ADHESIVES, THEIR USE AND PERFORMANCE IN STRUCTURAL LUMBER PRODUCTS

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This topic is probably too broad for adequate coverage in the time allotted me. Furthermore, this group probably would not be interested in a detailed discussion of the numerous types of adhesives now available for bonding wood, particularly those that are not of structural quality. Consequently, I will limit my remarks to the types of adhesives that currently play the major role in the structural laminating field, namely, casein glue and resorcinol and phenol-resorcinol resin adhesives. Since this audience is probably most interested in types of adhesives that are highly durable under practically any type of service, the greatest part of my talk will be devoted to the resorcinol and phenol-resorcinol types, as well as their use and performance in exterior service.

**Casein Glue**

The basic constituent of casein glue is dried casein, which, when combined with alkaline chemicals (usually lime and one or more sodium salts) is water soluble. Prepared casein glue comes in powder form, and when mixed with water in the correct proportions, is ready for use.

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1 For presentation at Symposium on Wood for Railroad Uses, May 26, 1960, Chicago, Ill.

2 Maintained at Madison, Wis., in cooperation with the University of Wisconsin.
It sets as a result of chemical reaction and loss of moisture to wood and air. Hence, its rate of setting is affected by the temperature of the wood and surrounding atmosphere, the moisture content of the wood, and other factors. Longer setting time is required in a cold shop than in a warmer one, and wood high in moisture content will retard the setting rate as compared to wood that is somewhat lower in moisture content.

Casein glue will set at temperatures almost as low as the freezing point of water, but the setting period required to develop strong joints at such temperatures varies from several days to several weeks. The time will depend also on the species glued and the moisture content of the wood. The wet strength developed at low temperatures may never be as good as that developed at room temperature. A pressing period of 4 hours at 70° F. is considered a minimum for straight members, and for curved members a somewhat longer period is desirable.

Casein glue will produce adequate bonds with wood at a wide range in moisture content from 2 to 18 percent, at least. It is recommended, however, that the lumber be dried before it is glued to a moisture content slightly below that expected in service. It is also highly desirable that all laminations for one assembly be of approximately the same moisture content (allowable differences between boards in the same assembly up to 5 percent) to avoid unequal shrinking or swelling as the moisture content equalizes in service.

Well-made casein glue joints will develop the full strength of the wood, especially in softwood species, and will retain a large part of their strength even when submerged in water for a few days.
To improve resistance to deterioration caused by molds or other micro-organisms, preservatives such as copper salts, mercury salts, or the chlorinated phenols or their sodium salts are sometimes added to casein glues. Amounts of chlorinated phenols in the neighborhood of 5 percent of the weight of the dry casein will often result in marked improvement in mold resistance without serious effect on the working properties of the glue. Prolonged exposures to conditions favorable to mold growth or other micro-organisms, however, will eventually result in failure even of preserved casein glue joints.

Under outdoor conditions or where high humidities, either continuous or intermittent, are involved, casein glue joints are not durable. Casein glues containing preservative have shown greater resistance to high humidities than have unpreserved casein, but the preservative did not prevent eventual destruction of the glue bonds under damp conditions. Consequently, casein glue is not considered suitable for laminated members intended for exterior use or for interior use where the moisture content of the wood may exceed about 18 percent for repeated or prolonged periods. Currently, the structural laminating industry generally limits casein-glued material to service where the equilibrium moisture content of the wood does not exceed 16 percent.

Casein glue joints have demonstrated good resistance to dry heat. Results of test exposures to temperatures as high as 158° F. for periods up to 4 years have indicated that the glue bonds are about as resistant
as the wood to this type of exposure. Temperatures that char and burn wood, however, will cause decomposition of casein glue. Charred wood exposed to fire, however, conducts heat to its interior very slowly, so that softening of casein glue joints takes place only next to the burning wood.

Laminated structures bonded with casein glue have given excellent service, when protected from exterior and damp conditions, for 25 years or more in this country. In Europe, similar structures 30, 40, and up to 50 years old are not uncommon. This should be adequate basis for confidence in casein glue as a structural bonding agent.

**Resorcinol and Phenol-Resorcinol Resin Adhesives**

The resorcinol adhesives are produced by the reaction of resorcinol with formaldehyde, and are marketed as liquids that consist of partly polymerized resin, usually in a water-alcohol solution. Because of the high cost of resorcinol, modified phenol-resorcinol resin adhesives, which are produced by copolymerizing the two resins during the resin manufacturing process, are now much more commonly used. Both resorcinol and phenol-resorcinol adhesives are dark red in color, and make dark joints when they set. A hardener, usually paraformaldehyde but sometimes formalin, and a filler, commonly walnut-shell flour, are mixed with the resin prior to use. In most cases, a mixture of hardener and filler is furnished by the manufacturer. Both resin and hardener may be stored for several months, and sometimes as much as a year or more, at ordinary room temperatures.
when kept in airtight containers. In general, the straight resorcinol adhesives have longer storage life than do the phenol-resorcinol types. They will produce adequate bonds on wood at a moisture content that ranges from at least 6 to 17 percent. As with other adhesives, however, the preferable moisture content, within this range, is slightly below that expected for the laminated member in service.

The curing requirements for these glues vary somewhat with the particular brand and with the species of wood used. When dense species such as white oak are laminated, curing for several hours at 150° F. is generally required. When softwoods such as Douglas-fir and southern yellow pine are laminated, curing at temperatures as low as 70° F. will generally give adequate bonds, but impractically long curing periods may be required at this temperature. Consequently, to speed up the cure, heating at temperatures of 110° to 120° F. is often the practice when these species are glued.

The durability of well-made phenol-resorcinol- and resorcinol-resin glue joints, based on about 16 years' experience, appears to be essentially equal to that of hot-pressed phenol-resin glue joints. They are highly resistant to moisture, high temperatures, chemicals, and micro-organisms. Thus, in general, joints properly made with these adhesives are as durable as the wood itself under practically any type of service.
Laminated Timbers for Severe Service Conditions

Research on glued-laminated wood products suitable for unprotected exterior use dates back to the development of resorcinol and phenol-resorcinol glues in about 1943. Glues available before that time either lacked the necessary water resistance or required very high curing temperatures, such as those used for exterior-type plywood. Because the resorcinols provided highly durable bonds when cured at moderate temperatures, interest soon shifted to the problem of making the wood as durable as the glue under conditions of service that favor decay.

There appear to be essentially two feasible methods for making preservative-treated laminated members: (1) by treating the already glued and machined laminate, or (2) by treating the lumber and then gluing members of the required size and shape. Both methods have advantages and disadvantages. Treatment of the laminated member permits application of preservatives after all cutting, boring and other framing has been done, and assures a protective coating on all exposed surfaces. Handling of material at the treating plant is often simplified when the finished members rather than the lumber are treated. Probably the most serious disadvantage of this method is the necessarily limited size of treating cylinders, which precludes treatment of larger timbers, and particularly of large curved ones. Penetration of the preservative has been found to be blocked to a certain extent by the glue line, and this, of course, is
an obvious disadvantage. Furthermore, when only an outer layer of a member is treated, checks that develop later in service may allow decay to start. On the other hand, bridge timbers produced by this method (pressure treated with creosote or creosote and oil mixtures after gluing) are in excellent condition after up to about 16 years of service.

Gluing of Treated Lumber

The need for laminating treated wood under certain conditions is rather obvious. Treating after the wood is glued, as already indicated, presents certain problems, particularly where large curved members are involved. Gluing treated wood is about the only way of obtaining curved treated members of appreciable size. By proper selection of sapwood or other easily penetrated materials, thin laminations of certain species such as red oak, southern yellow pine, and western hemlock can be completely penetrated with preservative chemicals. Laminated members produced from such stock can safely be shaped and bored without exposing untreated material. A disadvantage of the gluing of treated wood is the apparent necessity to surface each treated lamination before it is glued. This removes parts of the treated wood and may expose unpenetrated wood if complete penetration has not been obtained.

Modifications in gluing procedures are often required from one species to another, even with untreated wood, to obtain adequate bonds with a particular type of adhesive. When dense woods are glued, for
instance, more heat is required for adequate cure of a resorcinol-
type of adhesive than is needed when softer woods are glued. In a
similar manner, the optimum assembly period for a glue used on a highly
absorbent wood may vary considerably from that of a species difficult
to penetrate.

Treating the wood with various preservatives further complicates the
gluing problem. The oil-borne preservatives, which in general have caused
the greatest interference with adhesive bonding, might prevent proper
contact between adhesive and wood. Even though the wood is freshly
planed before the adhesive is spread, migration of oil over the cut
surface fibers is likely to occur, and thus make intimate contact between
the adhesive and the treated wood difficult. Minor experiments have
indicated that this hypothesis is very likely true. With a light solvent,
much less interference with bonding has been experienced than when a
heavier solvent such as No. 2 fuel oil has been used with pentachlorophenol
and copper naphthenate preservatives.

When water-borne preservatives are used, the presence of alkaline
or acid salts at the wood--adhesive interface may retard or prevent the
mechanism of adhesive bonding. Research has shown conclusively that
preservatives in general retard the cure of resorcinol and phenol-resorcinol
adhesives, and make necessary extended curing schedules for bonding treated
as compared to untreated wood. This retarding effect on the cure of
adhesives varies somewhat from one preservative to another.
It appears, therefore, that two main avenues of attack would be logical in attempting to solve the problem of gluing treated wood: (1) to modify the procedures that have proved satisfactory for gluing untreated wood, or (2) to modify available adhesives or possibly develop adhesives especially for treated wood. The research conducted up to this time on gluing treated wood has been directed mainly toward finding procedures that would bond treated wood with available adhesives. Of late, there have been reports from the glue industry that work towards development of adhesives tailored particularly for treated wood is underway. No real break-through along this line, however, has yet been reported.

Results of Glue Joint Tests on Glued Treated Wood.—Considerable laboratory data have been developed that show it is feasible to glue wood that has been treated with a number of preservatives. Table 1 shows results of block shear and delamination tests on laminated red oak beams glued from lumber treated with various preservatives.

Tanalith (Wolman Salts) showed the least interference with bonding, and this factor has been experienced in other studies also. The retention used with Tanalith was lower than with other water-borne preservatives, and this may have influenced the results, but good results have been obtained with Tanalith-treated red oak with a retention slightly above 1 pound per cubic foot.
Tanalith-treated wood has also been glued to a considerable extent on a commercial scale, and the adequacy of the process has been proven by hundreds of laboratory tests as well as by service records.

Although the bond quality of wood treated with other preservatives in general was not entirely on the same level as that for the Tanalith-treated wood, the results, nevertheless, appeared satisfactory. Whether lower retentions would have resulted in improved bonds is not known, but it is possible they would have, and this factor would be worth investigating.

The methods used for evaluating glue joints in treated wood were the same as those commonly employed for evaluating untreated glued wood: block shear and delamination tests. These are ASTM procedures with the designations ASTM D805-52 and ASTM D1101-53, respectively.

All lumber was resurfaced immediately before it was glued, and this appears to be necessary regardless of what type of preservative is used, or at least with those that leave a deposit on the wood surface as the solvent evaporates. As was mentioned earlier, the type of solvent used has a marked effect when oil-treated wood is glued; the more volatile types cause less interference than the heavier ones. In a minor study where the treating solution was pentachlorophenol in naphtha, reasonably good glue bonds were obtained even when the lumber was not resurfaced after treatment. With all other preservatives, resurfacing appeared to be absolutely necessary.
There also appears to be a definite species effect when gluing oil-treated wood; the more easily treated species show less interference with gluing than the ones that are difficult to treat. Species that appear dry on the surface, even though they have a high retention of oil, can usually be glued satisfactorily, whereas those that "bleed" and present an oily surface in general bond erratically.

There is also evidence that the technique of spreading the glue might affect the bonding of oil-treated wood. It was mentioned earlier that a film of oil might actually prevent adequate contact between the wood and the adhesive. Minor experiments have indicated that passing the laminations through the glue spreader several times, instead of the customary single pass, resulted in improved bonds. It is conceivable that the several passes through the spreader tend to break down the oily films on the wood surfaces, and thus allow more intimate contact between wood and glue.

With woods that have a tendency to bleed, a thorough steam cleaning or cleaning by other methods prior to final surfacing and gluing would contribute to improved glue bonds. Hence, the method of treatment is also rather important when treating wood for gluing.

Long-Term Tests on Laminated Members Treated After They Were Glued

Initial high strength of glue joints is no guarantee that they are durable; durability can be established beyond doubt only by long-term tests or service. Resorcinol and phenol-resorcinol glues have shown
excellent durability on untreated wood over the more than 1-1/2 decades they have been in use. Results are also available on laminated wood treated after it was glued and then aged up to 12 years before it was tested. The tests were conducted for the purpose of determining whether preservatives have any deteriorating effects on synthetic resin glue bonds of the melamine, resorcinol, and phenol-resorcinol types over long periods of aging.

The study was wider in scope than is feasible to include in this talk, and only some typical results on laminated red oak, southern yellow pine, and Douglas-fir will be discussed. Conditions of some of the tests were such as might be encountered in outdoor exposure of structural members of bridges, railroad trestles, towers, and similar structures.

Preservatives Used, Method of Testing, and Results.--Commercial preservatives (creosote, pentachlorophenol, chromated zinc chloride, and Tanalith) were applied by pressure treatment to sections from twenty-eight 12-foot laminated beams glued with 2 resorcinol, 1 phenol-resorcinol, and 1 melamine resin adhesive. The preservative retentions conformed closely to the requirements of the current Federal Specification TT-W-571C: Wood Preservatives; Recommended Treated Practice.

The glued beams were 6 by 6 inches in cross section, 12 feet long, and contained eight 3/4-inch laminations. The adhesives used for gluing the beams were cured according to schedules that previously had been found to produce glue joints in white oak, Douglas-fir and southern yellow pine.

that were strong and durable in prolonged exposure to weather and soaking. Each beam was cut into five 12-inch sections for weathering tests, five 3-inch sections for cyclic delamination tests, and fifteen 3-inch sections for block shear tests. This provided end-matched material for groups of four treatments and controls in the different tests. Only results of the block-shear tests and the weathering exposure will be discussed here.

Block shear tests were made immediately after treatment and after the specimens were aged for 2, 6, and 12 years in an unheated shed. The shear specimens were not exposed to the weather because it was desired to try to isolate the effects of the preservatives, if any, on the glue joints. Because of the shrinking and swelling that usually occurs under outdoor exposure, effect of the treatments on the glue joints might be masked by other factors. The 12-inch sections exposed to the weather were examined visually, and typical samples were photographed. In most cases, the test results on the treated material were approximately equivalent to those on untreated controls, both initially and after the various periods of aging up to 12 years. In the weathering test, however, the untreated control specimens of southern yellow pine showed some deterioration from decay after 12 years.

Figures 1 to 3 show the block-shear strengths of laminated red oak, Douglas-fir, and southern yellow pine treated after gluing and aged up to 12 years before testing. For practically none of the preservative-species
As combinations were there any indication of deteriorating effects of the treatment upon the glue joints. High shear strengths as well as high wood failures (latter not shown in figures) show that the initial high joint quality was maintained throughout the 12 years of aging.

Figures 4, 5, and 6 illustrate the condition of red oak, Douglas-fir, and southern yellow pine beam sections given various treatments and exposed for 12 years to the weather at Madison, Wisconsin.

The creosote-treated sections (marked B) in general showed the least effect of the exposure, in that they were almost entirely free from checking. Those treated with pentachlorophenol (marked C) were almost in the same category as far as Douglas-fir was concerned; the oak, however, showed some checking and a slight amount was also present in the pine.

The sections treated with water-borne preservatives checked about as much as the untreated controls, but the untreated pine, as indicated previously, appeared to be in excellent condition in all samples.

Service Records of Treated Laminated Timbers

At the current state of developments, treatment after the wood is glued is probably the most practical and economical method of producing treated laminated timbers. When laminated members do not lend themselves to treatment because of their size or shape, however, the gluing of treated material is the only known method of producing adequately treated members.
As already indicated, structures produced by treatment after gluing have now given adequate service for more than 15 years. The first installation on which records are available included 11 laminated stringers installed in the Texas and Pacific Railway, bridge No. 55.2, near Woodlawn, Texas, in 1944. The species were shortleaf pine grade C and better, except one stringer that was made of No. 1 common second-growth shortleaf pine. No. 1 distillate creosote was used in treating the timbers (by the Rueping process) to a retention of about 15.5 pounds per cubic foot.

These stringers were reported to have given entirely satisfactory service. They were inspected about a year ago, and aside from some end checking, appeared to be in excellent condition.

Another southern pine installation of laminated treated timbers is part of a trestle on the Southern Railway near Alexandria, Virginia. These timbers were installed in 1945 and will soon have been in service 15 years. They show practically no delamination and very little checking after nearly 1-1/2 decades.

Both 4/4 and 8/4 stock were used in making these timbers, and the caps had three 4/4 laminations of red oak on top and bottom to provide better bearing strength. Treatment was by the Rueping process, and the preservative was an 80-20 creosote coal-tar solution.

On the West Coast there are numerous installations of treated laminated Douglas-fir. Forest Service representatives and others make regular inspections of some of these installations, and the reports, in general, are very good. The exuded creosote makes the members shed water readily, and very little weather checking develops.
One highway bridge in Oregon is made with arches glued from wood treated with a water-borne preservative. The bridge was erected in 1948, and the laminated members now show an appreciable amount of checking as well as some joint separation. The salt treatment apparently does not provide the same protection against weathering as does the creosote treatment.

An installation of 20 railway stringers, which were glued from treated lumber, is in the bridge over the Palm River in East Tampa, Fla., on the Atlantic Coast Line Railroad. About half of these stringers were glued from creosote-treated southern yellow pine, and the remainder from Tanalith-treated pine. The last report stated that the stringers were in excellent condition. This would indicate that, at least with southern yellow pine, satisfactory gluing is possible even after treatment with creosote.

**Conclusion**

In conclusion it would appear that laminated timbers properly glued with resorcinol or phenol-resorcinol adhesives and adequately treated with a suitable preservative should provide satisfactory service under the most severe use conditions.

Treating the wood after it is glued, at least currently, appears to be the most practical method of producing such timbers. With certain species-preservative combinations, however, it is also feasible to produce satisfactory timbers by gluing the already treated lumber.
Table 1.—Results of block-shear and delamination tests on laminated red oak beams made of preservative-treated lumber with resorcinol and phenol-resorcinol glues

<table>
<thead>
<tr>
<th>Preservative</th>
<th>Average: Curing retention time: Specific gravity: of wood 2</th>
<th>Block-shear values 3: Control: Treated beams: Control: Treated beams:</th>
<th>Delamination 4: P.s.i.-Percent: P.s.i.-Percent: Percent: Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tanalith</td>
<td>0.70 : 9 : 150 : 0.68 : 0.67 : 2,636 - 80 : 2,584 - 87 : 0.1 : 0.3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Celcure</td>
<td>2.31 : 8 : 170 : 0.68 : 0.67 : 2,370 - 77 : 2,101 - 83 : 0.0 : 3.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Greensalt</td>
<td>2.10 : 9 : 150 : 0.68 : 0.68 : 2,636 - 80 : 2,523 - 78 : 0.1 : 3.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pentachlorophenol</td>
<td>11.0 : 8 : 170 : 0.67 : 0.68 : 2,275 - 80 : 2,302 - 77 : 0.0 : 1.8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Copper naphthenate</td>
<td>9.8 : 8 : 170 : 0.67 : 0.63 : 2,275 - 80 : 2,184 - 84 : 0.0 : 2.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Copperized chromated: zinc chloride</td>
<td>2.45 : 9 : 150 : 0.63 : 0.64 : 2,479 - 83 : 2,214 - 86 : 0.2 : 1.9</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Boliden salt</td>
<td>1.16 : 9 : 150 : 0.63 : 0.63 : 2,479 - 83 : 2,225 - 83 : 0.2 : 3.5</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

1 Temperature at the innermost glue line.
2 Specific gravity of untreated wood based on oven-dry weight and volume.
3 Made according to ASTM 905-49. The first figure in each column is shear strength; the second is percentage estimated wood failure. Each value is the average of results obtained with four different glues. Two 4-1/2- by 6- by 14-inch beams of treated and one of untreated material were made with each glue at the curing condition shown.
4 Made according to ASTM D1101-53.
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