container for 24 h. The soaked panels were transferred to the reactor with the washings, and boiled for 30 min. The boiling and soaking were repeated four more times. The wood panels were stored in a zipper bag and stored at 4 $^{\circ}$ C.

Parameter	Values	Parameter	Values
Temperature (°C)	100	AHP concentration (%)	3, 7, 10
Time (h)	3	Liquor ratio	6.25
рН	11.0±0.1	Stabilizer, EDTA (g)	2.1

Table 2. Delignification Conditions for Wood Panels

Estimation of delignification from the wood panels

The spent liquor and all washings were collected in a 50-L container. An aliquot was diluted to fifty-fold and the total UV absorbance was measured at 280 nm to estimate the dissolved lignin content.

Densification of the wood panels

The wood panels were subjected to cold-pressing followed by hot-pressing using a hot plate press (5T, Ilshin Autoclave, Daejeon, Korea). The panels were pressed into a stainless steel mold with perforations, perpendicular to the radial-longitudinal (*RL*) plane under varying conditions of temperature (25 to 140 °C), pressure (5 to 30 MPa), and time (0.5 to 24 h). After hot-pressing, the panels were immediately removed from the press. The degree of compression (*DoC*) was measured with respect to the changes in thickness at the longitudinal-tangential (*LT*) plane following Eq. 4,

$$DoC(\%) = (T_i - T_f) \times 100 / T_i$$
 (4)

where T_i is the initial thickness (mm) and T_f is the final thickness of the panel (mm). The density of the representative compressed wood panels was obtained using water displacement method at 25 °C.

Morphology and FT-IR spectra of the panels

Scanning electron microscopy (SEM) images of wood and densified panels were obtained using a scanning electron microscope (Supra 40VP, Zeiss, Germany) operating at 2 kV. Thin slices of wood panel and densified panel, cut along *RT* and *LT* planes, were sputter-coated with platinum. The FT-IR spectra of the original and densified wood panels were obtained using a FT-IR spectrophotometer with an ATR accessory, equipped with a lithium tantalate detector (Spectrum Two, Perkin Elmer, USA).

RESULTS AND DISCUSSION

Delignification of Panels

In a preliminary experiment to measure the delignification of panels, the woodmeal underwent AHP pretreatment, and the UV absorbance of the resulting filtrate was

compared with the actual measured lignin content to establish a correlation. Based on this correlation, the panels were delignified under the same conditions, and the UV absorbance of the filtrate was indirectly measured to determine the delignification level of the panels.

Various delignification conditions using a fixed concentration of 10% AHP were applied to the woodmeal, and the resulting residual yields, total lignin contents, and total UV absorbance were evaluated (Table 3). The residual yields ranged from 70.2 to 90.6%, while delignification varied between 13.8 to 44.1%. An inverse relationship was apparent between delignification and residual yield, which indicated that greater lignin removal resulted to higher material loss. The most effective delignification conditions occurred under conditions of 10% AHP, liquor ratio (LR) 12.5, 100 °C, and 3 h at pH 11, which achieved 44.1% delignification. These results indicated that AHP treatment led to high residual yields with moderate delignification, which suggests its suitability for applications requiring the retention of the wood's structural integrity, such as in the production of durable panel materials.

Delię	gnificatio	on Condi	tions	Residual		Lignii	n (%)		Deligni	fication
AHP (%)	LR	Temp (°C)	Time (h)	Yield (%)	Acid- insoluble	Acid- soluble	Total (residue)	Total (wood)	UV abs	DL (%)
		25	3	90.52	18.19	4.77	22.96	20.78	7.5	13.8
	12.5	70	3	76.24	13.89	4.98	18.87	14.39	14.7	40.3
		100	3	70.17	14.48	4.71	19.19	13.47	19.1	44.1
10		25	1	90.62	17.23	4.57	21.80	19.76	6.1	18.0
10		25	15	84.83	15.96	4.70	20.66	17.53	8.7	27.3
	6.25	25	27	81.14	15.60	4.28	19.88	16.13	11.5	33.1
		70	7	77.85	15.38	4.37	19.75	15.38	13.6	36.2
		100	7	72.94	15.23	4.06	19.29	14.07	18.9	41.6

Table 3. AHP Delignification Conditions, Yield, Lignin Content, and

 Delignification of Woodmeal

A delignification curve was prepared based on the AHP treatment of woodmeal. Several AHP conditions, designated as low, medium, and high delignification levels, are pointed out in Fig. 1a. The corresponding chemical composition at these delignification levels is shown in Fig. 1b. As delignification levels increased, the contents of cellulose, hemicellulose, and lignin decreased. Notably, when the untreated wood (no delignification) was subjected to high levels of delignification, the cellulose content decreased from 49.4% to 42.6%, which showed a reduction of only 6.7%. However, the hemicellulose and lignin contents were reduced by 24.0% and 10.6%, respectively. These findings indicated that AHP treatment selectively degrades hemicellulose and lignin (Ho *et al.* 2019).

The delignification of wood panels was indirectly quantified by extrapolating the total UV absorbance of the combined liquor after AHP treatment. Table 4 describes the AHP delignification conditions employed with wood panels. Delignification was carried out under the most effective delignification conditions (10% AHP, 100 °C, 3 h at pH 11), with the liquor ratio reduced from 12.5 to 6.25 for economic reasons. Based on the curve prepared from woodmeal (Fig. 1a), a delignification of 58.0% was achieved, exceeding the anticipated delignification threshold for Hyun aspen.



Fig. 1. Relationship between delignification and UV absorbance of woodmeal spent liquor (a), and changes in chemical composition of woodmeal at various delignification levels (b)

The effect of varying AHP concentrations under the same liquor ratio, temperature, and time was also examined. Delignification of 12 %, 42%, and 58% were obtained for 3%, 7%, and 10% AHP concentrations, respectively, showing that lower AHP concentrations resulted in less lignin removal, as expected. In a similar study, AHP treatment of quaking aspen (*Populus tremuloides*) using 5% AHP, LR 10, 22 °C, 72 h at pH 11 resulted in 32% delignification (Springer 1990). These results suggested that delignification was within a comparable range, taking into account the differences in treatment conditions and species.

	Delignificatio	Delignification			
AHP (%)	LR	Temp (°C)	Time (h)	UV abs	DL (%)
3				4.8	12.0
7	6.25	100	3	17.3	42.2
10	0.20			24.0	58.0

 Table 4. AHP Delignification Conditions on Wood Panels

Densification of Wood Panels

Densification conditions for wood panels with 12% delignification

A panel with 12% delignification was achieved using 3% AHP treatment. After cold-pressing for 1 h and hot-pressing at 105 °C for 6 h under a constant maximum pressure of 30 MPa, defect-free panels with an average degree of compression (*DoC*) of $58\pm3\%$ were obtained (Appendix Table A1, Samples 4-1 to 4-5). With an original thickness of 10 mm, more than half of the thickness was reduced and a density of 1.2 g/cm^3 was obtained. In addition, the low degree of delignification caused high resistance during densification. While several densification conditions were carried out, no panel defects (*e.g.* checks, shakes, split, or twist) were observed.

Densification conditions for wood panels with 42% delignification

For panels with 42% delignification, densification was easier compared to those panels with 12% delignification. Consequently, lower pressure conditions of 20 MPa and 10 MPa were also examined (Appendix Table A2, Samples 3-5 and 3-6, respectively).

However, panels subjected to these lower pressure conditions developed checks, split, and showed discoloration, likely due to incomplete removal of liquor. The most favorable conditions remained the same as previously described for the 12% delignified panels (Sample 3-3). Under these optimal conditions, a DoC of more than 70% was achieved, allowing for the manufacture of high-density, highly-densified panels with a density of 1.2 g/cm^3 . In addition, when the drying time was reduced from 6 to 5 h, the panel cupped, as shown in Sample 3-4. Thus, a minimum of 6 h of drying at 105 °C was determined to be necessary under this densification condition. For faster drying, if springback is minimal after compression, securing the panel to prevent warping (*i.e.* twisting, bowing, cupping) and performing air-drying at about 60 °C, similar to standard wood drying processes, could facilitate the mass production of panels. When a continuous process is implemented, it is important to ensure that steam can escape efficiently from inside the panel during compression. Panels produced under this delignification condition retained their original shape well, even under various densification conditions. Consequently, mass production of panels will be carried out under these conditions to optimize the production of highlydensified panels.

Densification conditions for wood panels with 58% delignification

For panels with 58% delignification, densification was easier to achieve compared to those with 12% and 42% delignified panels. The removal of over 50% of the lignin embedded in the wood cell walls and middle lamella contributed to this ease in densification. Lignin functions in wood similar to that of concrete, which provides stability and mechanical support to the wood cell wall; when lignin is partially or entirely removed, the wood cell wall becomes increasingly flexible (Miao et al. 2017). With this level of delignification, manufacturing highly-densified panels without applying high pressure are possible. Accordingly, a panel was assessed under a minimum pressure of 5 MPa at 105 °C (Appendix Table A3, Sample 2-2). However, the panel split in the middle, making it difficult to produce highly-densified panels. This splitting likely resulted from the steam generated inside the wood during compression, which was not adequately vented and caused internal rupture. As a result of manufacturing high-compression panels with 58% delignification under various conditions, Sample 2-4 yielded the best outcome. Under this condition, the panel thickness was reduced by 74%, achieving the highest DoC among all pressed panels. While these densification conditions lack commercial viability due to the high pressures and long drying times required, it has been confirmed that high-performance compression panels can be successfully manufactured when delignification reaches 40 to 50%. In addition, the density of panels at this delignification were 1.2 g/cm^3 . This result suggested that while the AHP pretreatment affected the chemical composition of wood panels, it had minimal impact on the overall density of the panels. In previous trials, the panel needed to remain under pressure for 24 h at 105 °C, but under these conditions, drying was completed after 20 h. Despite this slight improvement, the pressing time still remained lengthy and must be further reduced for efficient mass production. Therefore, additional research into faster drying methods is needed. To address this, the panels were pre-dried in an oven at 60 °C for 3 h prior to hot-pressing to quickly remove the liquor and reduce the drying time to 6 h (Sample 2-7 and 2-9). Nonetheless, the panels showed checks and discoloration despite these adjustments.

Fiber bonding mechanism

The primary role of AHP pretreatment was to partially remove lignin from the wood panel, enhancing bonding between delignified fibers during densification under heat and pressure without the need for adhesives. AHP pretreatment had a minimal effect on cellulose, preserving the integrity of the original wood structure. In principles, the AHP pretreatment can partially oxidize cellulose, particularly converting reducing end groups into carboxylic acids, and hot pressing may lead to dehydration forming esters. However, the IR spectra of the compressed wood panels detected no carbonyl peaks at 1735 cm⁻¹ region, which suggested that the amount of oxidized cellulose end groups was negligible. Conversely, carbonyl groups were detected only in the original wood panel, which originated from lignin moieties (*i.e.* α -carbonyl, esters). Characteristic C–O bond peaks at 1240 and 1035 cm⁻¹ were assigned to lignin and cellulose/hemicellulose, respectively.



Fig. 2. FT-IR spectra of the original and densified (58% delignified) wood panels

On the other hand, the Masonite process utilizes steam explosion to break down wood chips into fibers, which are then pressed into panels. This process retains lignin, utilizing it as a natural bonding agent. While the AHP pretreatment focuses on partially removing lignin to enhance fiber bonding, the Masonite process depends on lignin to facilitate fiber bonding.

Morphology of densified panels

The SEM image of wood along the *RT* plane showed typical hardwood cell structures, primarily consisting of vessels and fibers (Fig. 3a). Following densification, most fiber lumina had collapsed, while the lumina of several vessels maintained their shape (Fig. 3b). As shown in Fig. 3c, the collapsed fiber lumina developed wrinkles due to compression. In general, vessels have higher lignin content than fibers and are enriched with guaiacyl units (Saka and Goring 1985; Boerjan *et al.* 2003). This high lignin content contributed to the structural integrity of the vessel walls, allowing them to retain their luminal structure despite the compressive forces applied during densification.

On the LT plane of wood (Fig. 3d), intercellular spaces between vessels and fibers were observed; however, these gaps were nearly not present in the densified panels (Fig. 3e). Despite the consolidation of fibers, there were some fractures (perpendicular to the LT plane) observed, which were likely caused by the release of trapped gas and moisture during the hot-pressing process. The strength and hardness of the densified panels can be attributed to the enhanced bonding between fibers, as shown in Fig. 3f. Future work will involve the examination of the mechanical properties of the wood panels, including flexural strength, tensile strength, impact resistance, and surface abrasion resistance.



Fig. 3. SEM images of wood in RT (a) and LT planes (d), and densified wood in RT (b,c) and LT planes (e,f)

CONCLUSIONS

- 1. Partial delignification (40 to 50%) of Hyun aspen wood panels by alkaline hydrogen peroxide (AHP) treatment followed by compression showed a potential in the production of highly-densified panels.
- 2. The delignification of wood panels was indirectly quantified by analyzing the UV absorbance of the spent liquor after AHP treatment, wherein delignification ranged from 12 to 58%.
- 3. Panels subjected to cold-pressing (25 °C) at 30 MPa for 1 h, followed by hot-pressing (105 °C) at 30 MPa for 6 h, then immediately removed after heating, resulted in durable, highly-densified panels with no defects.

4. The role of AHP pretreatment was to partially remove lignin from the wood panel, enhancing bonding between delignified fibers during subsequent densification under heat and pressure without the need for adhesives.

ACKNOWLEDGMENTS

This research was supported by the Technology Innovation Program through the Korea Evaluation Institute of Industrial Technology (KEIT) funded by the Ministry of Trade, Industry, and Energy (MOTIE, Korea) (Carbon Innovation Stars Program, Grant No. 20018304).

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Article submitted: November 6, 2024; Peer review completed: November 23, 2024; Revised version received: December 8, 2024; Accepted: December 10, 2024; Published: December 19, 2024.

DOI: 10.15376/biores.20.1.1600-1613

APPENDIX

Table AL. Densincation Conditions for wood Parters with 12% Dendrincation	Table	A1. Densification	Conditions for	Wood Panels with	12% Delignification
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							0	
Sample 4-1 (<i>DoC</i> = 52%)			Sample 4-2 (58%)			Sample 4-3 (60%)		
P (MPa)	Time (h)	T (°C)	P (MPa)	Time (h)	T (°C)	P (MPa)	Time (h)	T (°C)
30	1	25	30	1	25	30	1	25
30	6	105	30	6	105	30	6	105
0	-	-	0	-	-	0	-	-







Sample 4-4 (58%)			Sample 4-5 (60%)			Sample 4-6 (45%)		
P (MPa)	Time (h)	T (°C)	P (MPa)	Time (h)	T (°C)	P (MPa)	Time (h)	T (°C)
30	1	25	30	1	25	30	24	25
30	6	105	30	6	105	30	24	60
0	-	-	0	-	-	0	-	-







Table A2. Densification Conditions for Wood Panels with 42% Delignification

0.		20()	0-	0 = 1 = 0, 0, (700)					
Sar	mple 3-2 (6	9%)	Sar	Sample 3-3 (70%)			npie 3-4 (68	iple 3-4 (68%)	
P (MPa)	Time (h)	T (°C)	P (MPa)	Time (h)	T (°C)	P (MPa)	Time (h)	T (°C)	
30	1	25	30	1	25	30	1	25	
30	6	105	30	6	105	30	5	105	
30	-	-	0	-	-	0	-	-	
0	-	50							







Sample 3-5 (67%)			Sample 3-6 (63%)			Sample 3-9 (66%)		
P (MPa)	Time (h)	T (°C)	P (MPa)	Time (h)	T (°C)	P (MPa)	Time (h)	T (°C)
30	1	25	30	1	25	30	1	25
20	6	105	10	6	105	30	3	120
0	-	-	0	-	-	0	-	-







Table A3. Densification Conditions for Wood Panels with 58% Delignification

Sar	mple 2-1 (7	0%)	Sar	mple 2-2 (59	9%)	Sar	0%)	
P (MPa)	Time (h)	T (°C)	P (MPa)	Time (h)	T (°C)	P (MPa)	Time (h)	T (°C)
20	3	25	5	3	25	10	3	25
20	15	105	5	15	105	10	15	105
20	-	-	5	-	-	10	-	-
0	-	50	0		50	0		50







Sar	mple 2-4 (74	4%)	Sample 2-7 (71%) Sample 2-9			nple 2-9 (67	67%)	
P (MPa)	Time (h)	T (°C)	P (MPa)	Time (h)	T (°C)	P (MPa)	Time (h)	T (°C)
30	3	25	0	3	60	0	3	60
30	20	105	20	6	105	30	6	105
30	-	-	20	-	-	30	-	-
0		50	0		50	0		50





