Testing Casein Glue

Casein glue is tested in accordance with current Army, Navy, or Federal specifications. The current specification numbers are as follows: Federal C-G-456, Navy 5263b, and Army 3-152-A. All three specifications are similar and all require the making of two types of joint tests, the block shear test and the plywood shear test, and observing certain characteristics of the glue solution. Following is a summary of the requirements of the current Army and Navy specifications:

1. Form.—The glue shall be supplied in the form of a dry powder, ready to be mixed with water.

2. Working life.—At least 5 hours at 65° F.

3. Set.—After the expiration of the working period the glue shall set to a stiff jelly.

4. Block shear test.—Average at least 2800 pounds per square inch.

5. Plywood shear test (dry).—Average at least 340 pounds per square inch.

6. Plywood shear test (wet).—Average at least 140 pounds per square inch.

Block Shear Tests

The following procedure is used in making block shear tests:

1. Use sugar maple selected to obtain material of a specific gravity of at least 0.652, of straight grain and free from defects. Condition to a moisture content of about 7 percent.

2. Cut the material into pieces about 1 by 2.5 by 12 inches or of such other width and length as to provide at least four specimens of the dimensions shown in figure 1. Surface the pieces smoothly to a uniform thickness and glue promptly after surfacing.

1 This mimeograph is one of a series of progress reports issued by the Forest Products Laboratory to aid the Nation’s defense effort. Results here reported may be revised as additional data become available.

2 Based on oven-dry weight and volume.

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3. Glue at least two joints for each test.

4. Follow the manufacturer's directions carefully in mixing the glue. Weigh the component parts. Do the gluing during the second hour after the mixing has been completed.

5. Spread the glue evenly on one of the two pieces at the rate of about 35 grams of wet glue per square foot of glue joint and apply pressure uniformly to the joint when the glue is at the proper consistency. (See directions for gluing hardwoods given in Forest Products Laboratory mimeograph 1340 for further details.) The quantity of the glue spread can be determined by weighing the pieces immediately before and after spreading.

6. Leave the test blocks under pressure not less than 3 hours and condition them for 6 additional days at room temperature before testing.

7. Cut the glued blocks into specimens of the form and dimensions shown in figure 1 and test on a Universal testing machine equipped with a shearing tool illustrated in figure 1. Apply the load to the specimens at a rate not greater than 0.0125 inch per minute.

8. Record for each specimen tested the breaking load and the approximate percentage of wood failure occurring over the glue-line area. Compute the breaking load in terms of pounds per square inch.

9. Reject the glue if the average breaking strength of the joints is less than 2800 pounds per square inch. Whenever a specimen fails at a load of less than 2800 pounds per square inch and the failure occurs 50 percent or more in the wood, the specimen shall be disregarded in computing the average. When the average is less than 2800 pounds per square inch but the breaking strength of one or more specimens is 2800 pounds per square inch or higher and the variation among individual specimens is 10 percent or more (based on the strongest joint), the test shall be made again.

Plywood Shear Tests

The following procedure is used in making plywood shear tests:

1. Glue at least 4 three-ply panels with the grain of the face plies at right angles to that of the core. Use one-sixteenth inch yellow birch veneer selected for firmness, straightness of grain, and freedom from defects.
Fig. 1. Block shear joint test: (1) Test specimen and shearing tool; (2) Testing machine.
Fig. 2. Plywood joint test; (1) Test specimens and grips; (2) testing machine.
2. Each panel should be of a size sufficient to produce at least 10 specimens of the form and dimensions shown in figure 2. A panel measuring 4 inches with the grain and 12 inches across the grain of the faces is a convenient size for cutting the required number of specimens.

3. Condition the veneer to a moisture content of about 7 percent.

4. Follow the manufacturer's directions carefully in mixing the glue.

5. Glue one-half the panels at the end of the first hour after mixing the glue and the other half at the end of the fifth hour after mixing.

6. Glue the plywood under carefully controlled conditions (see detailed directions in Forest Products Laboratory mimeograph 1340).

7. Leave the panels under pressure for at least 3 hours and then condition them for at least 3 additional days at room temperature before testing.

8. From each panel cut 10 specimens of the form and dimensions shown as specimen A in figure 2. Number the specimens from each panel successively from 1 to 10.

9. Test in the dry condition the odd-numbered specimens from each panel in a cement briquette testing machine equipped with special grips as shown in figure 2. Apply the load to the specimens at a rate between 600 and 1000 pounds per minute.

10. Soak the even-numbered specimens in water at room temperature for 48 hours and then test them as soon as removed from the water in the same manner as described in subparagraph 9 above.

11. Record for each specimen tested the breaking load and the approximate percentage of wood failure occurring in the test.

12. Reject the glue if the average breaking strength, either wet or dry, is less than the specified values (at present 340 pounds per square inch dry and 140 pounds per square inch wet). Whenever a specimen fails at a load less than the specified value and the failure occurs 50 percent or more in the wood, the specimen shall be disregarded in computing the average.\footnote{Cross grain through the face plies or severe checks in core plies result in low test values. Veneer showing such characteristics should not be used in the test.}
Inspection of Cold-setting, Synthetic Resin Glues

Cold-setting, synthetic resin glues should be tested according to Navy Aeronautical Specification No. G-29 (April 4, 1941). Following is a summary of the requirements of this specification:

1. **Form.**—The glue shall be supplied in the form of a dry powder ready to be mixed with water. It shall consist of synthetic resin and not more than 20 percent on the dry resin basis of inert, insoluble filler.

2. **Catalyst.**—The catalyst or hardener may be incorporated in the glue or supplied separately.

3. **Filler.**—The insoluble organic filler shall be incapable of dissolving or being swelled appreciably in water.

4. **Stability.**—The powdered glue shall be stable for 1 year when stored in a dry place at ordinary room temperatures.

5. **Working life.**—The glue shall have a working life of not less than 1 hour nor more than 8 hours when mixed for use at 70° F. It is permissible to adjust the working life by use of suitable hardeners.

6. **Block shear.**—Average at least 2,800 pounds per square inch.

7. **Plywood shear test (dry).**—Average at least 340 pounds per square inch.

8. **Plywood shear test (wet).**—Average at least 280 pounds per square inch.

The amount and type of filler cannot be readily determined in the field by approximate methods. To determine this point an analysis would be necessary. The working life may be determined in an approximate way by observation of the glue solution when it is kept at approximately 70° F. For accurate determination a viscometer is required in which the temperature of the glue can be maintained at 70° F. plus or minus 1° F. and from which the readings may be converted into poises. The glue shall be considered to have reached the end of the working life when its viscosity reaches 600 poises.

Block shear tests.---The procedure for making the block shear tests closely resembles that described for casein glues. The gluing pressure, however, is specified as from 150 to 300 pounds per square inch. The test blocks shall be held under pressure for 8 to 24 hours at a temperature of from 75 to 85° F. and allowed to stand for 6 additional days at this temperature. The temperature of the gluing room...
Fig. 3. Dough type glue mixer satisfactory for casein glue. Equipped with two sizes of bowls, will mix batches of 2 to 20 pounds of wet glue satisfactorily. Has three speeds but two are ample. Also made in larger sizes.

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and wood should not be below 70° F. The amount of glue spread should be in the order of 20 to 25 grams per square foot instead of 35 grams per square foot as prescribed for casein glues. The remainder of the test procedure follows precisely that described for casein glues.

**Plywood shear tests.**—Five, three-ply, test panels shall be glued midway of the working life as specified by the manufacturer. The amount of glue spread should be between 20 and 25 grams per square foot. The panels shall be placed under pressure within 5 to 10 minutes after spreading and the pressure shall be between 150 and 300 pounds per square inch. Pressure shall be maintained from 8 to 24 hours at 75 to 85° F. and the panels shall be allowed to stand an additional 6 days at a temperature from 75 to 85° F. before testing. Other details of testing procedure are the same as those described for casein glue.

If any of the specimens of glue selected to represent a particular lot fails to meet the requirements of this specification, the remainder of the sample shall be tested. If the sample fails on retest, the entire lot it represents shall be rejected.

**Inspection of Hot-setting, Synthetic Resin Glues**

One of the large uses of hot-setting resin glues in aircraft is in the production of flat plywood that is later used in the fabrication of the plane. When so used, the inspector is not concerned directly with the testing of the glue. The plywood is inspected in the finished form according to the specification governing aircraft plywood of the most highly water-resistant grade.

Synthetic resin glues of both the thermosetting and thermoplastic types are frequently used in the production of molded plywood parts for aircraft. In such cases, inspections are usually made on the finished product and no inspection or tests of the glue itself are carried out by the inspector.

**Inspection of Blood Albumin Glues**

The use of blood albumin glues in aircraft is limited, particularly since the development of synthetic-resin glues, and, when used, they are confined to plywood that is manufactured in commercial plants and subjected to inspection in the finished form. Such inspection does not require tests be made on the glue itself.
Before the use of plywood is permitted, it is inspected and tested in accordance with current Army and Navy specifications (the present specification applicable is An-NN-P-511 Amendment 1, Oct. 1, 1940: Army-Navy Aeronautical Specification for Aircraft Plywood) or has been correctly certified by an authorized Government agency.

The current specification provides for "Standard Plywood" (type I) and "Waterproof Plywood" (type II).

For type I (Standard Plywood) the method of test and testing equipment are the same as those required in the plywood test for casein glue, described above. Not less than one sample from each of four separate panels shall be selected from each lot. Each 8-hour run for each press shall be represented by at least one sample. Each sample shall be of a size sufficient to make 15 to 25 specimens of the size and shape illustrated in figure 2. Five specimens from each sample shall be tested dry and five immediately after soaking in water at room temperature for 48 hours. In the case of panels consisting of more than three plies, the test specimens shall be stripped of all except three, selected from any section of the panel. Whenever the face plies are thicker than one-twentieth inch, the specimens shall be made like Specimen A, figure 2, but when the faces are one-twentieth inch or thinner the specimens shall be cut like Specimen B, figure 2. When Specimen B, with a one-half square inch shearing area is used, the test value shall be reduced by 10 percent in computing the average. Any specimens in which the failure occurs 50 percent or more in the wood, at a load less than the specified minimum, shall be disregarded in computing the average. If the average values obtained with Specimen A, or with Specimen B reduced by 10 percent, are as specified (300 pounds per square inch at present) or above when tested dry and as specified (160 pounds per square inch at present) or above when tested wet after soaking 48 hours, the plywood may be accepted. In case the plywood meets one of the tests but fails to meet the other, the test in which it fails shall be repeated on four or more new samples from four or more panels not previously tested. If the average of the total number of samples, including the original and retest samples, fails to meet the requirements, the lot represented shall be rejected.

Type II (Waterproof Plywood) shall be glued with synthetic-resin glue applied by hot pressing. The method of sampling, of preparing the specimens, of making the shear tests dry and after immersion in water are the same as those described for type I plywood. The minimum average dry test value is (at present) 300 pounds per square inch just as it is for type I plywood but the minimum average wet test value is 250 pounds per square inch with the further provision that no individual specimen shall show a value less than 200 pounds per square inch. In addition, the following test shall be carried out to insure a joint highly resistant to severe service conditions.

4This test serves the same purpose as the so-called fungus test described in AN-NN-P-511.
Five specimens of the type shown as Specimen A, figure 2, shall be cut from each sample. If the number of plies exceeds three, the cuts shall be made so as to test any two of the joints, but the additional plies need not be stripped except as demanded by the limitations of the width of the retaining jaws on the testing machine. The specimens shall be submerged in water at room temperatures for a period of 48 hours and dried for 8 hours at a temperature of 145 ± 5°F., and this followed by two additional cycles of soaking for 16 hours and drying for 8 hours under the conditions described above. The samples shall again be soaked for a period of 16 hours and tested while wet. Test specimens must show no delamination during the soaking and drying cycles and must show not less than 30 percent minimum and 60 percent average wood failure when tested. No definite strength value is required.

Mixing Casein Glues

The manufacturer's directions should be followed in the mixing of prepared casein glues, unless some change is definitely known to give better results. In any case the directions should be definite and followed carefully. For most prepared glues a ratio of 1 part of dry glue to 2 parts of water (by weight) gives a proper consistency for ordinary joints. However, the glue-water proportion must be varied somewhat for the different glues, kinds of woods, and types of joints in order to get the best results (see Forest Products Laboratory mimeograph 1340).

In mixing prepared casein glues the water should first be placed in the bowl of the mixer and the glue sprinkled or sifted in slowly with the paddle in rapid motion (100 to 120 revolutions per minute). After the dry glue has all been added, the motion of the paddle should be slowed down to 50 to 60 revolutions per minute and the stirring continued until all particles of casein are dissolved (as determined by examining a small smear on the palm of the hand) and a smooth mixture of even consistency results (usually requiring from 20 to 30 minutes). With certain glues the mixture must be allowed to stand a specified length of time after the preliminary mixing, but such special cases are covered in the manufacturer's directions. Too rapid stirring beats an excessive amount of air into the mixture, which is bad in any joint, but particularly bad where high pressures are not applied to the glued joints during setting. In case a batch contains lumps at the end of the mixing, it should be rejected. Attempts to beat lumps out are inadvisable, because they will usually produce a foamy glue. The dough mixer shown in figure 3 has been found to be one of the best types available for casein glue. Casein glues in which the raw materials are combined at the time of mixing (called "wet-mix glues") require even more careful observance of directions in mixing than do prepared glues.
Mixing Synthetic-resin Glues

The mixing directions for synthetic-resin glues supplied by the manufacturer should be followed carefully. The quantity of the different components should be determined by weight, particularly so because some of the components may be added in comparatively small quantities. The resin glues in film form are, of course, ready for use when received; many of the mixtures are supplied in liquid form to which only the "catalyst" or "hardener" need be added; others are supplied as dry powders to be mixed with water and "hardeners." Mixers of the type illustrated in figure 3 are suitable but cleanliness is essential. All traces of alkali (such as remnants of casein glue from previous mixtures) should be removed, because even minor amounts of alkaline material will seriously affect the working properties of the synthetic-resin glues. In general, the use of fillers and extenders in mixing aircraft glues should be discouraged. Some cold-setting, urea resin glues in powder form contain fillers when received. To these no additional filler should be added. Other cold-setting, urea resin glues appear to give slightly better joints when small amounts of fillers are added. When used, the filler should be a material that does not dissolve in water and is not appreciably softened by water. The amount should not exceed 20 percent (by weight) based on the weight of the dry resin.

The cold-setting, resin glues set by chemical action and this reaction is accelerated by an increase in temperature and retarded by a decrease in temperature. When the temperature of the glue room increases, as in the hot days of summer, the working life of some of the cold-setting resin glues may become inconveniently short. In such cases, it may be advisable to provide a water bath for the glue container in which cool water may be circulated and the temperature of the glue solution maintained somewhere between 70 and 80° F. Since a decrease in temperature retards the setting action, the time in the clamps should be increased as the temperature drops. It is good practice to avoid using cold-setting resin glues at temperatures, either of the room or of the wood, below 70° F. Below 65° F. unsatisfactory results may be expected.

Cleaning Glue Receptacles

Mixers, spreaders, workmen's brushes, glue pots or cups, and all other glue receptacles that are not discarded should be cleaned thoroughly at least once during each working day to prevent deterioration of the glue and the inclusion of old glue in freshly prepared mixtures. This rule applies to all glues. Paraffin-coated paper cups may sometimes be used to advantage by the workmen and discarded at the end of the day, or when a new batch of glue is prepared.
When synthetic resin glues are used, added precautions in cleaning are necessary. In addition to insuring the absence of alkali, mentioned above, all spreaders and mixers must be cleaned before the end of the working life. If the glue sets on the spreader (or in the mixing containers) it will be extremely difficult to remove. If the glue sets on the rubber covered rolls of the glue spreader, the roll may be seriously damaged in removing the glue and replacement may be necessary.