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**CHEMISTRY OF STEAM STABILIZATION: PRELIMINARY  
ANALYSIS OF STEAM PRESSED WAFERBOARD**

by

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#### SUMMARY

Dimensionally stable steam-pressed waferboard, prepared by direct steam injection during pressing, under conditions more drastic than those to be expected in practice, has been fractionated by solvent extraction. The fractions have been analyzed for lignin, xylan, and cellulose or other glucan, and the results have been compared with those from the original aspenwood wafers. The results show a small increase in the ethanol-soluble lignin and a very small increase in the water-soluble xylan - much less than that found in aspenwood wafers, steam-pretreated to the degree necessary to obtain comparable dimensional stability after conventional pressing.

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## 1.0 OBJECTIVE

To determine the chemical changes occurring in wood as a result of steam pressing under conditions which produce dimensional stabilization.

## 2.0 INTRODUCTION

Dimensional instability has hindered the growth of highly densified wood-based composites in many areas where high humidity or water exposure is expected to be encountered. Fortunately, it has been proven that dimensionally stable wood composites can be produced from steam treated furnish or by steam pressing (i.e., injecting steam into the mat or board during hot pressing). It was found that steam pretreatment resulted in some chemical changes in wood, especially in the hemicelluloses. The cellulose and lignin fractions were not changed significantly. However, the full extent of the chemical changes are not known. If this technology is to be implemented, a better understanding of the chemical changes occurring is necessary.

In comparison with steam pretreatment, shorter steaming times are required for the production of dimensionally stable products by steam pressing.

## 3.0 BACKGROUND

Steam pretreatment of aspenwood, under more drastic conditions than those proposed for steam pressing, has been shown to result in hydrolysis of both xylan and lignin, to the extent that they become largely soluble in water and organic solvents, respectively. Even the cellulose, which is more resistant, undergoes some hydrolysis which reduces its degree of polymerization. These same changes undoubtedly occur to at least a slight extent during steam pressing. Whether or not these small chemical changes, which accompany dimensional stabilization, are necessary for the development of stabilization is not known.

#### 4.0 STAFF

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#### 5.0 MATERIALS AND METHODS

##### 5.1 STEAM-PRESSED WAFERBOARD

The MOR specimens, remaining after the mechanical testing of steam-pressed waferboard No. 19, were selected on Oct. 13, 1988 for chemical analysis. This board, which had been prepared as described in Table 1 as part of an unpublished Forintek study, "Steam Pressing Technology", had given good thickness swelling of only 3.5 and 7.2% after 24 and 72 h water soaking respectively. The pressing conditions were relatively drastic and should, therefore, have resulted in chemical changes at least as great as any to be expected in practice.

##### 5.2 SAMPLE PREPARATION

The MOR specimens of steam-pressed waferboard were split into smaller pieces with a hammer and chisel and were ground in a Wiley mill to pass a 1 mm screen.

A control sample of the original wafers was similarly ground to pass the 1 mm screen.

##### 5.3 SOLVENT EXTRACTION

An accurately weighed sample of the ground waferboard of known moisture content, with an oven-dry equivalent weight of 250 g was extracted in a large soxhlet with benzene. Benzene was found in separate experiments to be a good solvent for slack wax, in contrast to the conventional wood-chemistry solvent ethanol-benzene (1:2) which is too polar for slack wax. After removal of the benzene-solubles, extraction was repeated with ethanol-benzene and, after evaporation of the ethanol-benzene, the residual ethanol-benzene extract was

Table 1

Preparation of Steam-Pressed Waferboard

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wafers	1.5 x 0.028 inches
slack wax	1.5%
resin	2%, BD003 Reichhold
moisture content of wafers	5%
press temperature	210°C
steam injection from both top and bottom	
steam temperature	198°C (200 psi)
press closing time	30 s
steam injection from top	2 min
steam injection from bottom	2 min
board density	approx. 38.5 pcf

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divided into two fractions: a small additional benzene-soluble fraction, and a larger benzene-insoluble fraction which was completely soluble in ethanol.

The ground waferboard fraction, remaining undissolved after the ethanol-benzene extraction, was air-dried and was then extracted three times with water at room temperature.

A similar series of extractions was conducted with the ground original wafers, with the exception that no initial benzene extraction was performed.

#### 5.4 CHEMICAL ANALYSIS

The water-insoluble material (WI) remaining after the water extractions, was analyzed for acid-insoluble lignin (Klason lignin) by TAPPI Standard Method T222 os 74. The resulting acidic filtrate was injected directly into an HPLC fitted with a Bio-Rad HPX-87H column which was kept at a temperature of 70°C. The eluant was 0.01N sulfuric acid at a flow rate of 0.6 mL/min. A Varian LC system (Model 5000) interfaced with a Varian CDS 407 data system was used. A refractive index detector (Varian RI-3) was used for monitoring the separated substances. The samples were injected with a Rheodyne HPLC-injection valve (Model 7146) which was fitted with a 20  $\mu$ L sample loop. Similar analyses were performed on the water-soluble fraction (WS) and on the ethanol-soluble fraction (ES) of the ethanol-benzene-soluble fraction of both the ground waferboard and the ground original wafers.

### 6.0 EXPERIMENTAL PLAN

It is by no means assured that conventional wood-chemistry analysis of steam-pressed boards can reveal the reason for the observed stabilization. It may be that changes in the largely unknown wood ultrastructure are involved—changes which could occur and yet not be reflected in gross measurable chemical changes in the wood components. The plan, therefore, is to look, first, for evidence of the most likely chemical change which can be measured, i.e. solubilization of part of the easily hydrolyzed hemicellulose and of the lignin. For this reason, therefore, solubilities in water and in organic solvents were measured.

### 7.0 RESULTS AND DISCUSSION

The weights of the vacuum-dried extracts, and of the oven-dry insoluble material remaining after the water extractions, are listed in Table 2. The weights listed are normalized to 100 g of original oven-dry wood in the case of the control wafers, and to 103.5 g of oven-dry weight in the case of the



Table 2

Comparison of Amounts of Extractives in Original Wafers  
and in Steam-Pressed Waferboard

Fraction	Original Wafers (g/100 g wood)	Waferboard (g/103.5 g board) <sup>1</sup>
Benzene solubles	not done <sup>2</sup>	2.73
Ethanol-benzene solubles		3.30 <sup>3</sup>
Benzene-soluble fraction	1.51	0.57
Ethanol-soluble fraction	1.00	2.45
Cold-water solubles	0.96 <sup>4</sup>	1.83 <sup>5</sup>
Cold-water insolubles	96.53	94.95
	100.00	102.52

- 1) 103.5 g of board prepared from 100 g wafers + 1.5 g wax + 2.0 g resin
- 2) Benzene solubles in wood should all be removed by ethanol-benzene
- 3) Total weight of 3.30 g of benzene solubles should contain the 1.51 g benzene solubles present in original wafers, plus 1.5 g slack wax
- 4) 1st 0.748 g, + 2nd 0.125 g, + 3rd 0.036 g = total 0.96 g
- 5) 1st + 2nd 1.720 g + 3rd 0.109 g = total 1.83 g

waferboard. If no non-volatiles had been lost on pressing, this weight of 103.5 g of board would, therefore, have originated from 100 g of wood plus 1.5 g of wax and 2.0 g of resin.

Table 2 shows that the total benzene solubles from the waferboard (3.30% of wood weight) exceeded that of the original wafers (1.51%) by only 1.79%, of which 1.5% should have been the added slack wax. There was, therefore only a very slight increase in benzene solubles (approximately 0.3%) as a result of the steam pressing. Table 2 also indicates somewhat greater increases of 1.45% in ethanol solubles, and of 0.87% in cold-water solubles.

The compositions of the ethanol-soluble, the water-soluble, and the water-insoluble fractions, of both the original wafers and of the waferboard, are listed in Table 3. The corresponding distributions of the three components (glucan, xylan, and apparent lignin), among the three fractions (WI, WS and ES), are shown in Table 4. No attempt was made to compare the compositions of the benzene solubles, both because of the difficulty of selectively removing the slack wax from other benzene-soluble material, and because the steam pressing had apparently increased the amount of benzene solubles only slightly, as discussed in the preceding paragraph.

Table 4 shows that, of the original 19.77% of xylan in the original wafers, only 0.4% was lost in the steam pressing (e.g. by conversion to furfural or other decomposition products which could not be converted to xylose on subsequent acid hydrolysis). An additional 0.56% became water-soluble as xylose or xylose oligomers. These data indicate a very little change in the easily hydrolyzed xylan component. Table 3 shows that the xylan content of the water-insoluble fraction of the original wafers decreased by only 0.69% (original wood basis), from 20.06% to 19.37%, as a result of steam pressing. This decrease is much less than that reported necessary by Hsu *et al*<sup>1</sup> for obtaining a comparable reduction in thickness swell, when the wafers were pretreated by steam before being pressed conventionally. Hsu *et al* reported that, to achieve dimensional stabilization, the xylan content of the water-insoluble fraction of aspenwood should be reduced from 18.5% to 16.5% or lower (i.e. by 2% or more) in the corresponding water-insoluble fraction of the steam-treated wood.

The results shown in Table 4 suggest that the weight of ethanol-soluble lignin (in the ethanol-benzene extract from 100 g of original wafers) increased from 0.77 g to 1.74 g, as a result of steam pressing. At the same time, the lignin in the water-insoluble fraction appears to have increased from 17.7 to 19.7, giving a theoretically impossible total increase in the weight of lignin of approximately 3.1 g. Even assuming that all of the resin (2.0 g) was acid insoluble and was weighed as lignin, this still leaves the total lignin high by 1.1 g.

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<sup>1</sup>Hsu, W.E., W. Schwald, J. Schwald and J.A. Shields. 1988. Chemical and physical changes required for producing dimensionally stable wood-based composites. *Wood Sci. Technol.* 22:281-289

Table 3

Comparison of Composition of Extractives and of Extracted Original Wafers  
and Extracted Steam-Pressed Waferboard

		<u>Analysis<sup>1</sup> (% of fraction)</u>	
		<u>Original Wafers</u>	<u>Waferboard</u>
Ethanol solubles (ES)	glucan	1.67	1.01
	xylan	0.00	0.31
	lignin	77.01	71.02
Water solubles (WS)	glucan	29.13	20.86
	xylan	42.95	53.09
	lignin	8.05	3.65
Water insolubles (WI)	glucan	47.53	45.35
	xylan	20.06	19.37
	lignin	18.32	20.75

1) lignin: apparent Klason lignin

glucan: glucose in Klason lignin filtrate expressed as glucan

xylan: xylose in Klason lignin filtrate expressed as xylan

Table 4

Comparison of Weights of Glucan, Xylan, and Apparent Klason Lignin, in the Ethanol-Soluble (ES), Water-Soluble (WS), and Water-Insoluble (WI) Fractions from 100 g of Original Wafers, with Corresponding Weights in Fractions from 103.5 g<sup>1</sup> of Steam-Pressed Waferboard

		<u>Weight of Component<sup>2</sup>(g)</u>			<u>Total (g)</u>
		<u>WI</u>	<u>WS</u>	<u>ES</u>	
Glucan	wafers	45.88	0.28	0.02	46.18
	board	43.06	0.38	0.02	43.46
Xylan	wafers	19.36	0.41	0.00	19.77
	board	18.39	0.97	0.01	19.37
Lignin	wafers	17.69	0.08	0.77	18.37
	board	19.70	0.07	1.74	21.51

1) Board prepared from 100 g wafers + 1.5 g wax + 2.0 g resin = 103.5 g total, therefore all data as % of original wood in wafers or board

2) Values listed, obtained by multiplying analytical values of Table 3 by recovery yields of Table 2, to put on basis of 100 g of original wood in wafers or in board.

It is quite possible that all of this remaining 1.1 g, of excess apparent lignin, was actually incompletely hydrolyzed cellulose which was weighed as lignin the Klason lignin analysis. The fact that the total glucan (determined in the Klason lignin filtrates) appears to have decreased by an even greater 2.7 g, also suggests incomplete hydrolysis of the cellulose. [Incomplete hydrolysis has been found to be more difficult to avoid with steam-treated wood than with untreated wood]. Such a large decrease in the true glucan is very unlikely, because the total xylan (which is much more easily hydrolyzed and destroyed) decreased by only 0.4 g.

This small loss of total xylan also indicates that very little (if any) acid-insoluble xylan decomposition products could have been formed. (Such xylan decomposition products have been found to lead to erroneously high Klason lignin analyses after more drastic steam treatments). Therefore most of the 1.0 g increase in the apparent lignin in the ethanol solubles (from 0.77 to 1.74 g), as a result of steam pressing, probably was solubilized lignin.

## 8.0 CONCLUSIONS AND RECOMMENDATIONS

Steam pressing of aspen wafers, under conditions more drastic than those currently believed most likely to be used to achieve dimensional stabilization, resulted in the solubilization (in water) of only 0.56 of the original 19.36% xylan, and the destruction (to material not hydrolyzable to xylose) of an additional 0.40%. This reduction in xylan content is much less than that previously reported to be necessary in the alternative process in which the wafers were first steam pretreated and then pressed conventionally without steam injection.

The amount of ethanol-soluble lignin, in the ethanol-benzene extract of aspenwood, also increased, by 1% (original-wood basis), as a result of the steam pressing. It is recommended that additional aspenwood wafers be steam-pressed, under the milder conditions expected to be used in practice, but without any added resin or wax, to simplify the interpretation of the extraction results, and that a similar fractionation and analysis be performed.

The degree of polymerization of the cellulose of the steam-pressed wafers should be determined and compared with that of conventionally pressed wafers (i.e. without steam injection). Attempts should be made to detect additional changes in the lignin by extracting with better lignin solvents.