Phimisten Procton, In.

# RESIN-TREATED, LAMINATED, COMPRESSED WOOD

May 1941



FOREST RESEARCH LABORATORY
LIBRARY

R1268

UNITED STATES DEPARTMENT OF AGRICULTURE FOREST SERVICE FOREST PRODUCTS LABORATORY Madison, Wisconsin

In Cooperation with the University of Wisconsin

# RESIN-TREATED, LAMINATED, COMPRESSED WOOD

By A. J. STAMM, Principal Chemist and R. M. SEBORG, Assistant Chemist

The authors have shown in previous publications (3, 4, 5) that the formation of synthetic resins within the intimate cell-wall structure of wood from polar resin-forming constituents greatly increases the moisture resistance of the wood. Moisture adsorption and swelling and shrinking have been reduced to one-fourth of normal under true equilibrium conditions and to a considerably greater extent under normal use conditions. Plywood made entirely from the treated plies or made with only the face plies treated showed a marked decrease in face checking under weathering conditions and a marked decrease in the passage of moisture through the plies under a relative humidity gradient (4, 5). The decay resistance of the wood was appreciably increased (4, 5) and the compressive strength properties were increased in greater proportion than the increase in weight of the wood (3).

A number of different resin-forming materials have been tried. The most successful of these is an alkaline catalyzed, practically unpolymerized, phenol-formaldehyde resin-forming mix with a pH of about 8 that is soluble in water in all proportions. The urea-formaldehyde resin-forming systems tried were considerably less effective than the phenol-formaldehyde systems in permanently reducing shrinking and swelling. This is perhaps partially due to the fact that the systems were initially too far polymerized for the resin-forming mixes to diffuse appreciably into the fine cell-wall structure. Vinyl, styrine, Glyptol, and methyl methacrylate resins were practically ineffective in permanently reducing the hygroscopicity of wood. This appears to be due to the fact that the monomers of these resins do not have sufficient affinity for wood and do not enter and bond to the cell-wall structure.

The extent to which wood swells in a resin-forming solution beyond the swelling in the solvent alone is a good gauge of the affinity of the wood for the resin-forming constituents. Aqueous phenol-formaldehyde resin-forming solutions cause considerably more swelling of wood beyond the swelling in water than is caused by any of the other resin-forming systems tried. The only resin-forming systems tried that are not so effective in reducing the shrinking and swelling of the wood subsequent to the curing of the resin as would be expected on the basis of the swelling of the wood in the original resin-forming mix are very high alkalinity commercial phenol-formaldehyde resin-forming mixes. The high alkali concentrations are used to keep appreciably prepolymerized resin in solution. On treating wood with such

Presented before the American Institute of Chemical Engineers, Chicago, Ill., May 21, 1941. (To be published in Institute's journal.)

solutions, the wood swells because of the selective adsorption of alkali and not because the resin forming constituents are entering the cell-wall structure.

## Treating

Bakelite Resinoid XR5995, a commercial phenol-formaldehyde resin-forming mix that is miscible in water in all proportions, was used as the treating material in all the experiments. It has better keeping qualities than any of the mixes that have so far been made at the Forest Products Laboratory.

The veneer can be treated by a diffusion of the resin-forming mix into green veneer direct from the cutter knives by merely soaking it in an aqueous solution of the mix (4, 5). The time required for the veneer to take up the desired amount of resin-forming constituents will vary directly as the square of the thickness of the veneer, directly as the specific gravity of the wood, and inversely with the moisture content of the wood and the temperature. Green sweetgum veneer 1/32 inch thick took up about 40 percent of its dry weight of resin-forming constituents from a 50 percent aqueous solution of Bakelite Resinoid XR5995 by diffusion in 1 hour at 100° F.

The veneer can also be treated in the dry condition by the cylinder treating method. The veneer is placed on edge in a galvanized iron tank inside of the treating cylinder. The tank is filled with the treating solution and the veneer weighted down so that it will remain immersed. The cylinder is closed and a pressure of 30 to 60 pounds per square inch of air pressure is applied for 1/4 to 1 hour, depending on the resistance to penetration of the wood. In the earlier experiments a vacuum was pulled before applying the pressure (4, 5), but this was found to be unnecessary for most of the veneers treated in thicknesses up to 1/8 inch. It was found that a take-up of solution equal to the dry weight of the wood was desirable. The concentration of the mix in water was adjusted so that the final resin content of the dry wood would be from 30 to 40 percent. This could, in general, be obtained when the mix consisted of one-half to two-thirds water.

When the veneer is treated by the cylinder method the solution is carried only into the coarse capillary structure. It takes time for it to diffuse into the cell-wall structure where it is desired. It is, therefore, necessary to stack the treated veneer under nondrying conditions for 1 to 2 days with a canvas thrown over it to cut down circulation.

The veneer is then slowly dried so that the resin-forming constituents can diffuse from the fiber cavities into the cell walls as water is removed from the fiber cavities. Drying on the drying chain of a continuous drier under normal veneer drying conditions is too rapid. The veneer can be dried nost satisfactorily by putting the sheets on edge in a drying rack with 1/8-inch spacers between and placing these racks in a dry kiln. The sheets should be inserted between the spacers with the spacers at right angles to the fiber direction of the sheets. Brass rods or resin-coated steel rods make good spacers. The drying can be done at a temperature of 150° to 160° F. or perhaps even a little higher without premature setting of the resin.

The relative humidity should be maintained at about 65 to 70 percent to make possible the continued diffusion of the resin-forming constituents. The conditions will give an equilibrium moisture content of 8 to 9 percent. This moisture content is attained in 3 to 4 hours under these conditions with the woods tested.

## Compression and Assembly

Wood treated and dried according to these methods is much more plastic at the normal hot-pressing temperatures of 300° to 350° F. than untreated wood. For example, spruce, cottonwood, or aspen can be compressed to one-half their original thickness under as small a pressure as 250 pounds per square inch. This pressure causes only a slight compression of the dry untreated wood.

There is an insufficient amount of resin-forming constituents on the surface of the dry resin-treated wood to give a bond between plies that are not compressed. When the wood is highly compressed, enough resin-forming constituents exude from the plies to give a bond without using additional bonding material. When the plies are all parallel a good bond is obtained without the use of additional bonding material, even when the compression is only about half of the total possible amount. Under conditions where insufficient resin-forming materials exude from the structure to form a good bond, Tego film has been used for the bonding.

Resin-treated compressed wood has been made up from the dry treated plies under pressures of 250 to 1,200 pounds per square inch at temperatures of 300° to 320° F., using pressing periods of 15 to 30 minutes per inch of original thickness of the wood. It is, in general, desirable to cool the wood below the boiling point of water before removing it from the press in order to avoid possible rippling or crazing of the surface. The cooling in the press, however, is unnecessary in the case of such soft, even-textured hardwoods as cottonwood or aspen.

The pressing time varies almost directly with the thickness rather than as the square of the thickness, as might be expected. This is due to the heat generated within the wood from the resin-forming reaction, contributing to the energy necessary to initiate the reaction a little farther towards the center of the wood. A thick, compressed panel 2.5 inches thick was made from a 6.5-inch thick pile of treated plies with thermocouples inserted at various places between the plies. In 3 hours the temperature at the center became equal to the platen temperature, and in another hour reached a temperature of 80° F. above that of the platens. In making thick material it is thus desirable to drop the platen temperature before the center of the wood reaches the platen temperature to avoid overcuring or burning.

Under a pressure of 250 pounds per square inch, spruce, cottonwood, and aspen are compressed to less than half their original thickness and the product has a specific gravity of at least 1. The same woods, under a

pressure of 1,000 pounds per square inch, compress to one-third of their original thickness and give products with specific gravities ranging from 1.3 to 1.4. This corresponds closely to complete compression as the density of wood substance is 1.46 (2). Different species of poplar and gum compress but slightly less under the higher pressure.

#### Properties

Compressed wood made in this way is far more homogeneous and water resistant than the commercial compressed woods now on the market. The resin within the cell-wall structure and bonding the plies together forms a continuous homogeneous structure. The resin is formed in the intimate cell-wall structure and chemically borded to the hydroxyl groups of cellulose and lignin that normally take up water rather than being mechanically deposited Figure 1 shows the difference between in the coarse capillary structure. the swelling of small blocks of a commercial resin-treated compressed wood and resin-treated compressed wood made according to the Forest Products Laboratory method. The former swelled more in thickness than the theoretical value for wood of the same specific gravity (about 20 percent). This is due to the fact that a considerable part of the dimension change of the commercial product was a relieving of compression rather than a true swelling. The commercial product was badly checked as a result of the swelling, whereas the Forest Products Laboratory product retained its smooth, glossy appearance.

The small swelling of the Forest Products Laboratory product occurs very slowly. This is shown by the percentage weight increase, after different times of immersion in water, of specimens (10 by 10 by 1 cm.) of compressed spruce containing 40 percent of resin that were pressed at 1,000 pounds per square inch for 25 minutes at 310° F. The weight increases after 1, 4, and 7 days were 0.5, 1.2, and 1.8 percent, respectively. The German specifications (1) allow a weight increase for laminated, resin-treated, compressed wood specimens of the same dimensions after the same periods of immersion of 5.0, 7.0, and 8.0 percent, respectively.

The Forest Products Laboratory compressed wood has a very hard, smooth, weather-resistant surface. Surface hardness values obtained with a Sword hardness tester varied from 65 to 90 for different species and amount of resin present, as compared to 100 for plate glass. Ordinarily smooth spruce gave a value of 6. The latter, with a good coat of varnish, gave a value of 13.

Mechanical tests have not been carried sufficiently far so as to compare critically the resin-treated, laminated, compressed wood made by the Forest Products Laboratory method with that made according to present commercial practice. Indications are, however, that the Forest Products Laboratory material will have practically equal, if not better mechanical properties, as preliminary strength values for compressed wood made from the mechanically inferior species are about the same as those reported for the commercial product made from maple and beech when compressed to the same specific gravity. The data indicate, however, that variations in the mechanical properties of

\_4\_

compressed wood of the same specific gravity made from different species will be considerably less than the variations in mechanical properties of different species of normal woods.

A few samples of parallel laminated, resin-treated compressed spruce with a specific gravity of 1.3 gave maximum tensile strengths parallel to the grain of over 40,000 pounds per square inch, modulus of rupture in static bending of over 40,000 pounds per square inch, maximum crushing strengths with the compression parallel to the grain of over 20,000 pounds per square inch, and modulus of elasticity values from the previous three properties ranging from 4 to 5 million.

# Combination of Compressed and Uncompressed Wood

It would be desirable to be able to produce a plywood with the hard, dense, water-resistant surface and with the improved mechanical properties that have just been described without a greatly increased weight and cost above that of ordinary plywood. This has been accomplished by compressing the resin-treated face plies and assembling them with an uncompressed core in a single operation. Such an assembly is made possible by the plasticizing action of the resin-forming constituents on the treated plies. Under a pressure of 250 pounds per square inch the face plies of a number of species can be compressed to half of their original thickness, whereas a dry untreated core of the same wood or a more compression-resistant wood will be compressed only about 5 to 10 percent. If cost is not a serious item and a high water resistance of the core, as well as of the faces, is desired, all the plies may be treated and the core plies precured by heating in an oven without an applied pressure. In this way the compressive strength of the core is increased by about 50 percent (3) and the core is even less subject to compression on assembly than are untreated cores. When assembling a treated uncured ply with an untreated ply or a treated ply in which the resin has been precured, it is desirable, but not always necessary to use an additional bonding material. If the resin-forming constituents are absorbed by the untreated wood as rapidly as they exude from the treated ply that is being compressed, a starved joint may result.

The hard, glossy surfaces of the compressed wood are difficult to glue to each other or to ordinary wood because of the nature of the surfaces. If it is desired to glue one or both faces of compressed wood, it is advantageous to make it up with treated, but precured faces. The treated wood that has not been compressed can be readily glued with any of the commercial glues  $(\underline{4}, \underline{5})$ .

Because of the difficulty of gluing resin-treated compressed wood, the manufacture of a combination of compressed wood and uncompressed wood in two steps would result in a weaker or less water-resistant bond than is obtained in the single compression and assembly method which is here described.

R1268

Specimens of plywood with 1/16-inch resin-treated compressed faces and an untreated 3/8-inch, 3-ply core were soaked in water for several days. The untreated cores took up water and swelled in thickness rather than transversely as a result of the lateral restraint due to the cross banding. After air drying, followed by oven drying, the specimens showed no face checking due to the stresses. Similar specimens were put through a temperature change cycle of 24 hours at 23° F. and 24 hours at 150° F. for over a month. No visible degrade occurred. Specimens exposed to the weather for a period of 6 months have shown no face checking in contrast to an appreciable face checking of the controls with untreated uncompressed faces.

#### Finishing

If the resin-treated compressed wood is scratched or marred in any way, the scratch can be sanded out and the specimen buffed, thus restoring the original finish.

Dyes have been added to the resin-forming mix. A few dyes, chiefly of the vat-dye type, uniformly penetrated the structure of woods like cotton-wood and aspen. All the dyes faded to some extent on light exposure. A few, however, show promise for out-of-door use and more of them show promise for indoor use.

Enamel paints used to paint insignia on airplanes gave a smooth 1-coat finish on the resin-treated compressed faces of spruce, whereas a single coat on the untreated wood showed an obvious need for building up of the finish. Weather exposure tests have not been sufficiently long to indicate the life of the 1-coat job on the resin-treated compressed faces. After 6 months' exposure, however, they look very good in contrast to the faces of the untreated, uncompressed controls which checked through the finish.

#### Possible Uses

The resin-treated compressed woods described in this paper show several possible uses in airplane construction. The most promising of these is the use of the material with the compressed faces on an uncompressed core for fuselage and wing covering. Because of the increased plasticity of the treated plies, they should respond nicely to the various types of bag molding now being tried. It seems hopeful that if the molding is carried on at pressures somewhat in excess of those now used that the highly desirable surface finish, water resistance, and improved mechanical properties can be imparted to the surface without appreciably increasing the weight of the material.

The highly compressed wood with resin-treated but uncompressed faces shows promise for use as spar plates which can be readily glued to the ends of the spars to take the bearing stresses where they fasten on the fuselage.

R1268 -6-

The treated veneer could also be used to advantage in making propeller blanks varying in specific gravity from one end to the other, or even in molding the propellers to their final shape. Figure 2 shows one of a number of possible ways of laying up the treated veneer to obtain blanks with varying specific gravity from one end to the other. Figure 3 shows the finished product. Three wedge-shaped piles of spruce veneer were placed in the press side by side with the high end of the middle pile at the opposite end from the high ends of the others and pressed simultaneously so as to distribute the stresses over the press platens. The highly compressed end of the specimens was subjected to a pressure of over 1,000 pounds per square inch, whereas the other end was subjected to a pressure just great enough for assembly, giving a specific gravity of about 1.35 at one end and 0.48 at the other.

Laminated wooden propellers are now being made (Schwarz type) with compressed hubs by scarfing half inch thicknesses of resin-treated compressed wood to normal spruce. The compressed wood, however, does not have so high a water resistance as is desirable. Moreover, the process entails the gluing of compressed wood to compressed wood and compressed wood to normal spruce with cold-press glues so that the finished product does not have the water resistance nor homogeneity of the Forest Products Laboratory product.

There is a distinct possibility that if the plies are pretailored, propellers can be molded in a suitable mold to the finished dimensions, thus giving a highly finished water-resistant finish to the whole blade. It is also possible to vary the specific gravity of the propeller blade at right angles to the blade direction, as well as in the blade direction. The tip of the propeller could be uncompressed with a compressed sheathing around it. This could be accomplished by treating only the outer plies near the tip.

Several other types of uses for the combination of compressed resintreated faces on an uncompressed core present themselves. This material should be highly satisfactory for flooring. The hard, finished surface should be very resistant to marring and grain raising, while the uncompressed core should furnish the desired resilience. The upkeep cost should be negligible as no finish is necessary. To maintain stress balance when used in thicknesses less than 3/4 inch, a bottom treated but uncompressed ply should be used. The uncompressed but treated ply will nail or glue readily. The material can thus be edge nailed or glued to the subflooring. The surface polish may prove excessive for home use. This can be avoided by partially curing the face plies at the time of drying.

The plywood with compressed faces could be used to advantage in furniture manufacture. It is of interest to note here that the resin-treated compressed faces are highly alcohol resistant as well as water resistant. The possibility of refinishing by merely sanding and buffing is also of considerable importance.

The resin-treated compressed faces serve as better moisture barriers than do the uncompressed resin-treated materials ( $\frac{14}{2}$ ,  $\frac{5}{2}$ ). The compressed material should thus be of considerable value for interior paneling.

-7-

# Summary

The treatment of veneer with an unpolymerized water-soluble phenol-formaldehyde resin-forming mix in such a way that the resin-forming constituents are deposited within the fine cell-wall structure and chemically bonded to the structure making possible the compression of the treated plies under much lower pressures than would otherwise be necessary and gives the product a water resistance not obtainable by other processes. The treatment also makes it possible to manufacture plywood with compressed resin-treated faces and an uncompressed core in a single compression and assembly operation. Properties of the materials are given and possible uses in airplane construction, flooring, paneling, and furniture manufacture are discussed.

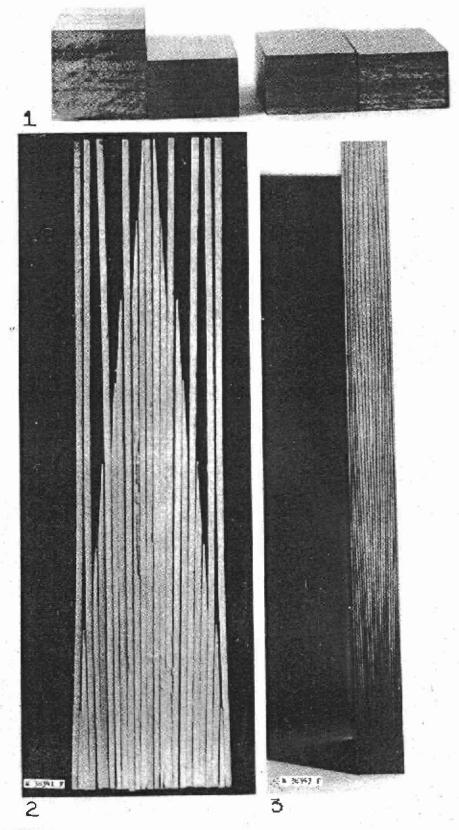
#### Literature Cited

- (1) Armbruster, F. Kunststoffe 30:58-62 (1940).
- (2) Stamm, A. J., and Hansen, L. A. J. Phys. Chem. 41:1007-1016 (1937).
- (3) Stamm, A. J., and Seborg, R. M. Ind. Eng. Chem. 28:1164-1169 (1936).
- (4) Stamm, A. J., and Seborg, R. M. South. Lbrman. 157-162 (Dec. 15, 1938).
- (5) Stamm, A. J., and Seborg, R. M. Ind. Eng. Chem. 31:897-902 (1939).

#### FIGURE TITLES

- Figure 1.--Swelling of resin-treated, laminated compressed wood.

  From left to right:
  - 1. Commercial material, water soaked for 50 days (54 percent swelling).
  - 2. Commercial material, air dry.
  - 3. Forest Products Laboratory material, water soaked for 50 days (3.6 percent swelling).
  - 4. Forest Products Laboratory material, air dry.
- Figure 2.--One means of stacking resin-treated veneer for compressing to form material with a varying specific gravity from one end to the other.
- Figure 3.—Specimen of resin-treated, laminated, compressed wood with varying specific gravity from one end to the other, pressed from material that was stacked as shown in Figure 2.



а м 39045 F