

## DECAY RESISTANCE AND DIMENSIONAL STABILITY OF

# Five Modified Woods<sup>1</sup>



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**A**LL PRESENT COMMERCIAL methods for increasing the decay resistance of wood involve impregnating it with toxic chemicals. Some available data, however, show the possibility of imparting decay resistance to wood by other means.

Baechler (3)<sup>2</sup> described experiments in which resistance to decay was produced by several types of treatments that did not result in the deposition of toxic chemicals in the wood. One type of treatment, cyanoethylation, consisted of a chemical modification of cellulose. The decay resistance of the resulting product was explained on the basis of the inability of the cellulase enzyme to decompose the cellulose derivative.

In this paper it is shown that the decay resistance of chemically modified wood may also be explained on the basis of the inability of the cell walls to absorb moisture needed by decay fungi. This presupposes that, even though water be present in the lumen, enzymatic degradation does not take place unless the cell walls themselves contain adequate moisture. While this conception is not in harmony with modern theories of the mechanism of the decay of wood, the consistent relation between decay resistance of modified woods and their reduced hygroscopicity is presented as a matter of interest.

Other methods that modify the chemical nature of wood in such a way as to reduce the water vapor absorption and swelling significantly have been shown to impart good decay resistance to the wood (9, 11-17).

Five types of modified wood—(1) wood impregnated with a fiber-penetrating phenolic resin, (2) wood with hydroxyl groups replaced with acetyl groups, (3) thermally modified wood, (4) wood cross-linked with formaldehyde, and (5) wood bulked with polyethylene glycol—showed good dimensional stability and decay resistance. Two causes for this resistance are (1) insufficient water can be taken up to support decay, and (2) susceptible hydroxyl groups are removed or blocked so that decay enzymes cannot dissolve wood components.

For example, when wood is impregnated with a water-soluble phenol-formaldehyde resin that penetrates the fibers and is then dried and heat-cured, the hydroxyl groups are chemically blocked. As a result, the hygroscopicity, swelling, and susceptibility of the wood to decay are all reduced (15). Likewise, when the hydroxyl groups of wood are replaced by acetyl groups during the acetylation of wood, its hygroscopicity, swelling, and susceptibility to decay are all reduced (16). When wood is heated under conditions that cause the loss of some water of constitution together with other chemical changes, the same thing occurs (11, 12, 13).

When wood is treated with formaldehyde vapor in the presence of an acid catalyst, the formaldehyde is believed to form cross-links between two hydroxyl groups on adjacent cellulose chains by eliminating one molecule of water, thus forming an oxygen-carbon-oxygen bridge (17). Formation of such cross links is believed to be the only way in which the large reduction in swelling can result from the addition of the small amounts of chemical needed. Further evidence for cross-linking is that the reaction reduces the dimension changes by reducing the swollen dimensions rather than by increasing the dry dimensions as with resin impregnation (15), acetylation (16), and other bulking agents. The swelling of wood reacted with formaldehyde is reduced not only in water but also in dilute alkaline solutions and in pyridine. This fact, which is not true for heat-stabilized wood, makes it appear that formaldehyde reaction involves cross-linking (12, 17) and heat stabilization does not (9).

Decay-resistance tests of various forms of modified wood have included different species of wood in different sizes and shapes. There was no uniformity in the way the decay tests were made. In some instances, the decay resistance could conceivably be due to residual toxic chemicals, such as phenol, formaldehyde, or such thermal degradation products of wood as furfural. With existing data it is hence impossible to compare the decay resistance imparted to wood by different wood-modifying treatments and to conclude beyond doubt that they impart decay resistance to wood in other than the normal way of depositing toxic chemicals within the structure. The research here reported was therefore undertaken to investigate other treatments by which decay resistance can be imparted to wood other than by the presence of toxic chemicals, and how various wood-modifying processes compare in imparting this decay resistance when the tests are made on matched material.

### Materials and Methods

A flat-sawn, straight-grained board of Sitka spruce 5 inches wide by 0.5 inch thick was cut into 0.5-inch lengths in the fiber direction. These stick-shaped specimens were randomized and used for most of the investigation. A few tests were made on a series of Sitka spruce cross sections 0.1 inch in the fiber direction cut from a straight-grained, 8- by 2-inch specimen, the annual rings of which had a minimum curvature and were parallel to two opposite faces.

Specimens of both types were impregnated with aqueous solutions of a water-soluble phenol-formaldehyde resin of different concentrations. The vacuum desiccator technique was used to attain a take-up of solution about

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<sup>2</sup>Maintained at Madison, Wis., in cooperation with the University of Wisconsin.

<sup>3</sup>Numbers in parentheses refer to the Literature Cited at the end of this report.

equal to the dry weight of the wood. The specimens were then air dried, and the resin was cured by heating the specimens in an oven at 105° C. for 24 hours. Resin contents ranging from 5.7 to 42 percent were obtained.

Three sets of sticks were impregnated with a 0.5 percent aqueous solution of zinc chloride and one set with the same concentration of sodium chloride by the vacuum desiccator technique to attain a take-up of salt of about 1 percent after oven-drying.

One set of the specimens that contained zinc chloride was acetylated by exposing them to the vapors of acetic anhydride within a desiccator placed inside an oven heated at 120° C., for times ranging from 10 minutes to 4 hours. Acetyl content calculated on the basis of the increase in the oven-dry weight ranged from 0.5 to 37 percent.

A second set of dry specimens impregnated with zinc chloride was cross-linked with formaldehyde vapor by exposing them for times ranging from 15 minutes to 21 hours over paraformaldehyde within a desiccator placed inside an oven at 201° C. Bound formaldehyde content, determined colorimetrically by the chromatropic acid method (4), ranged from 0.1 to 6.5 percent.

A third set of specimens impregnated with dry zinc chloride, the set impregnated with sodium chloride, and an unimpregnated set were heated in an oven between brass plates at 180° C. for 0.5 to 46 hours. Losses in weight due to thermal degradation varied from 0.5 to 25 percent.

A final set of stick-shaped specimens was impregnated by the vacuum desiccator technique with 8.7 to 63.7 percent of polyethylene glycol-1000, and was then conditioned to equilibrium with 80 percent relative humidity, weighed, and measured in the tangential dimension with a dial gage to the nearest 0.001 inch. The specimens were then conditioned to 30 percent relative humidity and again weighed and measured.

All of the specimens except those impregnated with polyethylene glycol were oven-dried, and the tangential dimensions of the sticks and the tangential and radial dimensions of the cross sections were determined. They were then immersed in distilled water, the air in them was removed by evacuation, and the soaking was continued until they came to swelling equilibrium. The dimensions were measured, and the swelling was calculated from the oven-dry to the water-swollen condition. Swelling was expressed in terms of the reduction in swelling relative to that of the untreated controls; that is, the average swelling of the controls, minus the swelling of the treated spe-

cimen, divided by the swelling of the controls.

Half of the water-logged specimens cross-linked with formaldehyde were soaked in a 1 percent solution of sodium hydroxide for a day. All of the water-logged specimens were then leached with running tap water for 6 weeks, conditioned to 30 percent relative humidity, and weighed.

All of the specimens, including those impregnated with polyethylene glycol, were subjected to the standard soil-block culture test (1) for 3 months after they were inoculated with *Lenzites trabea* 517. The fungus mycelium then was brushed off, and the specimens were again conditioned to 30 percent relative humidity and weighed to determine the loss in weight due to decay. In the case of the specimens impregnated with polyethylene glycol-1000, the chemical is water soluble and some leaches from the block during the test, so that the loss in weight was not an index of the extent of decay. In this case, the extent of decay was qualitatively judged by determining the ease with which a knife could penetrate the structure.

Agar cultures (2) were made with varying amounts of polyethylene glycol-1000 incorporated in the agar to determine if there is a polyethylene glycol content above which fungus will not grow. A gel was obtained only in concentrations below 75 percent by weight.

### Experimental Results

Fig. 1 gives the relationship between the square of the fractional reduction in dimension changes and the introduced chemical content for the Sitka spruce sticks. The square of the reduction in swelling and shrinking is plotted in preference to the reduction in swelling and shrinking, because it gives an initially linear rather than parabolic relationship. The slope of the plot for cross-linking with formaldehyde is about seven times as steep as those for the other three treatments, which reduce dimension changes by bulking the fibers (17).

Fig. 2 gives the relationship between the square of the fractional reduction in swelling and the weight loss of the sticks resulting from thermal degradation. Initially, the relationship is linear and the same for specimens containing zinc chloride, sodium chloride, and no catalyst. Weight loss is attained more rapidly, however, in the presence of zinc chloride catalyst than of sodium chloride, and more rapidly in the presence of sodium chloride than when no catalyst is used.

At a weight loss of about 10 percent and a reduction in swelling of 39

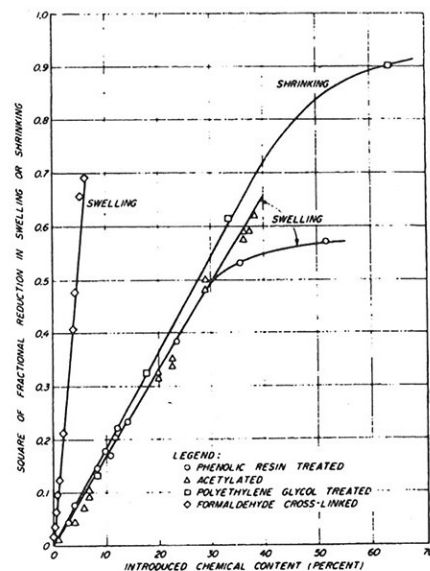


Fig. 1.—Relationship between the square of the fractional reduction in swelling or shrinking and the chemical content of Sitka spruce when acetylated, treated with phenolic resin or polyethylene glycol, and cross-linked with formaldehyde.

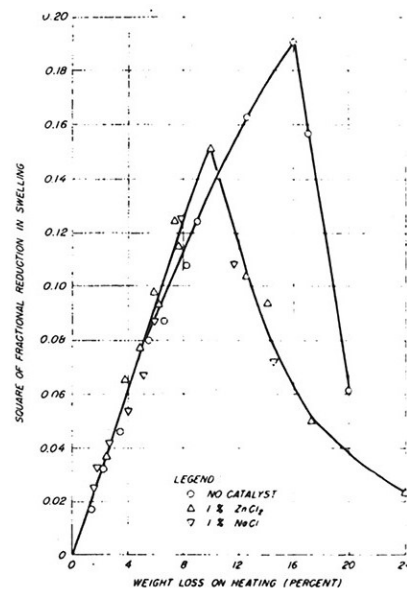


Fig. 2.—Relationship between the square of the fractional reduction in swelling and the weight loss for heat-stabilized Sitka spruce with and without a catalyst.

percent for the catalyzed thermal reaction, a sharp break occurs in the initial relationship. For the uncatalyzed thermal reaction, the break occurs at 16 percent loss in weight and 44 percent reduction in swelling. Seborg, Tarkow, and Stamm (9) had found a similar break in the plot to occur at 46 percent reduction in swelling for catalyst-free wood heated in air. They showed that heating in a closed system in which volatile products could not escape gave a continuously increasing reduction in swelling. Evidently, the nature of the thermal reaction changes above the break in the curve as a result of loss of volatile products.

Fig. 3 gives the relationship between the loss in weight of the speci-

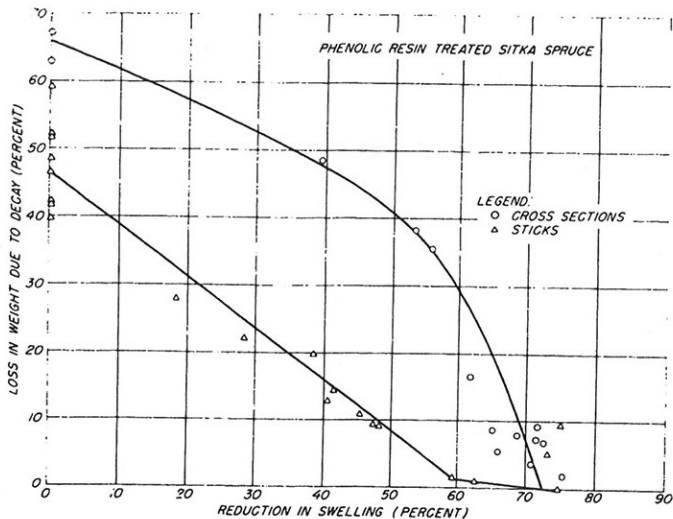


Fig. 3.—Relationship between loss in weight due to decay and reduction in swelling of Sitka spruce treated with phenol-formaldehyde resin.

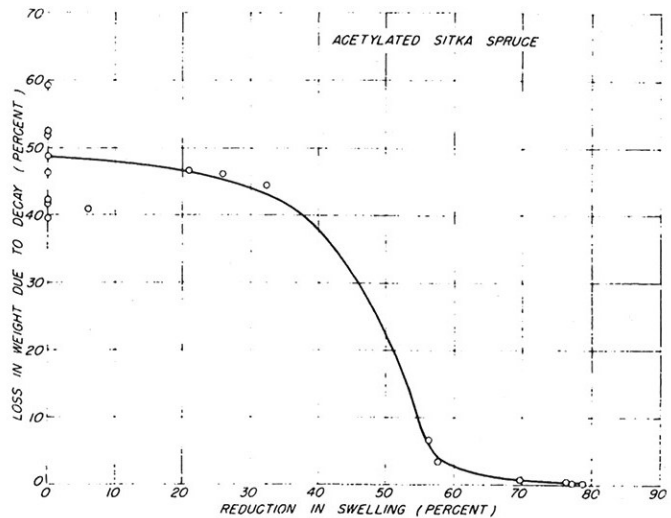


Fig. 4.—Relationship between loss in weight due to decay and reduction in swelling of acetylated Sitka spruce with 1 percent of zinc chloride at catalyst.

mens impregnated with phenol formaldehyde resin after being subjected to decay for three months and the reduction in swelling that resulted from the original treatment. The percentage weight loss for the cross sections was greater than that for the sticks, presumably because the greater surface-to-weight ratio of the cross sections accelerated the rate of decay. Under conditions where little or no decay took place, the size and shape of the specimens loses importance. A dimensional stabilization of 70 percent or more corresponds to practically complete elimination of decay for specimens of both shapes.

Fig. 4 gives a similar relationship for the acetylated sticks. Here, too, decay is practically eliminated when the reduction in swelling exceeds 70 percent.

Decay was practically eliminated in the case of the heat-stabilized wood at reductions in swelling of about 40 percent, as is shown in Fig. 5. The thermal reaction with zinc chloride catalyst present seemed to be slightly more effective in reducing decay than when no catalyst was present under the more drastic heating conditions. Data for the thermal reaction beyond the break in the plots of Fig. 2 are omitted from Fig. 5, to avoid confusion. The loss in weight due to decay was small, even though there was a reversal in the reduction in swelling. These specimens were so fragile that they could hardly be handled.

Fig. 6 shows that no loss in weight due to decay occurs for all specimens in which swelling is reduced 50 percent or more by formaldehyde cross-linking. This level of dimensional stability is attained with as little as 2.5 percent of combined formaldehyde.

The formaldehyde cross-linked specimens leached with a 1 percent sodium hydroxide solution before being

leached with water gave results comparable to those obtained with specimens that were leached only with water. This is a good indication that any condensed paraformaldehyde, which is soluble in sodium hydroxide, was effectively removed by the water extraction alone. It is thus highly improbable that any of the decay resistance shown in Fig. 6 is due to residual formaldehyde polymer.

The decay resistance of the specimens treated with polyethylene glycol-1000 could not be determined by the weight loss, as was the case in the other treatments, because of the water solubility of the chemical. It was hence judged qualitatively by the ease with which a knife would penetrate the structure. Specimens containing at least 18 percent of polyethylene glycol-1000 showed no sign of decay. Specimens containing only 8.6 percent showed slight decay, whereas the controls were badly decayed.

Tests were made of the toxicity of polyethylene glycol-1000 by the agar-flask method (2). The chemical showed no toxicity in low concentrations. At higher concentrations, up to 25 percent, a feeble growth was obtained. At concentrations approaching 75 percent, the limiting concentration at which a gel was formed, growth was entirely inhibited. Inoculum squares from the high concentrations failed to grow when transplanted to plain nutrient agar slants. This apparent toxicity in high concentrations is probably due to an osmotic effect in which the tendency of the polyethylene glycol to dissolve in water actually dehydrates the fungus to the point that it has lost its activity even after transfer to growth conditions.

#### Discussion

All five types of wood-modifying treatments investigated impart dimen-

sional stability and decay resistance to wood. Because of the severe leaching to which the specimens were subjected before the decay test, it is highly improbable that the decay resistance resulted from residual toxic agents in the wood. It is thus of interest to try to explain the reduction in susceptibility to decay in some other way.

The simplest case, that of acetylation, is considered first. It can be seen from Figs. 1 and 4 that decay is practically eliminated when the acetyl content is above 30 percent. The optimum weight increase of cellulose due to the replacement of the three hydroxyl groups per anhydroglucose unit with acetyl groups is 78 percent

$$\left( \frac{3 \times (59-17)}{162} \times 100 \right).$$

The optimum weight increase of lignin due to the replacement of its three hydroxyl groups per repeating unit of 840 is 16 percent

$$\left( \frac{3 \times (59-17)}{840} \times 100 \right).$$

Spruce wood, with a combined cellulose and hemicellulose content of 72 percent and a lignin content of 28 percent, should give an optimum weight increase due to acetylation of 60.5 percent. Decay is thus practically eliminated when 50 percent of the optimum acetylation occurs, or 50 percent of the hydroxyl groups have been replaced.

In a solid-phase heterogeneous reaction, such as the one used, acetylation will first occur in the amorphous regions of cellulose and on the surfaces of the crystallites. It has been estimated that 50 percent of the hydroxyl groups of wood occur in these accessible regions (10). It is thus highly improbable that any readily available hydroxyl groups still remain in the wood to be attacked by decay organisms. Since the susceptibility of wood to decay is dependent on the availabil-



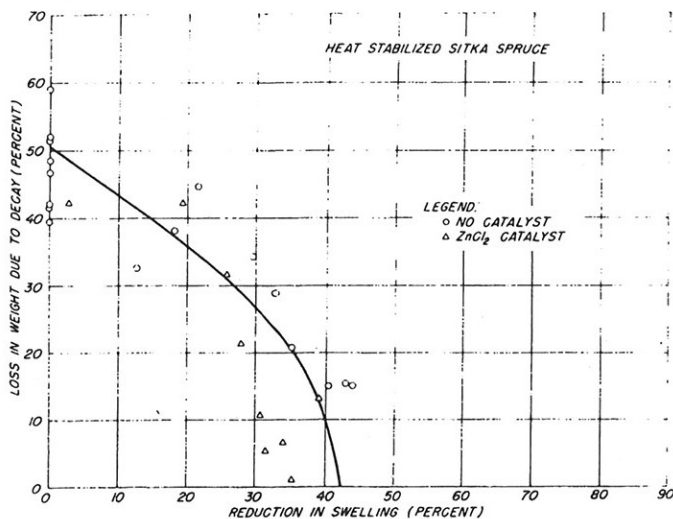


Fig. 5.—Relationship between loss in weight due to decay and reduction in swelling of heat-stabilized Sitka spruce.

ity of hydroxyl groups, it is not surprising that, at the 30 percent weight-increase level of acetylation, wood does not tend to decay even in the presence of adequate water.

The immunity of wood to decay at the 30 percent level of acetylation can also be explained without taking into account the chemical change caused by the acetyl groups. It has been previously shown that acetylation reduces swelling of wood as a result of bulking of the fiber (16). The swelling is reduced by 70 percent when the weight increase due to acetylation is 30 percent (Fig. 2). This amount of swelling corresponds to a reduction in hygroscopicity of about 60 percent (16). The cell walls of wood thus cannot take up more than 12 percent of moisture, if it is assumed that the wood has a fiber saturation point of 30 percent. Various tests have shown that a moisture content of about 20 percent is necessary to support decay (6). Thus insufficient moisture is available within the cell walls, even in the presence of free water, to support decay.

The alkali-catalyzed, water-soluble phenol-formaldehyde resins used for wood impregnation are believed to consist originally of a mixture of phenol alcohols with one to three reactive alcohol groups (5). When the resin is cured by heating, the alcohol groups react with each other, eliminating water, to form three-dimensional oxygen-bridged polymers. It is improbable that this very specific reaction will take place between the hydroxyl groups of cellulose and phenol alcohols. It is possible, however, for hydrogen bonds to form between the hydroxyl groups of cellulose and the phenol alcohols.

The theoretical maximum weight increase that could occur when three phenol dialcohols are hydrogen-bonded through the phenol hydroxyl group of each of the three hydroxyl groups of

cellulose per anhydroglucose unit, with loss of water on curing, is 247 percent

$$\left( \frac{3 \times (151-18)}{162} \times 100 \right).$$

For lignin, the increase would be 46.5 percent  $\frac{3 \times (151-18)}{840} \times 100$ ,

and for spruce wood, 191 percent.

It was shown during the discussion of acetylation that only half of the hydroxyl groups of wood exist in the amorphous regions and on the surface of the crystallites. Up to half of these may mutually satisfy each other through hydrogen bonding. Only one-quarter of the hydroxyl groups may thus be available for hydrogen bonding of resin, for a weight increase of 48 percent. This is still considerably more than the 30 percent of resin that is taken up by wood to saturate the fibers. It thus appears that a phenol alcohol group cannot be taken up by each available hydroxyl group, presumably because of steric hindrance. Because of this discrepancy, a physical rather than a chemical explanation for the decay resistance should be sought.

If phenolic resin with a specific gravity of 1.27 (14) could completely replace water within the cell walls of swollen wood, the take-up of resin should be 38 percent and there should be no subsequent swelling of the resin-treated wood. The best reduction in swelling attained is 76 percent, and that occurring at the inflection point of Fig. 1 is 70 percent. This indicates that the resin within the fiber contracts 24 to 30 percent on curing, and hence this amount of subsequent swelling is possible. These values are close to the amount by which the theoretical resin content exceeds the actual resin content on a percentage basis, namely, 21 percent  $\left( \frac{38-30}{38} \right)$ . It thus appears that

decay is practically eliminated when the resin-forming components com-

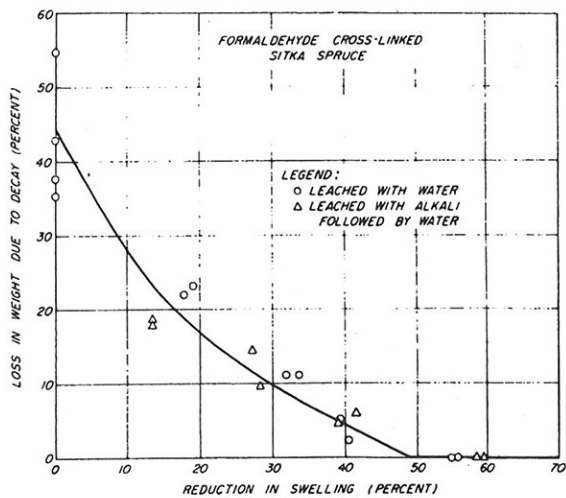


Fig. 6.—Relationship between loss in weight due to decay and reduction in swelling of formaldehyde cross-linked Sitka spruce with 1 percent of zinc chloride at catalyst.

pletely replace the water in swollen wood and are then cured.

Besides mechanical blocking the virtual elimination of decay can be explained on the basis of the reduction in hygroscopicity, which makes it impossible for the cell walls to take up enough water to support decay. This follows from the fact that the hygroscopicity and swelling are reduced by practically the same amount as in the case of acetylation.

When wood is heated to the point where some thermal degradation occurs, water of constitution and small amounts of carbon dioxide are given off. In the early stages, about 83 percent of the weight loss is that of water (7). The hemicellulose portion of the wood is thermally degraded most rapidly, followed by cellulose portion and then the lignin (11). In the early stages of thermal degradation, the effect on lignin can be neglected.

If the early loss of water from the combined hemicellulose and cellulose portion of the wood (72 percent) is assumed to involve the formation of internal ethers or some other rearrangement product, the optimum amount of water that can be lost in this way is 3 molecules of water from two anhydroglucose units, or 16.7 percent of the carbohydrate or 12 percent of the wood. Decay was practically eliminated when the total weight loss due to the thermal reaction resulted in 10 to 12 percent weight loss (Figs. 2 and 5), or a water loss of 8.3 to 10 percent. This weight loss accounts for 70 to 80 percent of the possible weight loss that could occur in forming internal ethers. It would be expected that the loss of water of constitution would occur first from the free hydroxyl groups and later from the hydrogen bonded hydroxyl groups within the crystallites.

The experimental weight loss of water of constitution necessary to prac-

tically eliminate decay will account for conversion of all of the readily available hydroxyl groups to internal ethers or some other rearrangement product, together with about half of the hydroxyl groups in the crystallites. This is considerably greater than the proportion of the hydroxyl groups that had to be eliminated in acetylation. The difference might be explained as follows:

The loss of water of constitution may not have been entirely due to splitting out of water between two hydroxyl groups. Part of it may have been between single hydroxyl groups and adjacent hydrogen atoms. If the reaction were entirely of the latter type, the experimental water loss would have occurred from half as many hydroxyl groups.

The decay resistance of the thermally changed wood can also be explained on the basis of the reduction in hygroscopicity. In this case a reduction in swelling of only 42 percent corresponds to practically complete elimination of decay, whereas a reduction of 70 percent was required with both acetylation and resin treatment. This is probably because the latter reduction in swelling resulted from bulking, which opens up the cellwall structure and makes it more accessible to decay.

In the case of the formaldehyde-treated wood, it may be suspected that the small amount of chemical required to prevent decay is because part or all of the formaldehyde is present in the form of a toxic polymer that is sufficiently insoluble to resist leaching. Reese (8) has discounted this possibility for formaldehyde-treated cotton and rayon containing 1 to 3 percent of combined formaldehyde. He showed that, by mixing the treated cellulose, which showed no decay, with untreated cellulose that decayed readily, the attack by the organisms was proportional to the amount of untreated cellulose present. If formaldehyde were present as an insoluble polymer, it should have prevented decay as a result of a slight vapor pressure of formaldehyde throughout the mixture. This is further support for the belief that all of the formaldehyde in the leached specimens is reacted with the wood, probably in the form of cross-links between structural units.

If all of the hydroxyl groups of cellulose could be cross-linked with formaldehyde, three cross-links would result for every two anhydroglucose units. The weight of formaldehyde reacted with the cellulose would be 27.8 percent ( $\frac{3 \times 30}{2 \times 162} \times 100$ ), the weight reacted with lignin would be 5.35 percent ( $\frac{3 \times 30}{2 \times 840} \times 100$ ), and the weight

reacted with wood would be 21.5 percent. From Figs. 1 and 6 it can be seen that only 2 percent of formaldehyde is needed to virtually prevent decay. This is only 9.3 percent of the theoretical total needed to eliminate all hydroxyl groups, or 18.6 percent of that necessary to eliminate all readily available hydroxyl groups in the amorphous regions and on the surface of the crystallites. This is far short of the amount of cross-linking that would be needed to prevent decay on the basis of elimination of available hydroxyl groups.

The virtual elimination of decay at such a low level of removal of hydroxyl groups thus appears to be due to the cross-linking, which ties the structural units of the wood together so effectively that the enzymes of decay organisms are prevented from entering the structure. This is not surprising when it is realized that these cross-links effectively minimize the entrance of water into the cell walls.

Decay is virtually prevented when the swelling is reduced by about 50 percent. This is only a little over half of the dimensional stabilization required for acetylation and resin treatment to eliminate decay. The two latter treatments, as stated before, involve bulking, so that more stabilization is necessary to keep the actual separation of the structural units below a minimum beyond which fungus enzymes can penetrate the structure.

In the case of the wood treated with polyethylene glycol, the decay resistance must be due to some physical effect, since the chemical in small concentrations is not toxic and does not react with wood, as evidenced by the ease with which it can be leached from the wood. The fact that agar gels containing 25 to 75 percent of polyethylene glycol do not support fungus growth in agar flask tests can be explained on the basis of an osmotic effect of the polyethylene glycol, which takes sufficient water from the fungus to prevent their future growth when transplanted to plain nutrient agar slants.

The effectiveness of polyethylene glycol when present in wood to the extent of 18 percent (corresponding to a reduction of the shrinkage to 0.58 of normal (Fig. 1)) in preventing decay can be explained on the basis that there is insufficient water within the cell walls to support decay even when the wood is completely swollen in water (about 12 percent).

In summary, the virtual elimination of decay by acetylation and by the thermal reaction can be explained on the basis that readily available hydroxyl groups are replaced by nondecay-

susceptible groups. The virtual elimination of decay attained by depositing a phenolic resin within the structure and by cross-linking with formaldehyde can be explained on the basis that the hydroxyl groups are physically blocked.

Only the polyethylene glycol treatment's effectiveness in preventing decay can be explained on an osmotic action basis, and even in this case there is some possibility that the chemical forms a loose chemical bond with the cell wall.

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