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LIGNIN-FILLED, LAMINATED-PAPER PLASTICS

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In Cooperation with the University of Wisconsin

(In cooperation with the
Canadian Department of Munitions and Supply,
the Howard Smith Paper Mills, Limited, Cornwall,
and the Forest Products Laboratories, Ottawa)

Introduction

The increasing demand for plastic materials of all kinds has resulted in an increasing scarcity of suitable bonding agents, such as phenolic and other types of resins. Lignin, when prepared in suitable form, offers possibilities as a bonding agent because of its ability to flow when subjected to heat and pressure. The potentially abundant supply of lignin gives the development of its plastics a particular value at this time.

A new approach for the utilization of lignin in the production of a strong laminated paper plastic was indicated by the results of preliminary work conducted by the Howard Smith Paper Mills, Ltd. of Cornwall, Canada. At the instigation of the Canadian Department of Munitions and Supply, a

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²This mimeograph is one of a series of progress reports prepared by the Forest Products Laboratory to further the Nation's war effort. Results here reported are preliminary and may be revised as additional data become available.

further investigation was conducted as a cooperative project at the U. S. Forest Products Laboratory.² This investigation was directed primarily towards the establishment, on pilot plant scale, of the most suitable procedure for handling and controlling the various operations involved, incorporation of desirable properties in the laminate, and the evaluation of the product for various war uses.

In the course of this investigation it was found that, with the exception of edge compressive strength a high temperature thermoplastic lignin-filled paper laminate of equal or superior strength to the conventional phenolic laminates can be made.

Of special interest is its exceptionally high resistance to impact, which may be considered as a measure of toughness. Comparison with recently published test data by the Army Air Forces, Materiel Center, on fabric- and paper-base phenolic laminates indicates that the lignin-filled paper laminates have face Izod impact values 3 to 6 times greater than the other types. The edge impact values are approximately equal to those of fabric-base laminates and 3 times greater than those of the conventional paper-base laminates. Both face and edge impact values are 2 to 3 times greater than corresponding values for papreg (a high-strength phenolic laminate under current development at the Forest Products Laboratory) although other strength properties thus far obtained are somewhat less.

Although lignin is thermoplastic it has been found that the loss in tensile strength obtained when lignin-filled laminates are heated to 200° F. is not greater than that for thermosetting phenolic papreg. At low temperatures the tensile strength of the lignin-filled laminate improves slightly over that measured at room temperature. There is some loss in impact strength both at high (200° F.) and at low (subzero) temperatures, but even at these temperatures the strength of the plastic compares favorably with that of other materials of this type even when they are measured under their most favorable temperature conditions.

In addition to the use of lignin as a filler in laminating paper for plastics, it has been found that it can be used as an extender for phenolic resins in impregnating varnishes for paper laminates. This work, currently under investigation at the Forest Products Laboratory, has shown promising results which will form the basis of a separate report.

²Maintained at Madison, Wis., in cooperation with the University of Wisconsin.

Manufacture of Lignin-Filled Paper Laminates

The process for manufacturing lignin-filled paper laminates comprises the following four steps:

(1) Isolation of lignin from the spent liquors of the alkaline pulping processes; (2) incorporation of the lignin in a papermaking furnish; (3) production of a moldable paper; and (4) conversion of the lignin-filled paper into a laminated plastic.

The lignin supplied by Howard Smith Paper Mills, Ltd., was produced from aspen wood soda-pulp black liquor in a pilot plant as described in a separate report.⁴ The investigations conducted at the U. S. Forest Products Laboratory were concerned exclusively with the three remaining steps in the process.

Several schemes for incorporating the lignin with the pulp prior to its manufacture into paper were studied, two of which were reduced to practical and readily controllable procedures. One of these methods consists of mixing the pulp with an aqueous solution of the crude lignin and then precipitating the lignin in a water-insoluble form of suitable particle size and physical characteristics by means of dilute sulfuric acid. A temperature of 203° F. (95° C.) and a pH of 3.0 was suitable for precipitation. The equipment used for this operation was a wood tank with a draft tube and centrally placed impeller to allow thorough mixing and agitation. Following the precipitation, the stock was washed by passing the mixture over a rotary vacuum filter to remove impurities which would cause excessive foaming on the paper machine. The excessive acid was neutralized with lime, to permit the use of a standard iron filter with iron auxiliary equipment.

The second method consists of precipitating the lignin directly from its solution, in the absence of the pulp, but obtaining the lignin in a physical condition similar to that obtained by the first method. By this procedure a finely divided purified lignin in the form of a paste or powder can be produced. In this form, lignin becomes available not only for use as filler in the paper furnish, but also as a coating material for augmenting the lignin content at the surface of the lignin-filled sheet. It is also in suitable form for use as an extender for phenolics in varnishes for impregnated-paper laminates. In most experiments of this type the pulp was added directly to the lignin without separating the lignin from the mother liquor. The resultant mixtures were washed on the rotary filter. In commercial production it should be possible to separate and wash the lignin in the absence of the pulp, thus producing purified lignin for mixing directly with the pulp in the paper mill beater.

⁴Restricted Forest Products Laboratory report, "Recovery of Lignin from Soda Pulp Mill Black Liquor," Apr. 7, 1943. Mimeograph No. 1578.

The laminating paper was made on the Laboratory paper-making equipment. The pulp-lignin furnish prepared by either of the methods described was run to the paper machine in the usual manner. No unusual paper-making problems were encountered except those involving retention of the lignin in the paper. Relatively simple methods gave essentially complete retention of the lignin at all stages of the process. These and methods were selective return of the white water and the use of glue/alum as flocculating agents. The results obtained indicate that the standard commercial Fourdrinier machine, with only minor adjustments, is entirely suitable for the manufacture of this type of paper.

It was demonstrated that lignin-filled papers thus prepared can be laminated between open cauls, using moderate laminating pressures and a temperature (356° F.) normally available at the paper-mill steam plant. It is necessary to cool the panel before removing it from the press. The pressures required for laminating are dependent on the lignin and moisture contents of the sheet.

Influence of Process Variables on the Physical Properties of the Paper Laminate

In order to establish, and where possible enhance, the physical properties that may be obtained in a lignin-filled laminate, six process variables were studied:

(1) Lignin filler content; (2) type of sulfate pulp used; (3) use of lignin as a surface coating on the lignin-filled paper; (4) orientation of paper in lamination; (5) laminating pressure; and (6) the use of special surface sheets to improve the resistance of lignin-filled laminates to water absorption.

The physical properties of a laminate pressed at 500 pounds per square inch and 356° F. using a moldable paper in which 43.6 percent added lignin had been incorporated with an experimentally produced sulfate pulp are given in table 1.2. These data illustrate, in a specific example, the physical properties that have been studied, and the values obtained. Supplementary data on electrical characteristics will be available later. Data for materials covering a wide range of process variables, indicating the effect of these variables on the various physical properties are given in table 2. This table is arranged in three sections:

(1) Panels parallel laminated, tested parallel to fiber direction;
(2) panels parallel laminated, tested perpendicular to fiber direction;
and (3) panels cross laminated.

⁵All tests on laminated plastics reported herein, if made at this Laboratory, were in accordance with Federal Specification for Plastics, Organic; General Specifications (Methods of Tests) L-P-406, Dec. 9, 1942.

Each section is further divided into three groups, one for each of the pulps used:

(1) Commercial bleachable sulfate pulp A obtained from a Wisconsin mill; (2) commercial bleachable sulfate pulp B obtained from a Quebec mill (Howard Smith Paper Mills, Ltd.); and (3) experimental high-strength laminating sulfate pulp made by U. S. Forest Products Laboratory (Dig. 2644 and 2645). In each of these groups the data are arranged in order of increasing lignin content of the laminate.

The principal data from Section 1 of table 2 has been rearranged in simplified form in table 3 to allow a more ready evaluation.

Effect of Lignin Filler Content

Data covering a considerable range of lignin filler content is available only for pulp A. In general, with increasing lignin content, the physical strength properties become somewhat poorer (table 3). In the range covered, 34.0 to 47.0 percent lignin content, the tensile strength of the plastic decreased with increase in lignin content from 24,000 to 19,000 pounds per square inch, the edge compressive strength from 16,000 to 12,000 pounds per square inch, the flexural strength (modulus of rupture) from 28,000 to 22,000 pounds per square inch, and the Forest Products Laboratory toughness value from 38 to 29 inch pounds per inch width. The modulus of elasticity in tension decreased from 2,400,000 to 1,900,000 pounds per square inch and the moduli in compression and flexure showed a similar trend. However, with increasing lignin content, the pressure required for satisfactory lamination decreased, and in general, the resistance to water absorption improved.

Effect of Sulfate Pulp Used

Most of the experiments were based on commercial sulfate pulps available at the time the project was started, and, except for the instance cited later, no attempt had been made to develop in the pulp such properties as might particularly suit them for use in the production of high-strength laminates. Although laminates with only a limited range of lignin content were prepared from pulp B, comparison of test data with that of laminates from pulp A of approximately the same lignin content indicates somewhat better tensile strength, toughness, and moduli of elasticity for pulp B accompanied with a slightly lower edge compressive strength and modulus of rupture (tables 2 and 3).

To determine the degree of improvement which might be expected if a special high-strength type of sulfate pulp previously found suitable for the production of high-strength phenolic paper plastics is employed, an experiment was made using such a pulp prepared from black spruce at the Forest Products Laboratory. The improvement in strength properties obtained in the plastic laminate from this pulp was quite marked, when compared with those of the laminate from the commercial pulp A of similar lignin content (43.2 and 43.6 percent lignin, respectively,

table 3). The laminate from the U. S. Forest Products Laboratory pulp gave values approximately 50 percent higher for tensile strength and toughness, 15 percent higher for modulus of rupture, 25 to 35 percent higher for the moduli of elasticity, together with a slight improvement in resistance to water absorption. However, no significant difference was obtained in the edge compressive strength value.

Inasmuch as strength properties can be improved by using a lower lignin content, it should be possible to prepare laminates of even greater strength from this type of laminating pulp. Therefore, in the commercial development of this process, particular attention should be given to the properties of the pulp used, as this will establish the strength level of the laminate at a given lignin content.

Use of Surface Coating of Lignin on Paper

Examination of the laminating paper, while in process on the paper machine and in the finished sheet, suggested that the lignin-fiber ratio on the surfaces of the sheet was probably lower than the average ratio in the whole sheet. In appearance, there was a definite two-sidedness to the sheet, probably resulting from a loss of part of the lignin originally associated with the fibers on the wire side. This loss might have taken place either during the initial period of formation on the wire, at the suction boxes, or at the felt. It was also observed that there was a slight pick-up of lignin from the top side of the sheet at the press rolls, particularly at the second press, which was made of stonite.

To enrich the lignin content on the surface of the lignin-filled sheet, experiments were made in which the partially dried paper was run through the paper machine size-press in which an aqueous suspension of purified lignin previously precipitated at 203° F. (95° C.) was used for the coating mixture. The lignin, having a particle size in the general range of 5 to 10 microns, was made into a 15 to 20 percent suspension and the pH (initially 4.8) was adjusted with lime to 6.1 for runs 35 and 36 (panel Nos. 2226 and 2229) and 7.12 for run 37 (panel No. 2237). The lignin pick-up under these conditions amounted to 1 to 9 percent. The results obtained were inconclusive, however. Comparison of the data (table 2, section 1) indicates that the laminates made from the coated paper from runs 35 and 36 are approximately 1,000 pounds per square inch higher in strength values than those made from the uncoated portion of run 35 whereas in run 37, with a portion of the paper coated and a portion uncoated, a reversed condition was observed for the tensile strength although other strength properties were not generally affected. The reason for this discrepancy has not been established although it is possible that the pH of the coating mixture, or the lime added, might have played an important role. Inasmuch as the range of improvement in properties that might be obtained from this procedure is not great, it is believed that this problem could be further investigated in commercial production, where sustained operation allows a more satisfactory study of the process variables involved.

Orientation of Paper in Lamination

In the manufacture of laminating paper the fibers tend to assume a higher degree of orientation in one direction (machine direction) and the resultant sheet exhibits anisotropic strength properties. Thus in laying up the paper sheets for lamination, this anisotropic characteristic can be carried through into the final panel by "parallel lamination." Alternatively by "cross lamination," (crossing the direction of alternate sheets) a panel having balanced strength properties can be obtained.

The general relationship of the strength values obtained in parallel and cross laminates is shown in table 4. With parallel-laminated samples, tested at room temperature, the most marked difference is shown in the tensile strength, wherein the crosswise tests average only about 33 percent of the lengthwise tests. The comparison for modulus of rupture is 50 percent, edge compressive strength 90 percent; and the moduli of elasticity between 35 and 56 percent. However, with toughness and Izod, the crosswise strength values may be somewhat higher than those in which the direction of test is lengthwise.

In cross laminated samples, the strength values in either direction are in the range of 90 to 105 percent of the average values obtained from crosswise and lengthwise tests on parallel-laminated samples with the exception of Izod impact strength (flatwise) and the Forest Products Laboratory toughness, which are about 65 percent. The reason for these losses in impact and toughness strengths in cross laminated material cannot be explained at present.

The relationship of strength tests in cross-laminated samples to the lengthwise test in parallel-laminated samples is also shown in table 4.

Laminating Pressure

In general, the pressure required for satisfactory lamination decreases with increasing lignin and moisture contents when the laminating temperature is held constant. Figure 1 (data from table 5) illustrates the relationship between moisture content of the lignin-filled paper at time of lamination and the water absorption of the laminated plastic when the laminating temperature is 356° F. Figure 1, A, with laminating pressure constant (500 pounds per square inch), shows that the moisture content of the laminating paper becomes less critical at higher lignin content. The plastic containing 47 percent lignin has a water absorption value of 6 percent when a laminating paper containing 4.8 percent moisture is used. To obtain a similar water absorption value with the plastic containing 38.8 percent lignin, 7.6 percent moisture in the paper is required. With a paper containing 38.8 percent lignin and having an appreciably lower moisture content, the water absorption value of the plastic is considerably increased.

Figures 1, B and 1, C compare the effect of varying laminating pressures with papers of 38.8 and 43.6 percent lignin content, respectively, using the same ordinates. From these curves it is evident that the moisture content of the laminating paper becomes less critical when higher laminating pressures are used.

If the laminating pressure is lower than is optimum for either the lignin content or the moisture content of the paper, the moisture absorption value of the plastic will not only be high, but the edge compressive strength will be low. (Table 2.) With increased pressure these properties improve, but when the pressure is still higher, certain of the strength properties, notably the tensile strength, decrease appreciably.

The approximate pressures required for laminating lignin-filled papers of various lignin contents are shown in table 6.

As examples of panels laminated in the lower pressure range, reference is made to panels 2203-H and 2237-C shown in table 2. These panels containing 47 and 45 percent lignin, respectively, were molded at 250 pounds per square inch. The strength values are 1,000 to 2,000 pounds per square inch lower than those obtained with the same material laminated at higher pressures. The water absorption values, 5.6 and 6.3 percent, respectively, although higher than those obtained with panels of the same lignin content laminated at higher pressures, are within an acceptable range for many purposes. At the time the various machine-runs were made, it was assumed that a laminate containing 45 percent lignin would be relatively brittle and low in strength properties and that higher lignin contents would be in an unacceptable range. However, the results subsequently obtained, particularly on the laminates containing high-strength fiber, indicate exceptional toughness when compared with other laminated materials. It is assumed that even with considerably higher lignin content, a desirable product can be obtained. No doubt a product of this type could be laminated in a lower pressure range.

Use of Special Surface Sheets to Improve Resistance to Water Absorption of Lignin-filled Laminates

None of the lignin-filled laminates discussed so far have contained resin, other than lignin. A study was made to establish the degree of improvement in resistance to water absorption that might be obtained through the use of phenolic resin applied to the surface sheets in the form of a varnish before lamination. The experiments also included a mixed lignin-phenolic varnish and a 100 percent lignin varnish. The data obtained are shown in table 7. Although the results are of a preliminary nature, a definite improvement in resistance to water absorption is indicated. The use of approximately 25 percent phenolic resin on the surface sheet was sufficient to reduce the water absorption from 5.0 to 2.9 percent. This quantity of phenolic resin is relatively small when based on the weight of the final laminate, being only 1.2 percent on a 1/8-inch panel. The appearance and mar-resistance of the panel are also considerably enhanced by this procedure. Accelerated weathering tests on both normal and surface-sheeted laminates are in progress.

Comparison of Lignin-filled Paper Laminates
with Other Laminated Materials

With the exception of edge compressive strength (14,000 to 15,000 pounds per square inch), the physical properties obtained appear to be equal or superior to the conventional-type paper-base laminates on the market. However, the strength properties so far obtained, with the exception of toughness and impact, are considerably lower than the papreg currently in development at the U. S. Forest Products Laboratory. As shown in table 8, Izod impact values, both flatwise and edgewise, are from two to three times as great as those so far reported for papreg and many times greater than those for the conventional paper laminates. The Izod face impact values are considerably higher than those for fabric base laminates and compreg, and about equal to those for heat-stabilized wood and parallel-laminated birch plywood which has the advantage of lower specific gravity. Parallel-laminated wood has a high edge impact value with the grain but low across the grain. Glass-fiber base phenolic laminates appear to have impact values considerably higher than any reported on the type of material under discussion,

The changes in tensile and Izod impact strengths of the lignin-filled paper laminate were determined for both low and high temperatures. At low temperatures the samples were first conditioned for 24 hours at -68° F. and then were packed in dry ice (-110° F.). The tensile specimens were packed for 8 hours and the Izod specimens were packed for 30 minutes to 1 hour. The tensile specimens were repacked in dry ice for test, the machine grips being cooled in an outer chamber maintained at -20° to -60° F. The impact tests were made on a machine in the open atmosphere, within 20 to 30 seconds after taking the specimens from the cold box. For tests at high temperatures the samples were placed in an oven at the desired temperature for 24 hours prior to testing, then tested on a machine at room temperature.

The results of these tests are shown in table 1. A parallel-laminated sample (2239-A), having a tensile strength of 28,520 pounds per square inch tested flatwise with the grain, at room temperature, was found to have a tensile strength of 19,480 pounds per square inch at 200° F. (2239-Q) a loss of 31.6 percent. Comparing the same material (2239-E) tested across the grain at room temperature and at 158° F. (2239-Q) a decrease of 26 percent in tensile strength was obtained. A cross-laminated sample of the same material (2239-F) dropped in tensile strength from 16,770 pounds per square inch at room temperature to 14,770 pounds per square inch at 158° F. (2239-G) a loss of 12 percent. A cross-laminated sample of papreg tested over the same range of temperatures was found to drop 26 percent in tensile strength between room temperature and 158° F. and 38 percent between room temperature and 200° F. In spite of the fact that lignin is a thermoplastic material, the loss in tensile strength at high temperatures is actually slightly less than that with the thermosetting phenolic.

When another sample (2238-I) of the lignin-filled plastic was tested at room and subzero temperatures (approximately -100° F.) an

increase in tensile strength from 25,600 to 27,100 pounds per square inch or 5.9 percent was obtained.

In general, the Izod impact strength values made on the lignin-filled paper laminates at high and low temperatures were somewhat lower than those made at room temperature (table 1), a characteristic of phenolic laminates as well. For example, a parallel-laminated panel having an Izod impact strength value of 12.82 foot-pounds per inch of notch when tested with the grain at room temperature gave a value of 10.47 foot-pounds at 200° F., a loss of 18.3 percent, and 9.76 foot-pounds at -68° F., a loss of 23.9 percent. In spite of these strength losses with change in temperature, the values obtained both at high and low temperature are still much higher than those obtained from papreg and conventional laminates tested under the most favorable conditions (room temperature).

Exploratory gluing tests using cold-setting uree high- and low-temperature phenolic-resin glues have indicated that good bonds can be obtained, with failures occurring almost entirely in the lignin-filled paper plastic rather than in the glue line, when the surfaces to be glued are lightly sanded.

In any application requiring large quantities of material having strength properties in the range of conventional plastics, and where high impact strength is of special importance, the lignin-filled paper-base laminates appear to offer excellent possibilities.

Application has been made by the cooperators for United States patents covering the preparation of lignin and its use for incorporation into paper for laminating purposes in the manner described herein. It is expected that the use of such patents will be made available to others by the Secretary of Agriculture on a wartime license basis.

Table 1.—*Physical and mechanical properties of 316Lm-filled, laminated-epoxy platelets from BSA V¹ (Plastic Nov 2017, 2218, and 2270).*

Panel (shape-specific) number	Specific gravity before soaking	Moisture content at time of load- ing action (difference free load)	Flexure strength		Edge compressive strength		Tensile strength		Water absorption - 24 hours		Impact strength (ft-lb)		
			28-day strength at time of load- ing action (difference free load)	28-day strength at time of load- ing action (difference free load)	28-day strength at time of load- ing action (difference free load)	28-day strength at time of load- ing action (difference free load)	28-day strength at time of load- ing action (difference free load)	28-day strength at time of load- ing action (difference free load)	28-day strength at time of load- ing action (difference free load)	28-day strength at time of load- ing action (difference free load)	28-day strength at time of load- ing action (difference free load)	28-day strength at time of load- ing action (difference free load)	
2297-40-135	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-136	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-137	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-138	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-139	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-140	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-141	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-142	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-143	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-144	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-145	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-146	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-147	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-148	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-149	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-150	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-151	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-152	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-153	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-154	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-155	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-156	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-157	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-158	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-159	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-160	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-161	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-162	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-163	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-164	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-165	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-166	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-167	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-168	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-169	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-170	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-171	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-172	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-173	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-174	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-175	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-176	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-177	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-178	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-179	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-180	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-181	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-182	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-183	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-184	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-185	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-186	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-187	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-188	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-189	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-190	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-191	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-192	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-193	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-194	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-195	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-196	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-197	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-198	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-199	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-200	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-201	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-202	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-203	1.39	1977-595 H.H. Soaked at 140°F.	28,530	7,660	2,530	13,660	2,770	2,770	2,770	5.2	0.6	23,145	2,075-01 0.40
2297-40-204	1												

^a Run N - Forest Products Laboratory experimental black spruce millrun pulp, solids 26% and 20% combined. Content of acid lignin = 15.2 percent. Limit-filled pulp from run 2879 was jarred, samples from 25% and 20% combined were jarred, and 50% combined was jarred after 10 minutes at 175°C. Samples were jarred before release of pressure.

Thickness of laminated plastic (in molding pressure direction).

(attached) and to the Director of the Bureau of the Census.

need applied to the edge of the silicon material (perpendicular to the direction of rolling stress).

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[illegible]

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Table 3.—Simplified summary of data on Alumin-filled laminated-wood plastics. Parallel laminated, all tests made in direction of grain.

[illegible]

For complete data, see table 2.

² Commercial bleachable sulfate pulp obtained from a Wisconsin mill.

Country	Year	Value	Unit	Source
Canada	1980	100	1000	Statistics Canada
France	1980	100	1000	INSEE
Germany	1980	100	1000	Statistik
Italy	1980	100	1000	ISTAT
Japan	1980	100	1000	Ministry of Finance
United Kingdom	1980	100	1000	ONS
United States	1980	100	1000	Bureau of Economic Analysis
Quebec	1980	100	1000	Statistics Canada

^a Used values taken from panel 2224-7. 36.6 percent lignin (no similarity data available on 2220-G, 17.0 percent lignin).

Table 4.—Distribution of strength in laminated samples resulting from orientation of paper in lamination. (Values given indicate range obtained in samples tested).

Strength	Parallel laminated	Cross laminated	
	Tested across grain	Percent ¹ of average values when tested with grain in parallel-laminated samples	Percent ¹ of value when tested with grain in parallel-laminated panel
	Percent ¹ of values when tested with grain	and across grain in parallel-laminated samples	
Tensile, Ultimate	31 - 35	89 - 94	59 - 63
Tensile, Modulus of Elasticity	37 - 56	95 - 105	72 - 74
Edgewise Compression, Maximum	85 - 95	101 - 104	95 - 100
Edgewise Compression, Modulus of Elasticity	35 - 52	89 - 105	60 - 75
Flexure, Modulus of Rupture	45 - 54	97 - 104	71 - 79
Flexure, Modulus of Elasticity	41 - 52	92 - 103	68 - 76
F. P. L. Toughness	96 - 125	59 - 71	58 - 75
Izod, Flatwise	77 - 130	65.7	66.4
Izod, Edgewise	91 - 113	91.6	87.4

¹For absolute values, refer to table 2.

Table 5.--Relationship between moisture content of lignin-filled papers of various lignin contents and water absorption of the laminated plastic when molded at various pressures.¹

Panel number	Moisture content of paper on moisture free basis	Laminating pressure	Water absorption 24 hours
	Percent	Lb. per sq in.	Percent
<u>38.8 Percent Lignin Content</u>			
2226-E	6.5	500	8.4
K	6.6	500	7.9
A	6.8	500	7.4
D	7.7	500	5.7
H	8.3	500	5.8
F	6.6	750	6.5
B	6.7	750	6.4
L	6.9	750	6.4
I	8.2	750	5.6
C	6.5	1,500	5.6
G	6.8	1,500	5.7
J	7.8	1,500	5.3
<u>43.6 Percent Lignin Content</u>			
2194-H	6.8	250	29.3
G	9.8	250	5.6
J	5.9	500	7.0
E	6.6	500	5.7
L	6.8	500	5.2
A	9.0	500	5.4
D	6.8	750	5.3
B	8.5	750	4.7
I	5.9	1,500	5.2
F	6.6	1,500	4.8
C	8.5	1,500	4.6
<u>44.9 Percent Lignin Content</u>			
2204-A	5.5	500	6.2
C	6.9	500	5.2
<u>47.0 Percent Lignin Content</u>			
2203-H	6.4	250	5.6
C	4.8	500	6.0
A	6.6	500	5.0

¹Laminating conditions: Temperature, 356° F; time, 10 minutes, panels cooled before release of pressure. Panel nominal thickness one-eighth inch.

²Split.

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Table 7.---Effect of special surface sheets on water absorption of lignin-filled laminates. (Lignin-filled laminating paper coated on one side with resin varnish of indicated composition.
One ply. resin side out, placed on each surface of lay-up).¹

Base sheets										Surface sheets										Laminates									
Panel:Run:	Moisture	Lignin	Paper:Lignin	Resin varnish	Resin	Added	Added	Water	Decrease	No.:	No.:	content on:	No.:	content	before	Lignin	Phenolic	on	resin	phenolic	absorp-:	in water	tion	absorp-	tion	24	hours	to surface	sheet
:	content	content	content	content	content	content	content	content	content	:	:	moisture-	:	moisture-	:	coating:	content:	paper	on	on	:	:	:	:	:	:	:	:	:
:	of paper	of paper	of paper	of paper	of paper	of paper	of paper	of paper	of paper	:	:	free	:	free	:	coating:	content:	paper	on	on	:	:	:	:	:	:	:	:	:
:	on	on	on	on	on	on	on	on	on	:	:	basis	:	basis	:	of total:	of total:	panel	panel	panel	:	:	:	:	:	:	:	:	:
:	moisture-	moisture-	moisture-	moisture-	moisture-	moisture-	moisture-	moisture-	moisture-	:	:	free	:	free	:	of total:	of total:	panel	panel	panel	:	:	:	:	:	:	:	:	:
:	free basis:	free basis:	free basis:	free basis:	free basis:	free basis:	free basis:	free basis:	free basis:	:	:	free	:	free	:	of total:	of total:	panel	panel	panel	:	:	:	:	:	:	:	:	:
:	Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent	:	:	Percent	:	Percent	:	Percent	Percent	Percent	Percent	Percent	:	:	:	:	:	:	:	:	:
2203A:	29:	6.6	47.0	2203:	47.0	0	2100	23.1	1.19	1.19	5.0	42																	
2203K:	29:	7.6	47.0	2195:	41.6	33	267	12.3	.50	.33	2.9	22																	
2203Q:	29:	6.5	47.0																										
2238A:	37:	7.6	43.2	2203:	47.0	0	2100	23.5	1.24	1.24	6.4																		
2238F:	37:	7.5	43.2	2195:	41.6	33	267	19.3	.89	.59	3.6	44																	
2238H:	37:	7.3	43.2	2226:	38.8	100		14.3	.70		4.3	33																	
2238E:	37:	7.2	43.2								5.5	14																	

¹ Laminating conditions: Pressure 500 lb. per sq. in.; temperature 356° F.; time, 10 minutes. Panels cooled before release of pressure. Panel thickness: one-eighth inch nominal.

² Spirit soluble resin BV-13850.

³ Water soluble resin BR-5995.

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Table 8.--Notched Izod impact values of laminated materials.

Material	Specific:	Izod impact			
	gravity :	With grain		Across grain	
	:	:	:	:	:
	:	Flatwise :	Edgewise :	Flatwise :	Edgewise :
		Ft.lb. per	Ft.lb. per	Ft.lb. per	Ft.lb. per
		in. notch	in. notch	in. notch	in. notch

Glass-fiber-base Phenolic Laminates					
Panelyte PG 5576 ¹	: 1.99	: 25.2	: 18.6	: 21.5	: 17.2

Parallel-laminated Wood Base Materials					
Birch ²	:.....:	11.8	: 10.9	:.....:	:.....:
Birch plywood ²	: .77	: 13.9	:.....:	:.....:	:.....:
Heat stabilized wood ³	: 1.35	: 13.0	:.....:	:.....:	:.....:
High impact compreg ²	: 1.35	: 6.6	:.....:	:.....:	:.....:
Low impact compreg ²	: 1.23	: 2.7	:.....:	:.....:	:.....:

Fabric-base Phenolic Laminates					
Grade L, light weight fabric base ⁴	: 1.34	: 3.8 - 4.8:	2.22	: 3.6 - 4.0:	1.5
Grade C, heavy weight fabric base ⁴	: 1.34	: 2.7 - 3.2:	1.4 - 1.5:	2.5 - 2.8:	1.8 - 2.0

Paper-base Phenolic Laminates					
Papreg ⁵	: 1.40	: 4.0 - 6.0:	0.7 - 0.8:	1.6 - 1.8:	0.5 - 0.6
Grade XX ³	: 1.34	: 1.7 - 1.9:	0.6	: 1.9 - 2.1:	0.6.

Paper-base Lignin-filled Laminates					
43.2 percent lignin, experimental sulfate pulp	: 1.40	: 12.8	: 1.8	: 13.3	: 1.7
38.8 percent lignin, commercial sulfate pulp	: 1.40	: 10.2	: 1.5	: 13.2	: 1.7

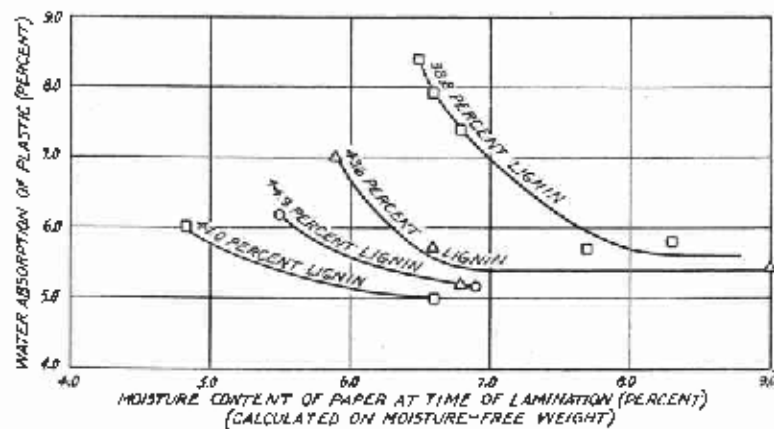
¹Barber, R. W., Mechanical Engineering, page 88, December 1942.

²Stamm, A. J., Seborg, R. M., Millett, M. A., U. S. Forest Products Laboratory Restricted Mimeo. No. 1386 (May 1943).

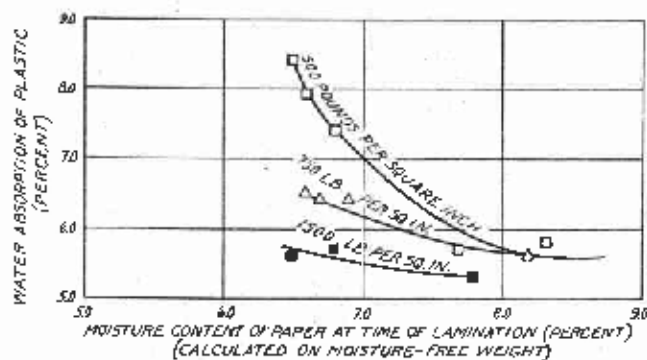
³Unpublished data from the U.S. Forest Products Laboratory (Restricted).

⁴Oberg, T. P., Schwartz, R. T., Shinn, D. A., Modern Plastics, 20, No. 8, 87, April 1943.

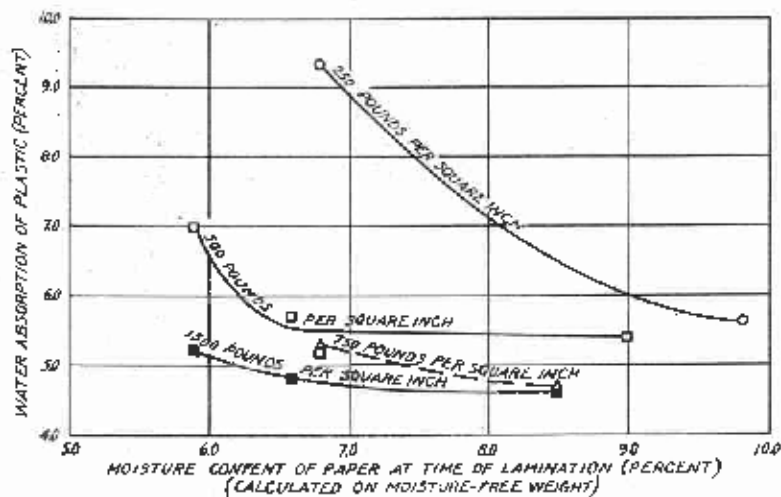
⁵Erickson, E.C.O., Boller, K. H., U. S. Forest Products Laboratory Restricted Mimeo. No. 1319 (March 1943) page 5.



A



B



C

Figure 1.—Relation between moisture content of lignin-treated paper at time of lamination and water absorption of the laminated plastic. Laminating temperature 350° F.; 1, effect of lignin content; loading pressure 500 pounds per square inch; 2, effect of loading pressure; lignin content 30.8 percent; 3, effect of loading pressure; lignin content 4.9 percent.