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DURABILITY OF PAPREG-TO-PAPREG AND

PAPREG-TO-BIRCH GLUED JOINTS¹

By

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Introduction

A series of gluing studies on papreg was made and reported in Forest Products Laboratory Report No. 1348 (Revised) "The Gluing of Laminated Paper Plastic (Papreg)" in which such factors as the preparation of gluing surfaces, evaluation of glues for the bonding, the range of good gluing conditions, the gluing of papreg to wood, and the nail gluing of thin papreg were investigated. In general, these studies show that, with the proper glues, surface preparation, and gluing conditions, joints that have high initial strength can be obtained between papreg and papreg, and papreg and wood.

It is the purpose of this study to determine the durability of such well-glued joints when aged under controlled conditions of moisture and temperature. Joints between papreg surfaces and between papreg and wood were prepared with five types of glue and were subjected to six exposure conditions. To detect changes in joint quality with age, specimens are to be tested at the ends of various periods up to 5 years. This report presents the results of the tests after periods of exposure up to 1 year.

Experimental Procedure

Papreg

The 1/16-inch-thick papreg used for this study was prepared by pressing together approximately 30 sheets of Mitscherlich paper,² impregnated with an alcohol-soluble, phenol-formaldehyde resin. Stainless steel cauls were used. All sheets were laminated with the fiber direction of the papers parallel, under a pressure of 250 pounds per square inch for 20 minutes at 320° F.

¹This is one of a series of progress reports prepared by the Forest Products Laboratory relating to the use of wood in aircraft. Results here reported are preliminary and may be revised as additional data become available.

²This impregnated paper was manufactured by a commercial paper company following recommendations of the Forest Products Laboratory.

The resulting papreg had a density of 1.3 to 1.4 grams per cubic centimeter and a resin content of 30 to 35 percent.

Wood

Yellow birch veneer (1/16 inch thick), selected for straightness of grain, was used in preparation of the papreg-to-wood joints.

Preparation of Material for Gluing

The birch veneer and papreg were conditioned for at least 1 week at 80° F. and 30 percent relative humidity before being cut into 5- by 12-inch pieces in preparation for gluing. The fiber direction was parallel to the 5-inch dimension of the pieces.

Number 1/2 garnet sandpaper was used on a portable belt sander to remove 0.002 to 0.003 inch from the face of the papreg panels before they were bonded together or bonded to the wood.

Type of Specimen

Panels 5 by 12 inches were prepared by gluing together two of the 1/16-inch papreg pieces or one of the papreg pieces and a piece of the yellow birch veneer.

From the completed panel, 10 lap shear specimens of the type shown in figure 1 were cut.

Glues

The following glues were used in preparing the joints:

- a cold-setting, urea resin,
- an alkaline, low-temperature phenolic resin,
- an acid, low-temperature phenolic resin,
- a casein glue plus a preservative,
- a low-temperature resorcinol resin.³

Gluing Conditions

Glue spread.---A double spread was used to provide a total of 15 to 20 grams of wet glue per square foot of glue joint.

³The resorcinol glue employed in this study was not typical of current resorcinol-formaldehyde glues in that it was not adequately water-resistant when cured at 75° F. and was shortly replaced thereafter by an improved formulation.

Assembly time.---A closed assembly time of 10 to 20 minutes was used.

Pressure.---A pressure of 150 pounds per square inch was employed for all gluing.

Curing time and temperature.---The joints glued with the urea-resin, casein, and low-temperature resorcinol glues were pressed for 18 hours at 75° F. The joints prepared with the two low-temperature phenolic resin glues were pressed for 18 hours at 160° F.

Conditioning.---All joints were conditioned at least 1 week at 30 percent relative humidity and 80° F. before being cut into specimens and placed in the exposure racks.

Distribution of Specimens

From the 48 panels or 480 specimens prepared with each glue, 60 specimens were selected as controls and the remaining 420 specimens were divided equally among six conditions of exposure. Ten specimens were tested from each condition after aging for 1, 3, and 6 months and 1 year. Ten specimens will be tested after 2, 4, and 5 years.

Distribution was made so that not more than two specimens per panel were included in any of the six exposure conditions and not more than two specimens per panel were tested as controls nor will be at any testing interval.

Exposure

The six conditions of exposure are:

1. Continuous exposure to 80° F. and 30 percent relative humidity; test dry.
2. Continuous soaking at room temperature; test wet.
3. Continuous exposure to 80° F. and 97 percent relative humidity; test on removal from the 97 percent relative humidity room.
4. A repeating cycle of 2 weeks at 80° F. and 97 percent relative humidity and 2 weeks at 80° F. and 30 percent relative humidity; test on removal from the 30 percent relative humidity room.
5. Continuous exposure to 200° F. and 20 percent relative humidity; test after cooling to room temperature.
6. A repeating cycle of 1 day at 200° F. and 20 percent relative humidity and 1 day at -25° F.; test at room temperature. When the schedule of changes falls on Sunday, the specimens are allowed to remain 2 days at the previous condition.

Testing

The specimens were tested in shear in a plywood-joint testing machine which loaded the specimen at a rate of 700 to 800 pounds per minute.

Summary of Results of First Year

The joint shear strength and the estimated percentage of failures in wood and papreg specimens tested after exposure to the various conditions during the first year are given in tables 1 and 2.

The control specimens all had high joint strengths and the average papreg and birch failures were over 90 percent. The joints glued with the resorcinol resin had the lowest average control strengths of 531 and 575 pounds per square inch for papreg-to-wood and papreg-to-papreg joints, respectively, as compared to minimum control values of 636 and 674 pounds per square inch for the joints bonded with the other glues, but showed the most significant change (an increase) in strength of any of the glued joints when aged at 80° F. and 30 percent relative humidity. The resorcinol glue used was one of the early glues of this type to be developed and it is possible that the curing conditions used did not produce a fully cured glue joint. There was some decrease in papreg-and-birch failures in urea-resin glued papreg-to-birch joints after aging for 3 months to 1 year at 80° F. and 30 percent relative humidity.

There was a pronounced loss of strength in the resorcinol- and casein-glued joints after 1 month of soaking. The joints glued with the acid, low-temperature phenol had somewhat lower strength than the joints glued with the alkaline, low-temperature phenol or the cold-setting urea-resin glues.

After soaking for 1 year, the joints glued with the two low-temperature phenolic resins and the cold-setting urea-resin glues had average papreg and birch failures of over 90 percent. The casein-glued joints had less than 15 percent papreg or birch failure after this exposure, and the resorcinol-glued joints had average papreg failures of 31 percent in the papreg-to-papreg joints exposed for 1 year.

Continuous exposure to 97 percent relative humidity at 80° F. caused general loss of joint strength but much of this loss can be attributed to the higher moisture content at test. The joints glued with the casein and resorcinol glues may have lost more than can thus be accounted for, but the results of further tests are required to make this certain.

The alternating cycle of 97 percent and 30 percent relative humidity at 80° F. has not, in 1 year, caused any significant change in either the strength or type of failure of any of the glued joints with the possible exception of the urea glue between papreg and birch.

The joints glued with the cold-setting urea-resin failed very rapidly when exposed continuously to 200° F. In 1 month, the papreg-to-papreg and papreg-to-birch joints failed over 95 percent in the glue, and at the end of 1 year the joint strength was one-third or less that of the original.

Papreg-to-papreg joints glued with the four other glues continued to maintain over 75 percent papreg failure at the end of 1 year exposure to 200° F. but the resorcinol and acid-low-temperature-phenolic-resin glued joints lost strength. The loss of strength in the acid-phenolic-resin glued specimens was probably due to the effect of heat and acid on the papreg, but this explanation would not appear to hold for resorcinol-glued joints since the pH of the resorcinol glue is not low.

The papreg-to-birch joints glued with the phenolic-resin and resorcinol glues developed over 90 percent wood or papreg failure after 1 year of exposure to 200° F., but the joint strengths were greatly reduced. Many of the joint specimens failed in the birch ply in tension. The casein-glued joints decreased somewhat in percentage of wood or papreg failure but the joint strengths were higher than those obtained with any of the other glues. The joints made with urea glue failed conspicuously at 200° F.

The cycle of 200° F. and -25° F. repeated for 1 year reduced the strength of the papreg-to-birch joints, that were urea-resin glued, to the same level as the continuous 200° F. exposure for the same period, but the rate of deterioration of the glue joint in the papreg-to-papreg specimens was slower than when exposed continuously.

After 1 year of exposure to the high- and low-temperature cycle, the papreg-to-papreg and papreg-to-birch joints glued with the three other resin glues failed over 90 percent in the wood or papreg, with low strength only in the joints glued with the acid, low-temperature phenolic resin.

The papreg-to-papreg specimens glued with casein withstood the high-low temperature cycle fairly well with papreg failure 90 percent or more, and with relatively high joint strength, but the papreg-to-birch joints had only 35 percent papreg or wood failure after 1 year of exposure and loss of strength was more pronounced.

Summary

All of the glues (casein, acid and alkaline low-temperature phenolic resin, cold-setting urea resin, and resorcinol resin) produced well-glued original joints of papreg-to-papreg, and papreg-to-wood, but there was some evidence that the resorcinol-glued joints were somewhat undercured.

The strength of joints glued with the acid low-temperature phenolic resin were much lower after certain exposures, but consistently showed high percentages of wood or papreg failure.

The joints glued with the alkaline low-temperature phenolic resin retained a high percentage of joint strength and had wood or papreg failures of 96 percent or better under all conditions.

The preserved-casein glued joints showed only a small loss of strength at continuous exposure to 97 percent relative humidity, and practically none from the alternating 97 and 30 percent relative-humidity cycle. Continuous soaking, however, greatly weakened the strength of these joints. At the high temperature exposures, the casein-glued joints maintained relatively high strengths, but the percentage of wood or papreg failure in the papreg-to-wood joints was considerably less than the percentage of papreg failure in the papreg-to-papreg joints.

The cold-setting, urea-resin glued joints had good durability at all conditions except where high temperatures were involved. Exposures involving temperatures of 200° F. quickly reduced the strength of these joints.

The resorcinol-glued joints, as prepared for this study, were probably undercured as they did not have the durability under continuous soaking that might be expected for well-cured joints. Under the other conditions of exposure, these glued joints had slightly higher strengths than those glued with the acid low-temperature phenolic resin.

Table 1.--Humidity test results on paper-to-resin joints glued with five glues and exposed to various conditions for periods up to 1 year.

| Glue | Aging period | Exposure condition | | | | | | | | | | | |
|---|--------------|---|---------------------------|-----------------------------------|---------------------------|-----------------------------------|---------------------------|---|---------------------------|-----------------------------------|---|-----------------------------------|---------------------------|
| | | 40° F. and 30 percent relative humidity | | | Water soaking | | | 60° F. and 97 percent relative humidity | | | 60° F. and 97 percent, 200° F. and 20 percent relative humidity | | |
| | | Shear strength lb. per sq. in. | Papreg failure Percent | Shear strength lb. per sq. in. | Papreg failure Percent | Shear strength lb. per sq. in. | Papreg failure Percent | Shear strength lb. per sq. in. | Papreg failure Percent | Shear strength lb. per sq. in. | Papreg failure Percent | Shear strength lb. per sq. in. | Papreg failure Percent |
| An acid, low-temperature phenolic resin | Control | 674 | 100 | 355 | 100 | 659 | 100 | 519 | 100 | 523 | 100 | 523 | 100 |
| | 1 month | 667 | 98 | 362 | 100 | 620 | 97 | 508 | 95 | 575 | 95 | 575 | 95 |
| | 6 months | 660 | 100 | 361 | 100 | 599 | 100 | 402 | 96 | 474 | 98 | 474 | 98 |
| An alkaline, low-temperature phenolic resin | Control | 763 | 100 | 472 | 100 | 785 | 100 | 789 | 100 | 757 | 100 | 757 | 100 |
| | 1 month | 768 | 100 | 476 | 100 | 756 | 100 | 690 | 100 | 754 | 100 | 754 | 100 |
| | 6 months | 784 | 100 | 466 | 100 | 736 | 100 | 739 | 98 | 724 | 100 | 724 | 100 |
| A casein glue plus a preservative | Control | 713 | 100 | 447 | 100 | 690 | 99 | 545 | 100 | 656 | 100 | 656 | 100 |
| | 1 month | 683 | 100 | 279 | 25 | 648 | 100 | 644 | 100 | 644 | 100 | 644 | 100 |
| | 6 months | 708 | 100 | 250 | 9 | 644 | 54 | 658 | 97 | 601 | 96 | 601 | 96 |
| A cold-setting, urea-resin | Control | 747 | 100 | 252 | 10 | 619 | 82 | 636 | 100 | 662 | 100 | 662 | 100 |
| | 1 month | 795 | 100 | 247 | 12 | 630 | 49 | 550 | 100 | 517 | 99 | 517 | 99 |
| | 6 months | 710 | 86 | 474 | 100 | 938 | 100 | 363 | 0 | 671 | 78 | 671 | 78 |
| A low-temperature resorcinol resin | Control | 860 | 92 | 480 | 100 | 771 | 100 | 136 | 0 | 442 | 11 | 442 | 7 |
| | 1 month | 575 | 97 | 362 | 76 | 656 | 70 | 658 | 92 | 643 | 89 | 643 | 89 |
| | 6 months | 558 | 84 | 257 | 48 | 477 | 79 | 536 | 89 | 536 | 85 | 536 | 85 |
| A low-temperature resorcinol resin | Control | 248 | 87 | 250 | 51 | 494 | 83 | 632 | 86 | 509 | 79 | 509 | 79 |
| | 1 month | 248 | 87 | 250 | 51 | 494 | 83 | 632 | 86 | 509 | 79 | 509 | 79 |
| | 6 months | 248 | 87 | 250 | 51 | 494 | 83 | 632 | 86 | 509 | 79 | 509 | 79 |

Each result for the control tests is the average of 60 specimens, and each result for the exposure tests is the average of 10 specimens.

Table 2.--Durability test results on osage-to-birch joints glued with five glues and exposed to various conditions for periods up to 1 year.

| Glue | Aging period | Exposure condition | | | | | | | | | |
|---|--------------|---|---------------------------------|---|---|--|--|-----|-----|-----|-----|
| | | 80° F. and 50 percent relative humidity | Water soaking | 80° F. and 97 percent relative humidity | 60° F. and 97 percent relative humidity; 80° F. and 50 percent relative humidity (2-week cycle) | 200° F. and 20 percent relative humidity | 200° F. and 20 percent relative humidity; to -25° F. (1-day cycle) | | | | |
| | | Shear strength | Shear strength and wood failure | Shear strength and wood failure | Shear strength and wood failure | Shear strength and wood failure | Shear strength and wood failure | | | | |
| | | Lb. per sq. in. | Percent | Lb. per sq. in. | Percent | Lb. per sq. in. | Percent | | | | |
| An acid, low-temperature phenolic resin | Control | 642 | 98 | 377 | 100 | 653 | 100 | 436 | 100 | 459 | 97 |
| | 1 month | 622 | 100 | 377 | 100 | 623 | 95 | 348 | 99 | 489 | 100 |
| | 3 months | 612 | 94 | 353 | 100 | 619 | 100 | 374 | 100 | 407 | 100 |
| | 6 months | 614 | 94 | 361 | 100 | 601 | 93 | 201 | 100 | 315 | 100 |
| An alkaline, low-temperature phenolic resin | Control | 686 | 98 | 453 | 100 | 728 | 96 | 700 | 100 | 676 | 98 |
| | 1 month | 719 | 108 | 424 | 100 | 705 | 100 | 499 | 100 | 653 | 100 |
| | 3 months | 675 | 100 | 427 | 99 | 704 | 98 | 526 | 100 | 612 | 100 |
| | 1 year | 675 | 98 | 427 | 99 | 704 | 100 | 277 | 100 | 538 | 100 |
| A casein glue plus a preservative | Control | 636 | 94 | 277 | 9 | 699 | 89 | 655 | 86 | 588 | 82 |
| | 1 month | 608 | 89 | 286 | 10 | 628 | 76 | 581 | 68 | 524 | 73 |
| | 3 months | 611 | 94 | 327 | 11 | 715 | 93 | 499 | 93 | 560 | 78 |
| | 6 months | 696 | 97 | 296 | 11 | 625 | 91 | 418 | 65 | 425 | 75 |
| A cold-setting, urea-resin | Control | 729 | 95 | 442 | 100 | 758 | 91 | 420 | 4 | 613 | 16 |
| | 1 month | 700 | 88 | 447 | 100 | 704 | 87 | 297 | 0 | 534 | 5 |
| | 3 months | 710 | 73 | 401 | 100 | 687 | 83 | 144 | 0 | 294 | 3 |
| | 6 months | 618 | 74 | 431 | 97 | 680 | 51 | 29 | 0 | 20 | 0 |
| A low-temperature resorcinol resin | Control | 531 | 91 | 282 | 84 | 657 | 79 | 672 | 94 | 694 | 94 |
| | 1 month | 627 | 88 | 230 | 60 | 615 | 93 | 570 | 98 | 647 | 100 |
| | 3 months | 570 | 95 | 251 | 80 | 619 | 91 | 425 | 97 | 564 | 95 |
| | 6 months | 602 | 89 | 277 | 85 | 711 | 91 | 309 | 97 | 557 | 98 |

Each result for the control tests is the average of 60 specimens, and each result for the exposure tests is the average of 10 specimens.

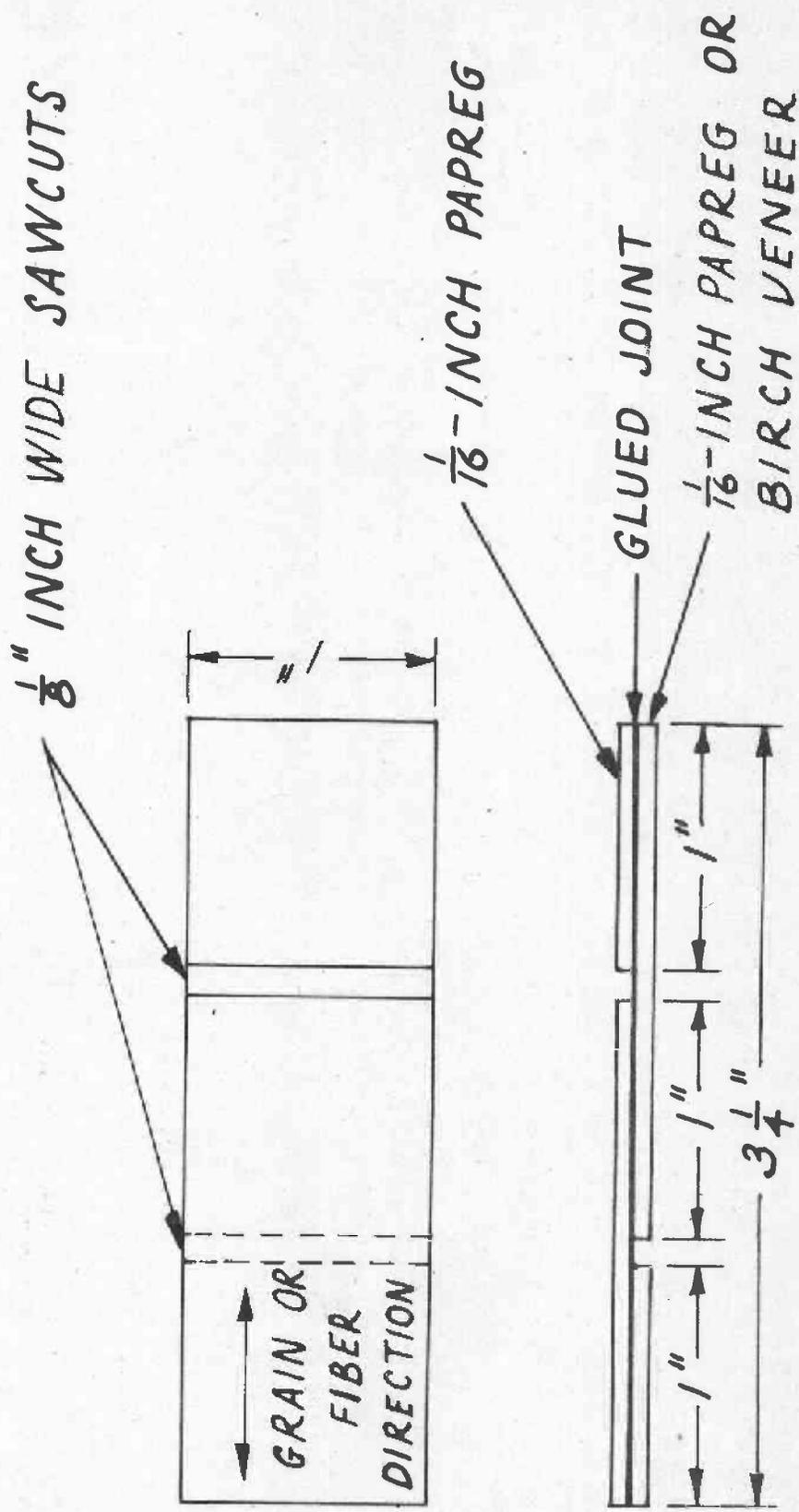


Figure 1.--Glued-joint test specimen of papreg-to-papreg or papreg-to-wood.