

INVESTIGATION OF THE RELATIONSHIPS
BETWEEN LIGNIN STRUCTURE AND ITS
MECHANICAL AND ADHESIONAL BEHAVIOR

Project 2421

Report Eleven

A Progress Report

to

PULP MANUFACTURERS RESEARCH LEAGUE

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SUMMARY

The effects of lignosulfonate adhesive viscosity and surface tension on the bonding strength of layups prepared from aged southern pine veneer have been examined in the adhesional phase of the program. The viscosity of electrodialed lignosulfonic acid (ELSA) was increased to the level of the reference formulated adhesive by several methods while maintaining essentially constant surface tension. The object of this unit was to compare the bonding strength afforded by ELSA with that provided by the formulated adhesive under conditions of nominally equal penetration and, hence, surface availability. The bonding strength resulting from the high viscosity ELSA was roughly twice that of the unmodified ELSA control but the level of strength attained under these conditions was notably less than that provided by the formulated adhesive. A limiting value to bond strength from the ELSA alone was indicated to fall in the range of 120-130 pounds per inch. The type of failure obtained from ELSA at the maximum strength level was predominantly cohesive.

Decreasing the surface tension of the high viscosity (autoclaved) ELSA through incorporation of surfactant tended to decrease bonding strength as was found previously with the low viscosity material. On the other hand, reduced surface tension had little or no effect on the bonding strength provided by the formulated adhesive, in which case the predominant failure was within the wood. These results are interpreted in terms of the amount of surfactant required to effect the desired surface tension level. Other potential means to improve bonding are described in the discussion.

The cohesion of an adhesive depends on its ability to distribute an applied stress. The viscomechanical properties of an adhesive should relate to its cohesive nature.

Attempts to isolate the cured LSA adhesive for direct mechanical measurements were of only limited success. Since the mechanical behavior of a cured-adhesive-saturated glass fiber mat is due mainly to the adhesive, this system was used to measure the viscomechanical and tensile properties of LSA adhesives, to determine their solubility, and to relate these data to plywood bonding.

The first series of adhesives examined were: (1) an ammonium base LSA, R-2, (2) ELSA R-44, (3) R-44 and hexamine (2:1) for cross-linking, (4) a methanol-soluble ELSA, low molecular weight, (5) a methanol-insoluble ELSA, high molecular weight, (6) R-44 and glycerin (4%) as a plasticizer, (7) R-44 in 0.01M FeCl_3 and acrylamide (5%) for graft polymerization, (8) R-44 heat concentrated, and (9) phenol-formaldehyde plywood adhesive. Loss tangent values (ratio of energy lost to energy stored) were calculated from vibrational data of reeds cut from the glass fiber mat. Stress-strain data were obtained on specimens using the Instron Tensile Tester. The solubility of the cured adhesive in boiling water was determined by a weight loss.

All of these materials, except the low molecular weight fraction, showed approximately Hookean behavior. This exception suggests that the curing conditions were probably not sufficient to cause much increase in the molecular weight of ELSA. There is a very general trend of loss tangent increasing with elongation to break, as suggested in the literature for linearly viscoelastic materials. It is noteworthy that phenol-formaldehyde, a good adhesive, and crosslinked ELSA have the high values and the FeCl_3 system and LSA, a usually poor adhesive, have the low value with the others in between. The difference between extremes is small, however, suggesting that neither

ELSA nor LSA is too different viscomechanically from a good adhesive. The glycerin did not increase the loss tangent as expected and the Fe^{+3} did not appear to effect graft polymerization, probably due to interaction with ELSA. The solubility data support the extent of cross-linking suggested from the above viscomechanical results, with ELSA 100% soluble - low cross-linking, ELSA-hexamine 80% soluble - intermediate cross-linking, and phenol-formaldehyde 3% soluble - high cross-linking.

The second set of adhesives examined were those used in the plywood bonding - adhesion studies. The range of values of loss tangent for the "nonformulated" adhesives is very small, while those for the formulated adhesives is about as the first set. The range of values of elongation to break is also small. The nonformulated specimens show a slight trend of loss tangent increasing with elongation to break, the cross-linked samples having the high values and ELSA the low values. The autoclaving and surfactants have no noticeable effect on viscomechanical behavior. The formulated adhesive was only 15% soluble, indicating reasonably high cross-linking. The tensile strength, Young's modulus, and loss compliance show a general ranking with ELSA low, cross-linked ELSA middle, and formulated adhesive high. These data, however, appear to depend somewhat on the amount of adhesive present, as was observed also for the first set of adhesives.

In view of the general trend of these viscomechanical data, the observed serious loss in plywood bond strength with surfactant and with cross-linking is unexpected. The role of adhesive penetration on this enigma is discussed.

Future work would (1) establish the role of penetration on bonding strength, (2) introduce cross-links to improve viscomechanical behavior and insolubility, and (3) clarify the role of wood meal as viscosity control, penetration control, cross-linking avenue, and/or agent for stress redistribution. Such information would aid in improving lignosulfonic acid formulations for plywood adhesives.

ADHESIONAL PROPERTIES OF LIGNOSULFONIC ACIDS

INTRODUCTION

Results given in Progress Report Ten revealed that lowering the ELSA surface tension through incorporation of surface-active agent tended to reduce rather than increase bonding strength in layups made from aged southern pine veneer. This was tentatively attributed to preferential sorption of the surfactant at the veneer-adhesive interface or possibly to reduced viscosity. The effect of viscosity on the depth of penetration of a liquid adhesive into a porous substrate is shown by the following equation.

$$l = \sqrt{\frac{\gamma r t \cos \theta}{2\eta}} \quad (1)$$

where l is the distance penetrated in time, t , γ is the surface tension of the liquid, θ is the contact angle formed by the liquid on the solid surface, and η is the coefficient of viscosity of the liquid. Differentiation of Equation (1) with respect to time gives the corresponding rate of penetration equation

$$\frac{dl}{dt} = \frac{\gamma r \cos \theta}{4\eta l} \quad (2)$$

Equation (2) is an idealized equation which is valid only when the contact angle is acute, when laminar flow exists, and when air resistance, gravitational, and inertial effects may be excluded or neglected. In spite of these limitations, Equations (1) and (2) are useful in describing penetration phenomena. It is apparent that both the depth of penetration and the rate of penetration are inversely proportional to the viscosity. It will also be noted that a reduction in surface tension, in itself, should reduce penetration but this effect is usually counterbalanced by

a concomitant decrease in contact angle and, hence, an increase in $\text{Cos } \theta$. Therefore, the net effect of reducing adhesive surface tension on penetration will depend upon the relative changes in viscosity, surface tension, and contact angle.

Addition of surfactant was previously shown to reduce ELSA solids content and viscosity and since reduced viscosity could lead to excessive penetration of the adhesive and poor contact in layup formation, it was decided to examine the effects of ELSA viscosity on adhesion before proceeding with the effects of reduced surface tension. The following summarizes results obtained in examining the effects of these factors on plywood adhesion in systems bonded with ELSA and the reference formulated adhesive.

EXPERIMENTAL

The Effect of ELSA Viscosity on Bonding Strength

The viscosity of ELSA (Run 38, 66-2) was increased by three methods:

1. by freeze drying and then reconstituting to 54% solids,
2. by heat treating (autoclaving) for a total of five hours at 230°F., and
3. by adding a small amount of a high molecular weight polymer.

The viscosity resulting from the first two methods was approximately that of the previously tested formulated adhesive containing wood flour and phenolic resin whereas the viscosity resulting from incorporation of polymer was somewhat higher than that of the unmodified ELSA but lower than that of the formulated adhesive.

In freeze drying, approximately 85 g. of 40% ELSA was reduced to the solid material. The freeze-dried product was then reconstituted to 54% solids by slowly adding distilled water with thorough mixing. Heat treatment consisted of autoclaving

80 g. of the 40% ELSA for a total time of five hours at 230°F. in approximately one-hour intervals. The stepwise treatment was necessary to establish the effect of time on viscosity. Viscosity was increased in the third case by incorporating 1.8% of an experimental high molecular weight polymer into ELSA. The polymer, known to be stable under acidic conditions, was added as a 3% aqueous solution.

The viscosity of the modified ELSA samples was measured at 27°C. with a Brookfield viscometer. Surface tension was measured in duplicate at 73°F. with a calibrated du Nouy Interfacial Tensiometer and the average values were adjusted for the Harkins-Jordan correction factors (1).

Layups were subsequently prepared from aged southern pine veneer utilizing the modified and unmodified ELSA as the adhesive. The veneer was aged 2-3 weeks at 73°F. and 50% R.H. prior to layup formation. Under these conditions the critical surface tension of the veneer has been shown to decline to approximately 28 dynes/cm. The procedures followed in preparing and testing layups were the same as those described previously. Bonding strength results, together with pertinent surface tension and viscosity data, are recorded in Table I.

Bonding strength attained with ELSA as a function of $(\gamma/\eta)^{1/2}$ [refer to Equation (1)] is shown graphically in Fig. 1. Viscosity in this case was apparent viscosity as measured with the Brookfield viscometer.

The Effect of ELSA Adhesive Surface Tension on Bonding Strength

It is apparent from Table I that increasing the viscosity of ELSA tended to increase in bonding strength regardless of the means taken to achieve the higher viscosity. With this information serving as background, a second series of layups was prepared which pursues the effect of lowering the adhesive surface tension to

TABLE I
THE EFFECT OF ELSA VISCOSITY ON PLYWOOD BONDING STRENGTH
(Southern pine veneer; aged 2-3 weeks)

Layout Set No.	ELSA Adhesive	Surface Tension of Adhesive (γ_L), dynes/cm.	Viscosity of Adhesive, cp., (Brookfield 12 r.p.m.)	Bonding Strength, lb./in.	Average	Standard Deviation	Predominant Type of Failure, approx. %
1	Unmodified ELSA No. 66-2 Run 38, approx. 40% solids	43.9	39.5	88.0 16.4 46.8 56.5 67.3 10.1	47.5	+29.9	Cohesional (within adhesive), 90-95
2	ELSA, freeze-dried and reconstituted to 54% solids	44.7	613	90.1 95.2 141.3 132.0 138.7 44.8	107.0	+37.7	Cohesional, 60-90
3	ELSA, autoclaved five hours at 230°F. approx. 40% solids	45.8	613	162.0 32.5 97.6 105.7 97.4 107.2	100.4	+41.2	Cohesional, 50-80
4	ELSA, thickened with 1.8% polymeric material final solids content 34.70%	45.8	115	73.0 64.0 56.7 69.9 105.3 79.0	74.6	+16.8	Cohesional, 80-90

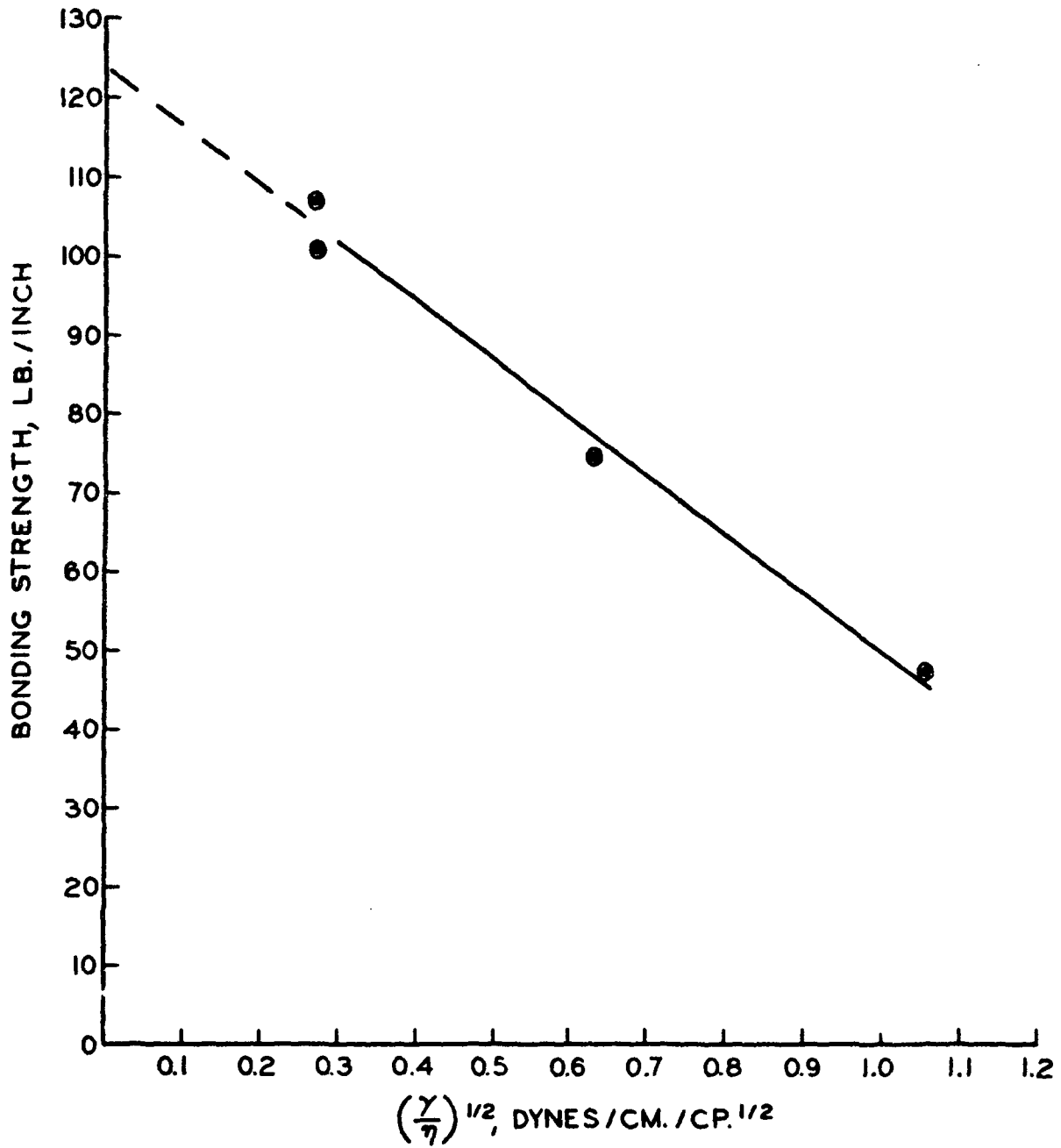


Figure 1. The Effect of ELSA Viscosity on Bonding Strength

a level approaching that of the critical surface tension of the aged veneer while maintaining a viscosity approximating that of the formulated adhesive. In addition to changes in surface tension, the second series examines the effects of a cross-linking agent and a plasticizer on the bonding afforded by ELSA. The cross-linking agent employed was hexamethylenetetramine and the plasticizer was glycerin.

The apparent viscosity of ELSA (Run 38) was adjusted to the level of the formulated adhesive by autoclaving at 230°F. This method was selected on the basis that it does not materially affect the solids content and, hence, the amount of adhesive available at the surface in layup formation. In order to arrive at an approximately equal viscosity level in the presence of the cross-linking agent and plasticizer which produced a marked reduction in viscosity, it was necessary to autoclave the ELSA a total of 9-11 hours to an initial viscosity of 2500 cp. The autoclaving was carried out in intervals of 1 to 3 hours each and, after each interval, the ELSA was cooled to 73°F. for a viscosity measurement.

Because of a limited supply of the reference ELSA (Run 38) it was necessary to substitute a similar material for preliminary surface tension determinations. A second electrolyzed ELSA was supplied by PMRL for this purpose. Candidate surface-active agents were selected on the basis of known properties such as stability in strongly acidic media and ability to reduce surface tension at low concentrations. The agents selected were in addition to those described in Report Ten (2). The surfactants were prepared as 10% stock solutions in distilled water. Metered amounts of the stock solutions were added to 100 grams of the substitute ELSA to provide surfactant concentrations of 0.01, 0.05, and 0.1%. The resulting surface tension was measured at 73°F. as previously described and the results are recorded in Table II.

TABLE II

THE SURFACE TENSION OF ELSA IN THE PRESENCE OF SURFACTANTS

Surfactant	Type	Supplier	Concentration, %	Surface Tension, dynes/cm.
None (ELSA Control)	--	--	--	47.7
Tergitol 12-M-10	Nonionic	Union Carbide Corp.	0.01	43.7
			0.05	40.2
			0.10	39.8
Tergitol 12-P-6	Nonionic	Union Carbide Corp.	0.01	37.6
			0.05	33.7
			0.10	32.5
Neutronyx 656	Nonionic	Onyx Chemical Co.	0.01	40.4
			0.05	38.3
			0.10	37.5
Atlas G-1234	Nonionic	Atlas Chemical Ind.	0.01	47.0
			0.05	42.6
			0.10	42.6
Triton X-200	Anionic	Rohm & Haas Co.	0.01	37.2
			0.05	37.0
			0.10	33.3

The data in Table II indicate that Tergitol 12-P-6 and Triton X-200 would reduce surface tension to a desired level of 30-35 dynes/cm. at concentrations of 0.05 to 0.1%. However, when the same amounts were incorporated into Run 38 ELSA, the resulting surface tension was 5-10 dynes/cm. higher and, hence, a greater amount of surfactant was needed. In general, a surfactant concentration of 0.1% was required or, in effect, about the same amount as previously found for the nonfluorinated surfactants described in Report Ten.

Layups were prepared from the aged southern pine veneer in accordance with the standard procedures for the unmodified and formulated lignosulfonate adhesives; i.e., layups utilizing unmodified and modified ELSA were pressed 30 minutes at 300°F. and those utilizing the formulated adhesive were pressed four minutes at the same temperature. The high viscosity (autoclaved) ELSA was used as is and in stepwise combinations with 0.06-0.1% of Triton X-200, 50% hexamethylenetetramine, and 4% of glycerin. In cases where the plasticizer and/or cross-linking agent were used, the surfactant was added last. The formulated adhesive comprised 200 g. of 30% ELSA (Run 38), 48 g. of 50% phenol-formaldehyde resin, and 30 g. of 200-mesh wood flour as given in Report Nine (3). The formulated adhesive was used as is and in combination with approximately 0.1% of Triton X-200 or Tergitol 12-P-6. Bonding strength results and other pertinent data are summarized in Table III.

DISCUSSION OF RESULTS

As reflected by the results in Table I, bonding with ELSA tends to increase with increase in viscosity but the level of strength attained was notably less than that provided by the reference formulated adhesive (Set 10, Table III). Plotting bonding strength in the manner shown in Fig. 1 would tend to suggest a limiting value for strength from the R-38 ELSA in the range of 120-130 lb. depending upon the means

TABLE III
BONDING STRENGTH IN LAYUPS FROM AGED SOUTHERN PINE VENEER
UTILIZING MODIFIED AND FORMULATED ELSA ADHESIVES

Set No.	Adhesive	Surface Tension of Adhesive (γ_s), dynes/cm.	Viscosity of Adhesive, cp. (Brookfield, 12 r.p.m.)	Bonding Strength, lb./in.	Average	Standard Deviation	Predominant Type of Failure, approx. %
5	ELSA No. 66-2, Run 38	44.6	39.5	40.8 61.2 94.1 52.1 44.3 81.8	62.4	±21.4	Cohesional (within adhesive) 85
6	Autoclaved ELSA	50.7	664	100.4 104.7 149.6 99.2 125.6 111.0	115.1	±19.5	Cohesional, 70
7	Autoclaved ELSA, 0.1% Triton X-200	36.2	650	79.6 75.1 72.9 85.1 106.0 64.1	80.5	±14.3	Cohesional, 80
8	Autoclaved ELSA with 50% hexamethylenamine and 0.06% Triton X-200	33.9	550	21.8 64.8 34.2 33.9 49.0 62.5	44.4	±17.3	Cohesional, 99
9	Autoclaved ELSA with 50% hexamethylenamine, 4% glycerin, and 0.1% Triton X-200	33.6	500	25.5 41.0 50.2 43.6 45.6 46.2	42.0	±8.1	Cohesional, 99
10	Formulated adhesive	42.3	650	259.9 180.8 226.2 296.1 289.1 308.3	260.1	±35.3	Within wood, 70-90
11	Formulated adhesive, 0.12% Triton X-200	36.5	700	228.1 254.6 249.5 234.6 312.8 218.9	250.0	±20.7	Within wood, 70-90
12	Formulated adhesive, 0.1% Tergitol 12-P-6	35.9	700	255.7 246.9 230.0 229.6 287.3 226.9	255.5	±26.6	Within wood, 80-90

taken to attain the given viscosity. The bonding strength value for the autoclaved ELSA in the second series (Set 6) also falls close to this range. In line with the measured strength values in Table I, the percentage of cohesive failure tends to decrease as viscosity increased, i.e., the higher viscosity resulted in a higher percentage of wood failure.

As was found previously, incorporation of surfactant into the ELSA lowered bonding strength rather than improving adhesion through improved surface wetting (Table III). In those cases involving the formulated adhesive (Sets 10-12) incorporation of surfactant had little or no effect on bonding strength although the percentage of wood failure was somewhat higher in Set 12. Possible causes for the poor bonding in the sets containing hexamethylenetetramine will be considered in the cohesive section of this report.

As indicated earlier, the amount of surfactant required to lower the surface tension of Run 38 ELSA and the formulated adhesive to 30-35 dynes/cm. was more than had been anticipated on the basis of the preliminary work with the substitute ELSA. In fact, the amounts required were generally in excess of the critical micelle concentration. Hence, the reduced strength in Set 7 may be due to surface excess of the surfactant or possibly to increased penetration due to lowering of the contact angle. Since the amount of surfactant required differed in the two ELSA samples, the adhesive effects found with the Run 38 product may not apply to all lignosulfonate adhesives. In the case of the formulated adhesive, the surfactant may have been adsorbed, in part, on the wood flour and, therefore, not present in excess at the surface. In any event, lowering the surface tension through incorporation of a surfactant has not proved to be a satisfactory approach to improved bonding with the Run 38 ELSA in spite of some evidence in the literature (4) to show that improved strength in Douglas-fir plywood with a urea-formaldehyde resin was attained in the presence of a surfactant.

Another approach to the problem of improved bonding is one of incorporating into the adhesive low molecular weight materials which are capable of cross-linking or polymerizing and thereby forming an integral part of the ultimate bond. Consideration was previously given to this approach by enriching the whole ELSA liquor with the low molecular weight fraction. However, this fraction represented only 4% of the total and presumably a substantial amount would be required to effect the desired decrease in surface tension. Hence, this approach was not actively pursued. An alternative means to improve bonding with the existing formulated adhesive would consist in treating the veneer surface prior to gluing to produce an increase in surface energy. This might be accomplished by an in-line corona treatment of the veneer. The effect of corona treatment on bonding between birch strips has been reported by Goring (5).

FUTURE WORK

While the results reported in this and previous reports indicate that improved bonding from ELSA may not be achieved through incorporation of commercial surfactants, it is not concluded that reduced surface tension and improved surface wetting is an implausible approach to improved bonding of southern pine veneer. The earlier surface chemical study revealed that an unfavorable condition for good adhesion existed as a result of the high surface tension of the lignosulfonate adhesive relative to the critical surface tension of aged veneer. It is proposed therefore to pursue the study of lignosulfonate surface tension as related to bonding strength. Low molecular weight lignosulfonates, including nonelectrodialyzed and possibly chemically modified materials, would be examined for their effects on surface tension and bonding. The study would not be confined to ELSA alone but may incorporate the use of viscosity modifiers and cross-linking agents. The goal of the program would be the development of commercially acceptable adhesive formulations tailored to meet the demands of specific plywood systems.

MECHANICAL PROPERTIES OF LIGNOSULFONIC ACIDS

INTRODUCTION

The ability of an adhesive to withstand an applied stress depends upon the stress distribution within the adhesive. Internal stress redistribution is accomplished through molecular relaxation, a property which is manifest in the viscomechanical behavior of the adhesive (6). It is the goal of the cohesional phase of this project to relate the viscomechanical properties of lignosulfonic acid (LSA) materials to their adhesive strength, particularly in plywood operations. Greater understanding of the relationship will aid significantly the development of adhesive formulations.

Previous work (2) has shown that the potentially adaptable vibrating reed method of measuring viscomechanical properties of adhesives was possible when a porous substrate such as paper was used for the reed. However, lightweight paper substrate, which was just sufficient to withstand the hot acid conditions of a mild cure, dominated the viscomechanical properties of the reed so that the contribution of the adhesive was difficult to discern.

The work presented in this report covers (1) the attempted isolation of the adhesive for direct viscomechanical measurements, (2) the viscomechanical and tensile measurement of adhesives cured in glass fiber mats, (3) the relationship of the mechanical properties of the adhesive to plywood bonding behavior, and (4) the extent of hot water insolubility of the cured adhesives.

EXPERIMENTAL

The Isolation of Adhesives

The electrodialed lignosulfonic acid (ELSA) 65-24-R44 (a sodium-based, heat-treated whole liquor) was used with Millipore filter (SM 5.0 μm . pore

size, cellulose acetate, Millipore Filter Corp., Bedford, Mass.). The substrate was (1) saturated with the adhesive, (2) put between aluminum foil, chrome plates, and finally 1/8-inch wood veneer, (3) placed between heated platens, and (4) cured at 310°F. at 150 p.s.i. for 30 min. The substrate was found to be severely charred and brittle from these conditions. It was reasoned that any organic soluble substrate would probably degrade severely under the temperature and acid conditions of the curing adhesive.

Layups made with paper in previous work were examined for potential adhesive isolation. The specimen was placed into concentrated sulfuric acid maintained between 18-20°C. to dissolve the paper without affecting the LSA. The lighter weight paper (49 lb./TAPPI ream) was removed in several hours from the 65-24-R44 heat-concentrated ELSA. Although the adhesive layer was coherent (some pinholes) it was too weak to be handled. This adhesive showed only small amounts of cellulose fiber under a polarized microscope. The formulated adhesive was similarly isolated and could be handled with care. In both cases some color developed in the sulfuric acid.

Specimens from the heavier paper (149 lb./TAPPI ream) layups were likewise placed in concentrated sulfuric acid in the temperature range 18-20°C. The paper never dissolved completely even after 24 hours and the sulfuric acid became very dark. However, an adhesive layer could be easily isolated from the substrate with the aid of dissecting needles. This layer contained many cellulose fibers as seen under the polarized microscope. The cellulose in the formulation appeared to be highly swollen.

A sample of 65-24-R44 adhesive in a glass fiber matrix was placed in concentrated sulfuric acid with the temperature held between 18-20°C. The acid became quite dark, indicating solution of some ELSA. Other cellulose solvents would probably also affect the ELSA.

Layups were made (65-25-R44 at 310°F. and 150 p.s.i. for 30 min.) using 149 lb./TAPPI ream kraft linerboard which had been treated with a low surface energy agent for easy release of the adhesive. The treatment consisted of spraying the paper surface with a silicon spray parting agent (Injection Molders Supply Co., Cleveland, Ohio) and placing it in an air-circulating oven at 360°F. for 5 min. This treatment was done twice.

The adhesive bled through the treated paper quite easily in spots via the big channels which is quite different from the usual wetting. The paper lamina could be peeled from the adhesive layer in areas where there was little or no bleed through. However, the isolated adhesive contained much fiber.

The Glass Fiber Mat System

It had been observed in previous work (3) that a glass fiber mat, saturated with adhesive and cured, derives most of its coherence from the adhesive. Such a system could then be used to measure the mechanical properties of the adhesive.

A 5.5-cm. diameter glass fiber mat (934AH, glass fiber filter disk, Reeve Angel, Clifton, N.J.) conditioned at 73°F. and 50% R.H. was weighted. It then was saturated with adhesive by dropping it from a pipet uniformly over the mat. Five of these were placed, as shown in Fig. 2, between 6 x 6 inch aluminum foil (Reynolds Wrap), which had been previously cleaned with acetone and treated with silicon parting agent (three spray coats, with five minutes air drying between coats and a final drying at 360°F. for five minutes in an air-circulating oven). The aluminum foil was then placed between chromed brass plates for flat support, and then between 1/8-inch pine veneer for temperature modulation. The whole system was then placed between heated platens for the desired temperature, pressure, and time.

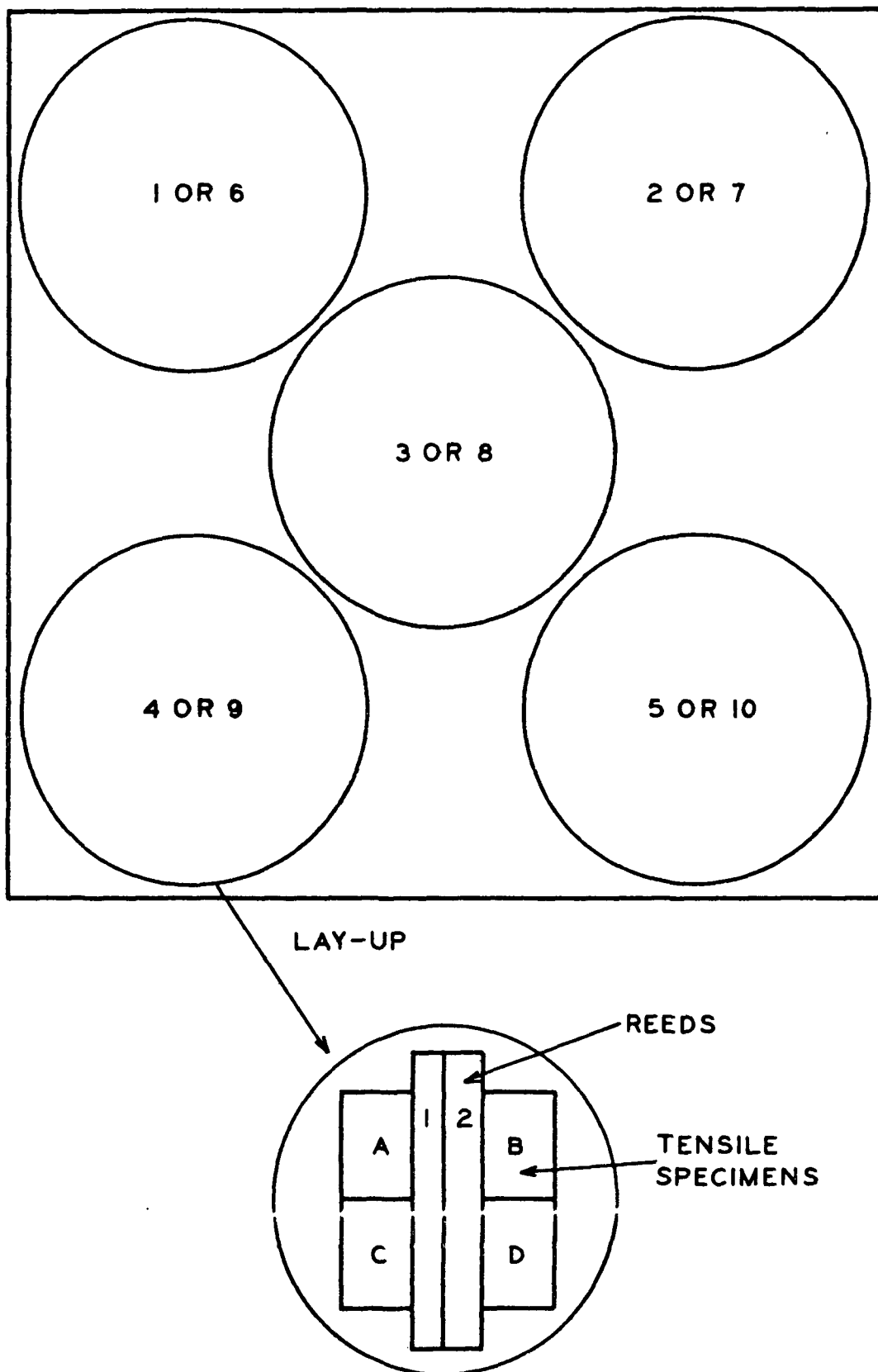


Figure 2. Disk Arrangement and Cutting Pattern

The cured disks in the aluminum foil were allowed to condition at 73°F. and 50% R.H. for several days. Three of the disks were removed from the foil and weighted. Two 0.25 x 2.0-inch reeds and four 0.50 x 0.75-inch tensile tabs were cut from each disk, as shown in Fig. 2, by means of a razor blade and accurate metal gages. The cutting duplication was within 1% and the actual area of the specimen was 97% of the true area (see Appendix I). Each reed and tab was weighted and the average caliper of each was determined with a thickness gage. The reeds were subjected to vibrating reed measurements and the tabs to tensile measurements.

The first series of adhesive materials examined were:

- (1) 67-36-R2, an ammonium base LSA, 39% solids
- (2) 65-24-R44, a sodium-base, heat-treated ELSA, 37% solids
- (3) 65-24-R44 with hexamine (hexamethylenetetramine), 2:1 by weight, for cross-linking of phenols
- (4) a methanol-soluble ELSA, low molecular weight
- (5) a methanol-insoluble ELSA, high molecular weight
- (6) 65-24-R44 and glycerin, 4% by volume, as a plasticizer
- (7) 65-24-R44 in 0.01M FeCl_3 and acrylamide, 5% by weight for graft polymerization on the carbohydrate present
- (8) 65-24-R44 which had been heat concentrated, 38% solids
- (9) phenol formaldehyde resin, 50% solution (CR 9357, Catalin Corp. of America, Chicago, Ill).

All of these materials, except the methanol fraction, had been used in previous studies (2). These adhesives were cured at 310°F. and 150 p.s.i. for 30 min. All of them stuck to the aluminum foil to some degree. The phenol formaldehyde was especially tight but the foil did peel off. The samples with glycerin and with acrylamide tended to disintegrate in removal so that big enough parts of only two disks from each were usable.

The second set of adhesives were those employed in the adhesion study (see Table III, p. 12, for their description). When cured at the same temperature and pressure used above and in the plywood layups, the saturated disks squished out into

distorted shapes. By cutting the pressure back to 28 p.s.i., the disk shape stayed essentially intact, so the curing conditions for this set of adhesives was 310°F. at 28 p.s.i. for 30 min. for the ELSA samples and 5 min. for the formulations. The time of cure was the same as that used for the plywood layups.

Vibrating Reed Measurements

The vibrational measurements of the reeds were obtained as described in Progress Report Nine (3) by use of an audiooscillator coupled through an amplifier to a recording head (vibration transducer) with input voltage to the head held constant at 3.6 volts. To increase the accuracy of the frequency, an electric counter (Hewlett Packard) was attached to the recorder head for measurement of the input frequency.

For reeds from the first set of adhesives, the vibrational amplitude was measured by means of a traveling telescope. A plot of amplitude vs. frequency was used to determine the resonance frequency, ω_0 , and the band width, $\Delta\omega$, as previously described (2). The loss tangent is equal $\Delta\omega/\omega_0$. The accuracy of the loss tangent is ± 0.002 and is due in the most part to variations between disks.

For reeds from the second set of adhesives, the ω_0 and $\Delta\omega$ were observed by a method which was much faster and as accurate as the above method. Rather than measure the reed vibrations by means of a traveling telescope, an oscilloscope was used as follows: a beam of light from a 6 volt bulb was shown on a photocell, the output of which was fed to the oscilloscope. The end of the vibrating reed was placed at a slight angle in the light beam, such that the intensity of the light striking the photocell was proportional to the vibrational amplitude of the reed. Thus, by slowly scanning through the frequency range, the point of ω_0 was observed directly, then the frequencies for amplitude of $1/\sqrt{2}$ times the maximum amplitude were observed, their difference being $\Delta\omega$.

Tensile Measurements

The 0.5 x 0.75-inch tab was placed in an Instron Tensile Tester by means of line clamps with an initial setting of 0.6-inch apart and moved at the rate of 0.002 in./min. The force in pounds required to maintain this strain rate was recorded as a function of time until the specimen ruptured or pulled apart. See Fig. 3 for typical Instron distance-force plots.

Water Solubility of the Adhesive

Fragments of a disk left over from cutting reeds and tabs were weighed and placed in gently boiling water for a period of three hours. The water level was maintained constant by periodic addition of boiling water.

Determination of the Weight of Adhesive in a Tab

The weight of adhesive in a disk, \underline{M}_A , is

$$\underline{M}_A = \underline{W}_A - \underline{W}_D \quad (3)$$

where \underline{W}_A and \underline{W}_D are the weights of the disk with and without adhesive, respectively.

The weight of adhesive in a tab, from this disk, \underline{m}_A , is

$$\underline{m}_A = \underline{W}_A (\underline{M}_A / \underline{W}_A) \quad (4)$$

where \underline{W}_A is the weight of a tab with adhesive.

RESULTS AND DISCUSSION

In order to achieve maximum sensitivity of observations to adhesive behavior, efforts were made to isolate a cured LSA adhesive for direct measurements of mechanical and solubility properties. An organic soluble porous substrate (cellulose acetate Millipore filter) degrades under the high acid and temperature conditions

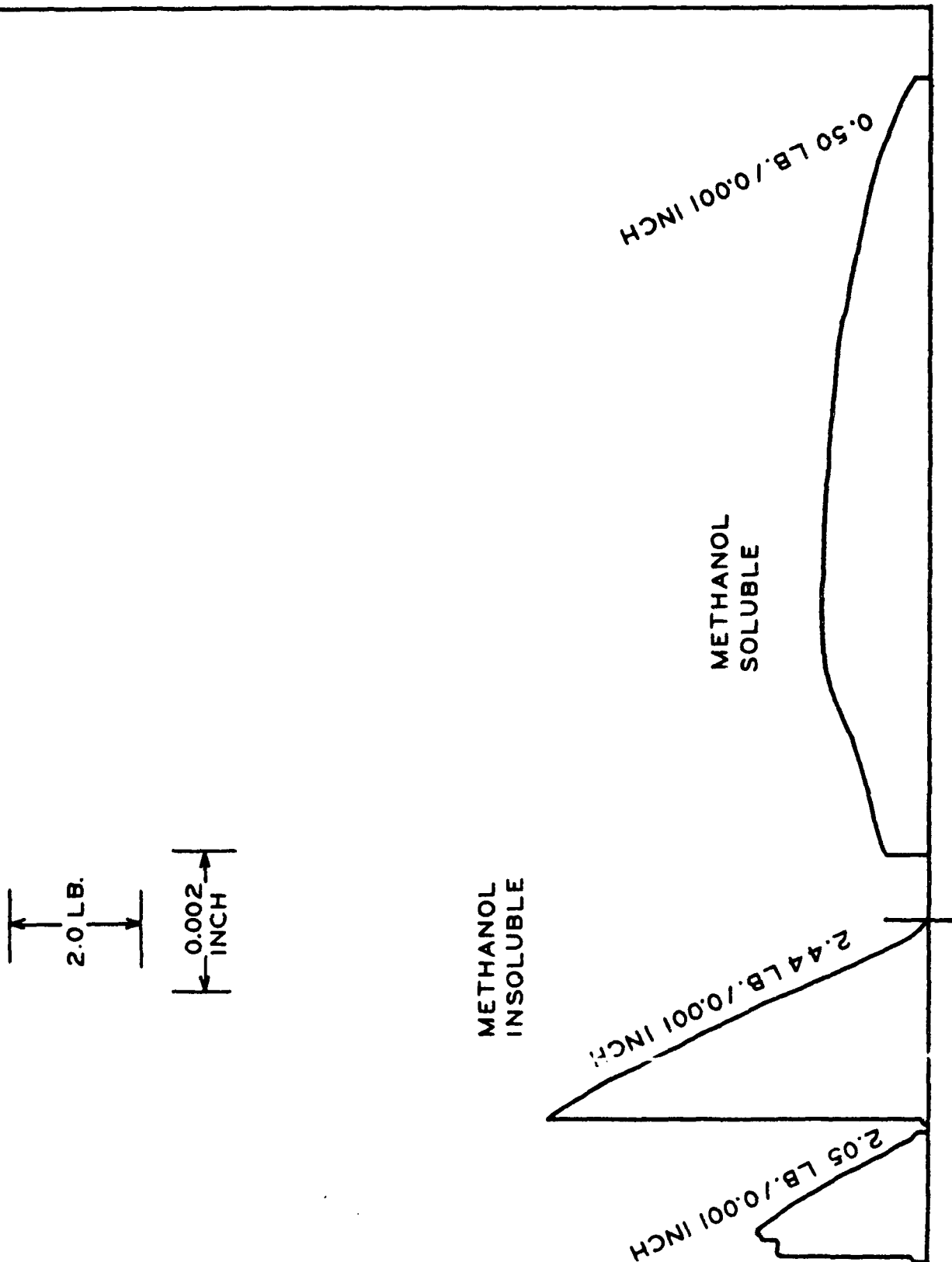


Figure 3. Example of Data From the Instron Tensile Tester

of the adhesive cure, and it is reasoned that more stable substrates will be insoluble. An adhesive layer was isolated from a paper layup by treatment with cold sulfuric acid. However, cellulose fibers are contained in the layer and some ELSA dissolves. Mechanical isolation of an adhesive layer is possible from paper treated to give a low energy surface, but here too some fibers are contained in the layer.

Preliminary work (3) with adhesive-saturated glass fiber mats showed that they derive most of their mechanical strength from the adhesive. This system was thus chosen to study the viscomechanical, cohesive, and solubility behavior of adhesives.

The specimen's loss tangent (the ratio of energy lost to energy stored), reflecting both fluidlike and solidlike behavior while being independent of sample geometry, was determined from vibrating reed data. The tensile properties of the specimen were determined by means of the Instron Tensile Tester.

The first series of materials examined were: (1) an ammonium-base LSA R-2, (2) ELSA R-44, (3) R-44 and hexamine (2:1) for cross-linking, (4) a methanol-soluble ELSA, low molecular weight, (5) a methanol-insoluble ELSA, high molecular weight, (6) R-44 and glycerin (4%) as a plasticizer, (7) R-44 in 0.01M FeCl_3 and acrylamide (5%) for graft polymerization, (8) R-44 heat concentrated, and (9) phenol-formaldehyde plywood adhesive. (See p. 19 for more detailed descriptions of the adhesives.) These were cured at 310°F. and 150 p.s.i. for 30 min. Both (6) and (7) tended to disintegrate when removed from the processing aluminum foil.

Figure 3 is an example of the force-elongation data from the Instron. The sample from the methanol-insoluble ELSA are typical of adhesives examined, having almost Hookean behavior. The slope of the initial linear part of the curve is a macroscopic measure of the Young's modulus, \underline{E}' . A microscopic measure of \underline{E}' is possible from vibrating reed data (3), but it assumes the sample is homogeneous and it is very

dependent on sample thickness. The elongation to break, ϵ_b , and the force to break are also noted, with the break being abrupt and usually located close to one of the clamp lines. The only exception was the methanol-soluble material which is not Hookean and pulls out like taffy (right curve, Fig. 3) suggesting that the curing conditions were probably not sufficient to cause much increase in the molecular weight of ELSA. In order to express the force as a stress, the force was divided by the weight of the adhesive in the tab. This was necessary since the heterogeneous and possibly porous nature of the matrix makes the geometrical cross-sectional area of the specimen meaningless for stress calculation.

The results are listed in Table IV along with the loss compliance, $\underline{J}'' = (\text{loss tangent})/\underline{E}'$, a property dependent on both the loss tangent and Young's modulus, and the solubility of the cured adhesive in boiling water (3-hr. extraction). (See Appendix II for the basic data.)

The range of values of loss tangent and ϵ_b is fairly small. The loss tangents of phenol-formaldehyde, methanol-soluble, and cross-linked ELSA have the high values and the LSA, FeCl_3 system, and methanol-insoluble have the low values as might be anticipated from their usual adhesive behavior and their observed flow behavior. It is surprising that the glycerin did not change the loss tangent. The Fe^{+3} ion was probably tied up with ELSA minimizing any graft polymerization. The tensile strength and \underline{E}' values are influenced to some extent by the mass of adhesive in the tab and thus \underline{J}'' reflects this.

An abstract of the paper "The relation of the small-strain loss compliance to the ultimate elongation capability of an amorphous elastomer" by Landel (7) reports that for a linear viscoelastic material the relationship

$$\epsilon_b \propto \sqrt{\epsilon_d} \sqrt{J''} \quad (5)$$

should be valid, where ϵ_d is energy dissipated. Since

$$\epsilon_d = E' \text{ (loss tangent)} \quad (6)$$

when the loss tangent is small as in this case,

$$\epsilon_b \propto \sqrt{E'(\Delta\omega/\omega_0)} \sqrt{(\Delta\omega/\omega_0)/E'} \propto \text{(loss tangent)} \quad (7)$$

This relationship seems reasonable since the greater the flowlike behavior, the better the stress distribution, the larger the elongation to break.

Plotted in Fig. 4 is the loss tangent vs. ϵ_b for all but the methanol-soluble material which is not linearly viscoelastic. The wide scatter is probably not too surprising but it is of interest to note that the phenol-formaldehyde, a good adhesive, and cross-linked ELSA are at the upper right and the FeCl_3 system LSA, usually a poor adhesive, are at the lower left with the rest in between. Also, note that the difference between extremes is small suggesting that neither ELSA nor LSA is too different visco-mechanically from a good adhesive.

Other correlations were viewed and the best result was \underline{J}'' vs. ϵ_b (see Fig. 5) with a similar left-right distribution as above. This is encouraging since \underline{J}'' includes both loss tangent and Young's modulus from both microscopic and macroscopic measurements, respectively. The large value of \underline{J}'' for R-44 heat concentrated may be a result of the high adhesive mass with possibly some fracturing making the supporting "area" smaller than the apparent "area." Plotted in Fig. 6 is \underline{J}'' vs. tensile strength with little apparent correlation. More will be said of this later.

Cross-linking has been assumed and the solubility data support this as the ELSA is 100% soluble (fiber mat disintegrated) showing little cross-linking, while the ELSA-hexamine is only 80% soluble and the pieces remained intact showing measurable

TABLE IV
VISCOMMECHANICAL DATA FOR THE FIRST SET OF ADHESIVES

Description	Loss Tangent $\Delta\omega/\omega_0$	Adhesive in ^a Tensile Specimen, g.	Elongation to Break, $10^3 \times$ in.	Young's Modulus ^a , E' , lb./0.001 in./g. adhesive	Loss ^b Compliance $10^3 \times J''$	Adhesive Bond Strength, lb.	Adhesive Solubility, %	Tensile Strength, lb./g. adhesive
Glass fiber mat	0.059	--	0	0	--	--	--	0
○ R-2 (LSA)	0.0268	0.0070	2.2	341	7.9	--	--	723
	0.0256	0.0067	2.2	363	7.1	--	--	759
	0.0242	0.0058	2.0	400	6.1	--	--	748
X R-44	0.0332	0.0047	2.0	398	8.3	--	100	785
	0.0370	0.0039	1.5	563	6.6	--	--	793
	0.0286	0.0047	1.8	384	7.5	--	--	741
□ R-44-Hexamine (2:1)	0.0401	0.0047	2.7	315	12.7	--	--	816
	0.0380	0.0051	3.0	333	11.4	--	80.2	807
	0.0353	0.0050	2.4	321	11.0	--	--	660
△ MeOH sol.	0.0384	0.0052	6.2	136	28.2	--	--	--
	0.0333	0.0067	8.7	74	45	--	--	--
	0.0385	0.0060	8.6	78	49	--	--	--
◇ MeOH insol.	0.0242	0.0065	2.3	335	7.2	--	--	831
	0.0268	0.0072	2.3	297	9.0	--	--	689
	0.0277	0.0063	2.3	399	6.9	--	--	805
⊗ R-44 + 4% glycerin	0.0310	0.0033	2.0	597	5.2	--	--	854
	0.0304	0.0027	1.6	571	5.3	--	--	709
	--	--	--	--	--	--	--	--
⊙ R-44 - 0.01M FeCl ₃ + 5% acrylamide	0.0272	0.0051	1.7	311	8.8	--	--	594
	0.0273	0.0052	2.7	360	7.6	--	--	895
	--	--	--	--	--	--	--	--
● R-44 heat concentrated	--	--	--	--	--	--	--	--
	0.0311	0.0315	2.8	121	25.7	--	--	197
	--	--	--	--	--	--	--	--
▽ Phenol-formaldehyde	--	--	--	--	--	--	--	--
	0.0370	0.0089	3.2	295	12.5	--	2.6	807
	--	--	--	--	--	--	--	--

^aThe specimens were 3/4 x 1/2 inch with the clamp distance = 0.60 in. and the strain rate equal to 0.002 in. per min.

^bThe loss compliance, J'' = loss tangent/ E' .

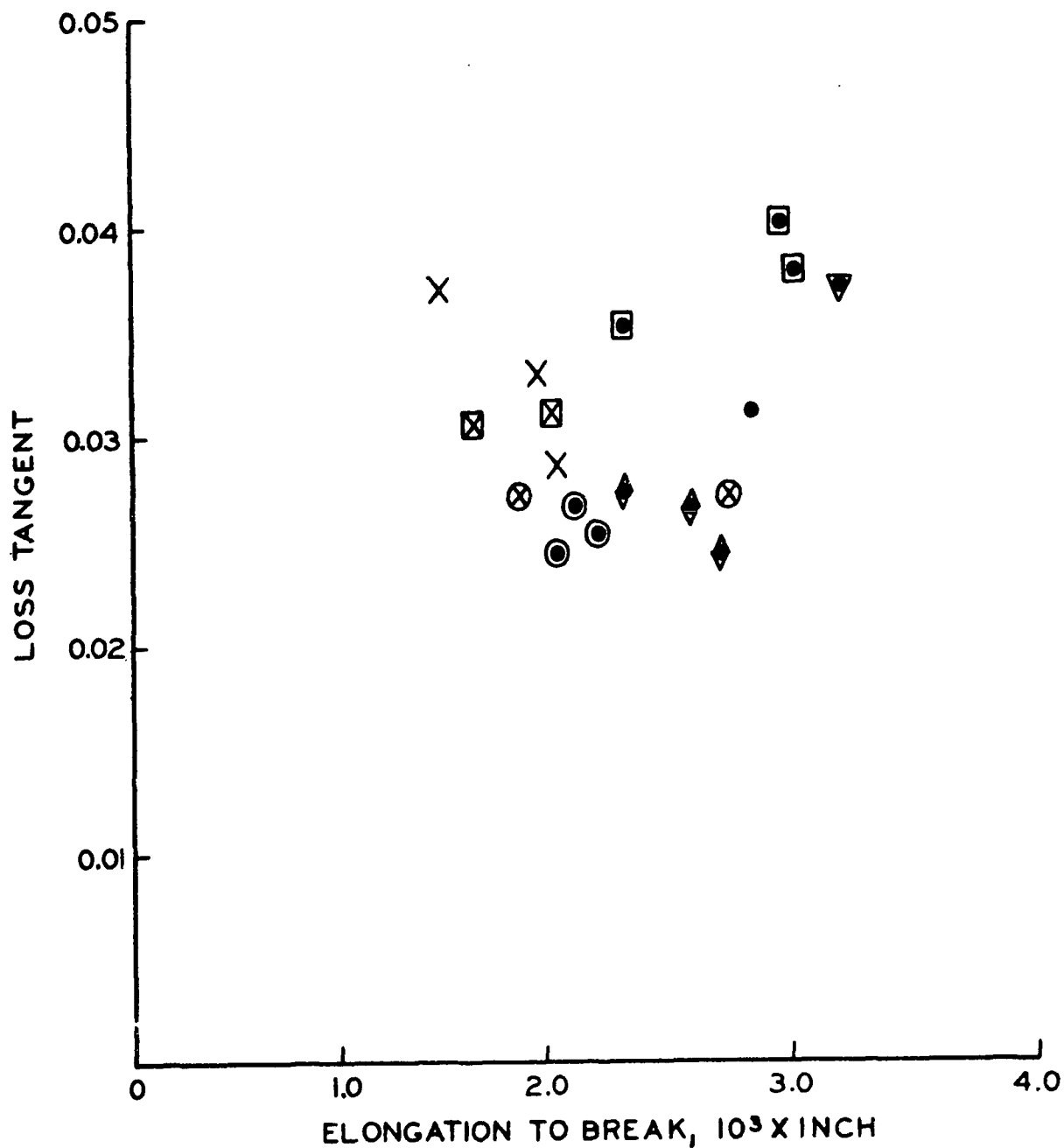


Figure 4. Loss Tangent vs. Elongation to Break for the First Set of Adhesives (See Table IV for Data Identification)

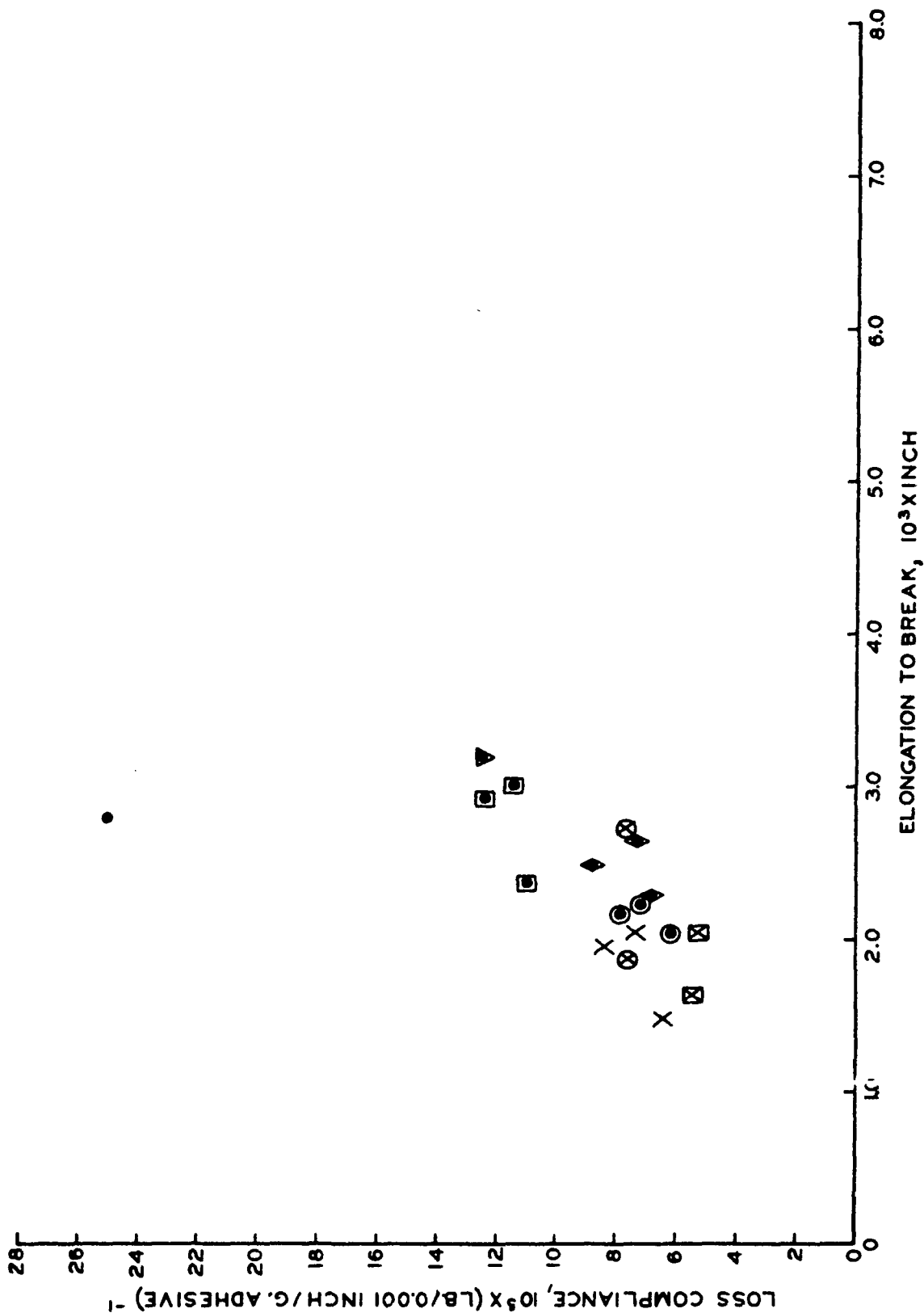


Figure 5. Loss Compliance vs. Elongation to Break for the First Set of Adhesives (See Table IV for the Data Identification)

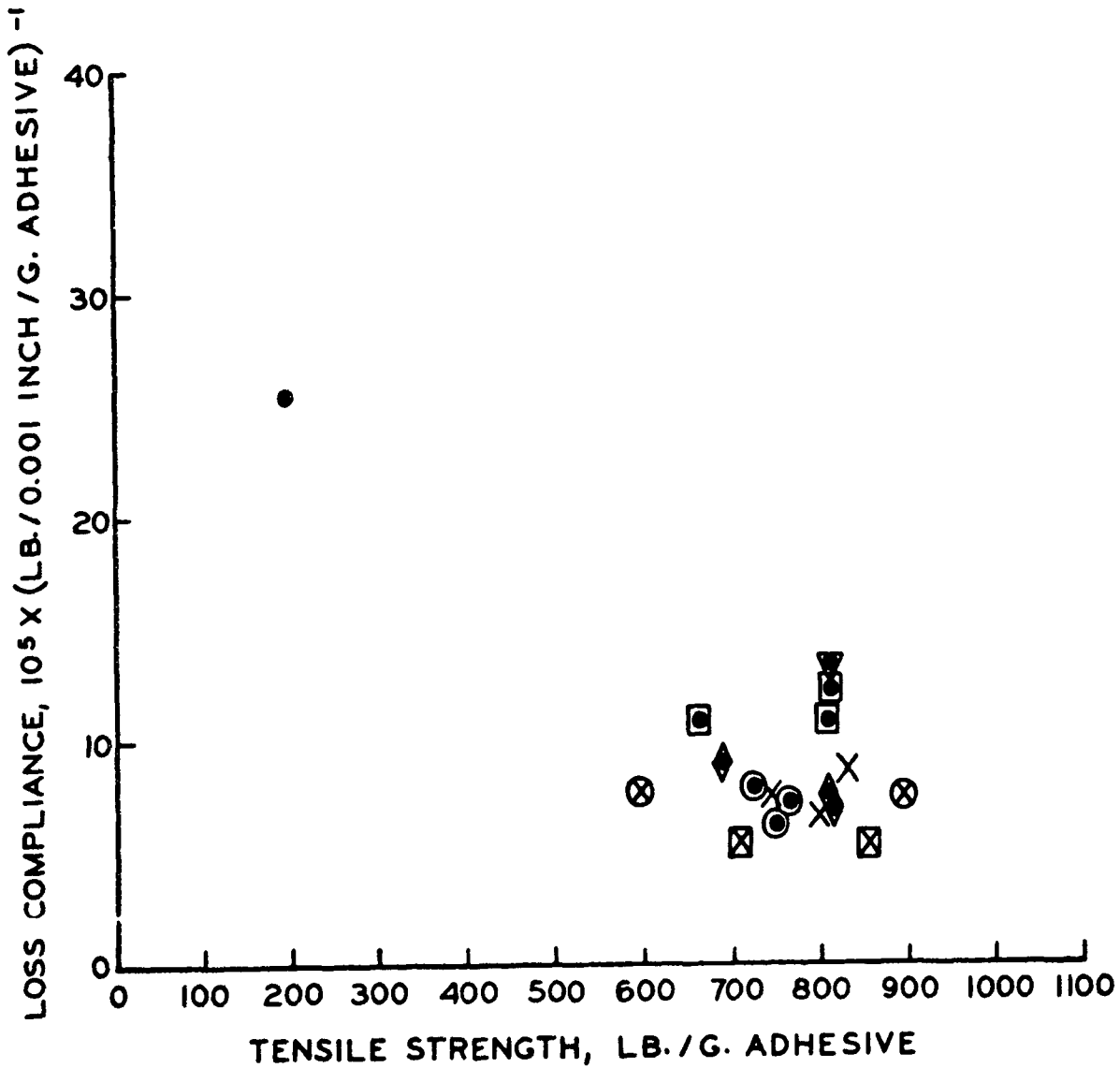


Figure 6. Loss Compliance vs. Tensile Strength for the First Set of Adhesives (See Table IV for Data Identification)

cross-linking. The phenol-formaldehyde is 3% soluble and the pieces remained intact showing very good cross-linking.

The method was repeated on the adhesives used in the adhesion studies of plywood layups (see the first section of this report) for the correlation of visco-mechanical data to plywood bond strength. The adhesives studied were: (1) ELSA R-38, (2) R-38 autoclaved (viscosity similar to formulation), (3) autoclaved R-38 with 0.1% Triton X-200, (4) same as (3) with hexamine (2:1), (5) same as (4) with 4% glycerin, (6) R-38 phenol formaldehyde-wood meal formulation, (7) same as (6) with 0.12% Triton X-200, (8) same as (6) with 0.1% Tergitol 12-P-6. Serious glass mat deformation was experienced at 150 p.s.i. with these ELSA (R-38) based materials (not experienced with R-44) so the pressure was reduced to 28 p.s.i. The normal cure time was 5 min. for the formulation samples. The results are listed in Table V. (See Appendix II for the basic data.)

The range of values of loss tangent for the "nonformulated" adhesives is very small, while the range for the formulated adhesives is about as the previous data. However, a filtration of the wood meal was noted when the adhesive was applied to the glass fiber mats leading to a heterogeneous distribution of components which may cause increased scatter to the measured data. The range of values of elongation to break is small. The tensile strength and E' , and thus J'' values are influenced to some extent by the mass of the adhesive in the specimen.

From a plot of loss tangent vs. ϵ_b (Fig. 7) the nonformulated data show a slight trend with the cross linked samples upper right and ELSA lower left. The autoclaving and surfactants have no noticeable effect on the viscomechanical behavior. The results from the formulated adhesives are quite scattered due to the loss tangent data.

The plot of \underline{J}'' vs. ϵ_b (Fig. 8) shows only the ranking of \underline{J}'' as the ϵ_b values do not vary much. The ranking shows the ELSA samples on the bottom, the cross-linked samples in the middle, and the formulated adhesives on top. The autoclaving and surfactants have no noticeable effect on \underline{J}'' .

The plot of \underline{J}'' vs. tensile strength (Fig. 9) is very interesting at first sight suggesting a functional relationship. However, since ϵ_b is about the same for all samples and since all samples have approximately linear stress-strain curves, the tensile strength is approximately proportional to \underline{E}' . Since the loss tangent is about the same for all samples, particularly the nonformulated ones, this plot is approximately a hyperbola, $1/\underline{E}'$ vs. \underline{E}' . For about the same reason, the plot in Fig. 6 fits with that of Fig. 9, even the R-44 concentrate point.

Since only 15% adhesive weight loss was found by boiling water extraction of the formulated adhesive, reasonably high cross-linking is suggested. The pieces did not disintegrate but curled badly due to the nonuniform distribution of wood meal.

The general trend of these viscomechanical data is seen in the similar loss tangents and the increase in \underline{J}'' from ELSA-only to the formulated adhesives. When comparing these observations with the measured plywood bond strength, the serious loss in bond strength with surfactant and with cross-linking is unexpected. The increased viscosity by autoclaving ELSA with little change in viscomechanical behavior leads to increased bonding by reducing the penetration, leaving more adhesive in the bonding region. If the reasoning is followed using the Washburn equation [see Equation (2), p. 4] describing penetration rate into a capillary, increasing viscosity reduces penetration, as mentioned above. Reducing the surface tension may reduce penetration if the $\text{Cos } \theta$ value does not change much with a change in γ , for example with θ close to zero (good wetting). However, with θ in the midrange, the

TABLE V
VISCOMETICAL DATA AND ADHESIVE STRENGTH FOR THE SECOND SET OF ADHESIVES

Description	Loss Tangent $\Delta w/w_0$	Adhesive in ^a Tensile Specimen, g.	Elongation to Break, 10 ³ x in.	Young's Modulus, E', lb./0.001 in. ² /g. adhesive	Loss ^b Compliance 10 ⁵ x j''	Adhesive Bond Strength, lb.	Adhesive Solubility, %	Tensile Strength, lb./g. adhesive
○ R-38	0.0352 0.0342 0.0333	0.0071 0.0077 0.0074	2.3 2.5 2.5	318 267 330	11.1 12.8 10.1	62	--	705 591 686
□ R-38 autoclaved	0.0334 0.0334 0.0329	0.0098 0.0076 0.0089	2.2 2.7 2.3	223 315 256	15.0 10.6 12.9	115	--	452 751 499
△ R-38 autoclaved + 0.1% Triton X-200	0.0314 0.0315 0.0297	0.0121 0.0129 0.0133	2.1 2.2 2.2	241 212 225	13.0 14.9 13.2	81	--	429 432 454
● R-38 autoclaved-Hexamine (2:1) + 0.06% Triton X-200	0.0316 0.0312 0.0331	0.0155 0.0179 0.0189	2.3 2.4 2.3	165 169 140	19.2 18.5 23.6	44	--	375 350 338
◇ R-38 autoclaved-Hexamine (2:1) + 0.06% Triton X-200 + 4% glycerin	0.0344 0.0338 0.0332	0.0219 0.0197 0.0189	3.1 2.4 2.8	133 149 147	25.9 22.7 22.6	42	--	367 318 374
X Formulated adhesive	0.0416 0.0283 0.0218	0.0314 0.0292 0.0337	2.7 2.5 2.4	92 137 81	45.2 20.7 26.9	260	15.8	-- 306 219
⊠ Formulated adhesive + 0.12% Triton X-200	0.0375 0.0420 0.0316	0.0398 0.0315 0.0339	2.1 2.3 2.3	100 105 92	37.5 40.0 34.3	250	--	216 273 213
⊙ Formulated adhesive + 0.1% Tergitol 12-P-6	0.0388 0.0332 0.0282	0.0287 0.0317 0.0329	2.5 2.4 2.4	112 111 112	34.6 29.9 25.2	255	--	246 238 256

^aThe specimens were 3/4 x 1/2 inch with the clamp distance = 0.60 in. and the strain rate equal to 0.002 in. per min.

^bThe loss complianc., j'' = loss tangent/E'.

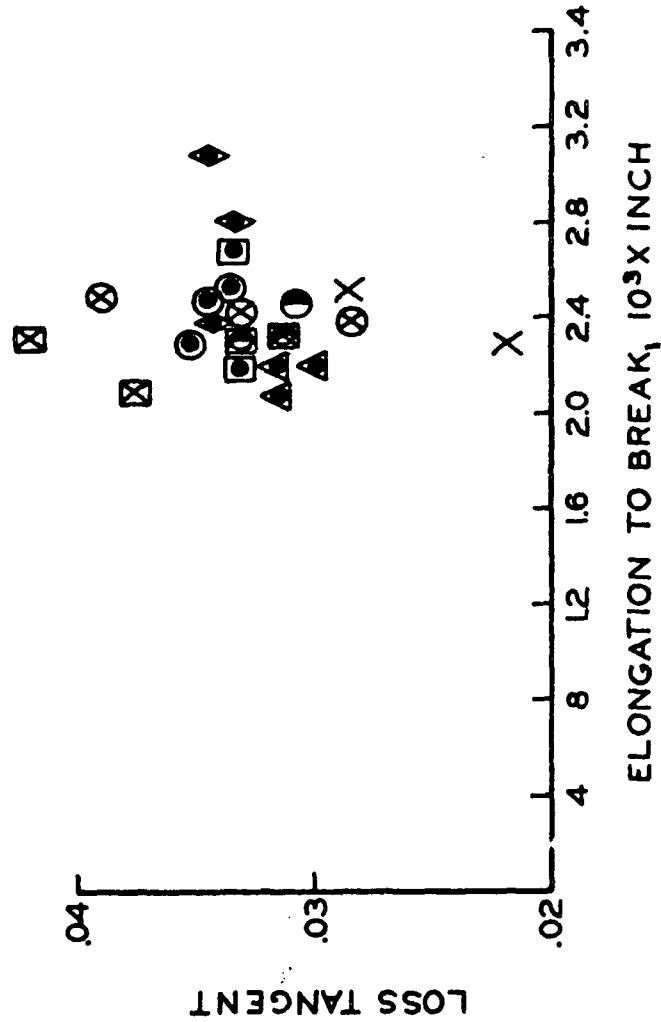


Figure 7. Loss Tangent vs. Elongation to Break for the Second Set of Adhesives (See Table V for Data Identification)

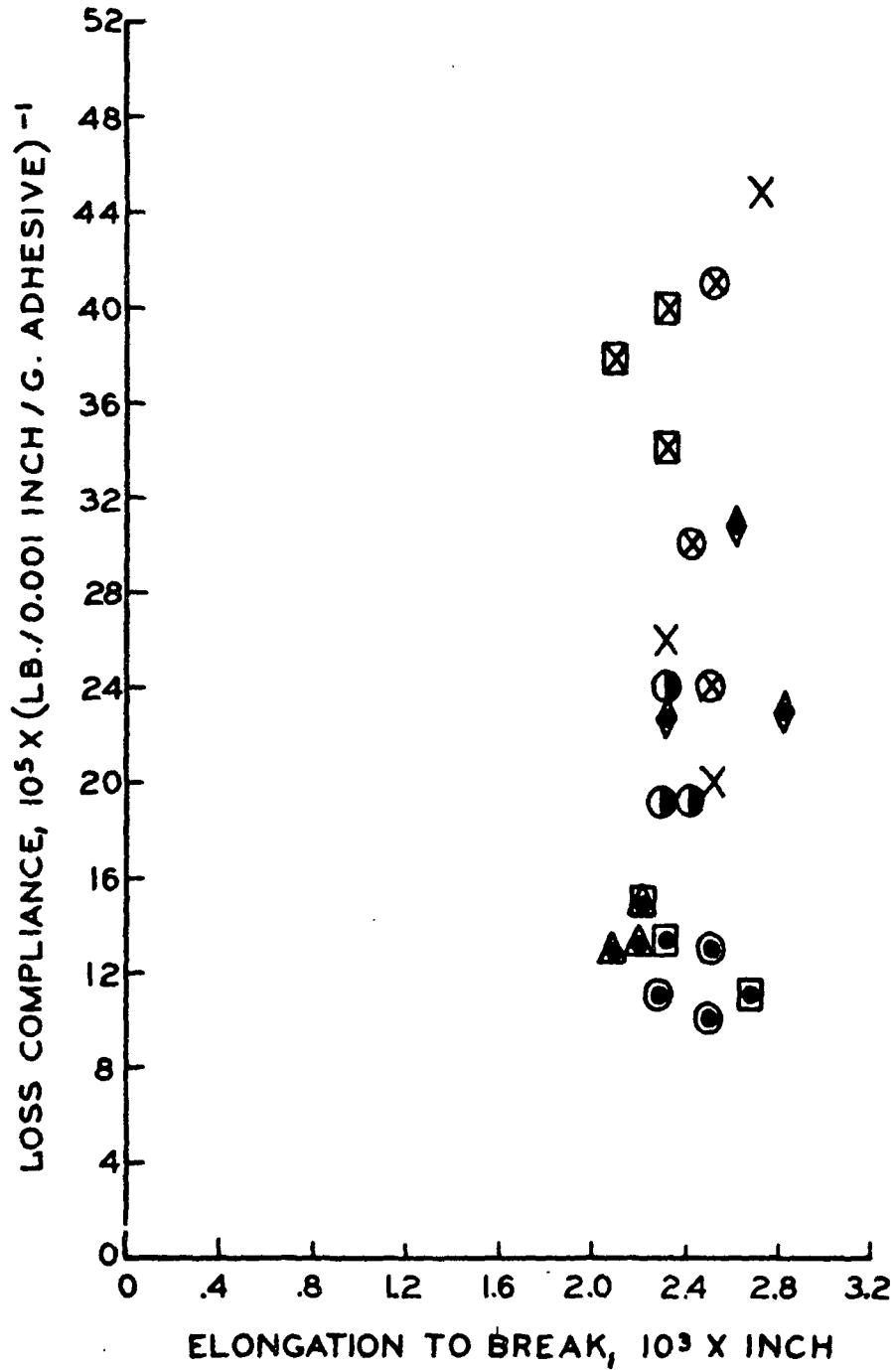


Figure 8. Loss Compliance vs. Elongation for Break for the Second Set of Adhesives (See Table V for Data Identification)

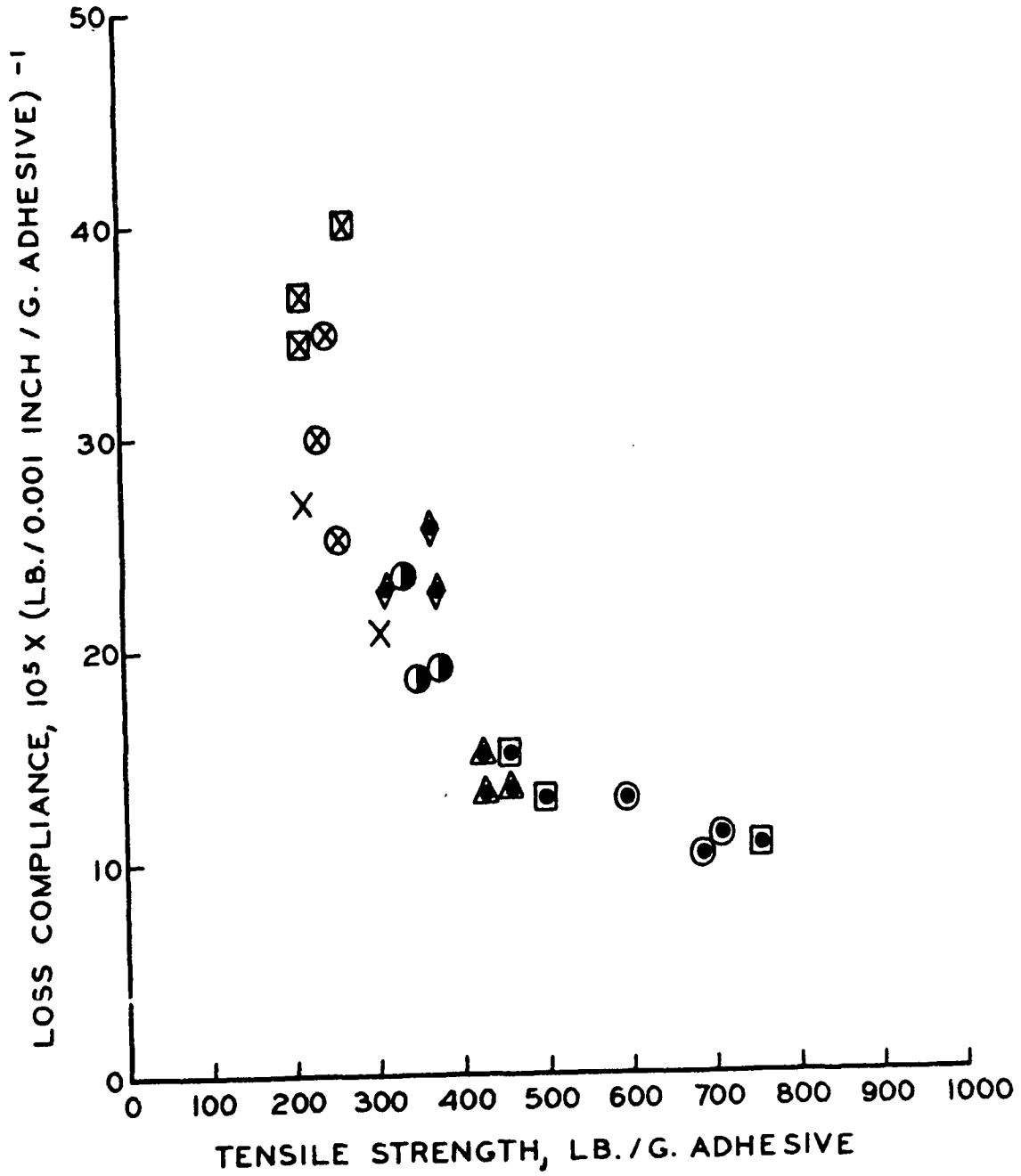


Figure 9. Vibrative Reed Data

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$\cos \theta$ values increase quite fast. So depending on how close γ is to the value of the critical surface tension of the solid, the penetration could increase with reducing γ . It is possible that the reduction in surface tension of the autoclaved R-38 might have increased the penetration, depleting the bonding zone of adhesive, lowering bond strength. When the hexamine is added, the curing heat breaks it down to formaldehyde (cross-linking agent) and ammonia, the latter may be gaseous, driving more adhesive from the bond zone, reducing bond strength. The role of wood meal in the formulated adhesive may be more than a viscosity control agent. It may serve to limit penetration and it may cross-link into the matrix providing a means of stress distribution.

FUTURE WORK

The work to date has shown that the surface tension and mechanical properties of lignosulfonic acid material are not too different than those of good adhesives and, thus, these materials have great potential. In order to develop this potential, the future work should focus on: (1) the role of surface tension as it affects adhesion and penetration; (2) the role of cross-linking as it affects cohesion and insolubilization; (3) the role of wood meal as it affects viscosity, penetration, and stress redistribution; and (4) the role of lignin reactivity as it is affected by electro dialysis, phenol group content, and other factors.

Penetration may be measured by color observation of a tapered grind of the adhesive interface. With this and wetting data the role of surface tension may be more fully evaluated. Cross-linking seems to be necessary to improve viscomechanical behavior and insolubility. More desirable cross-linking agents than hexamine are possible and improving lignin reactivity can be considered. The viscomechanical properties of LSA are not much different than ELSA; it seems possible that electro dialysis may not be necessary. The role of wood meal needs clarifying through a series of control runs.

The program, as outlined in the August, 1969 agreement, would be continued with elements of as many of the above factors introduced as practical within the budget limits.

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LITERATURE CITED

1. Harkins, W. D., and Jordan, J. F., J. Am. Chem. Soc. 52:1751(1939).
2. Progress Report Ten, Project 2421, July 30, 1969.
3. Progress Report Nine, Project 2421, April 25, 1969.
4. Herczeg, A., Forest Prod. J. 15:499-505(Nov., 1965).
5. Goring, D. A. I., Pulp Paper Mag. Can. 68:T372-6(Aug., 1967).
6. Weidner, C. L., and Crocker, G. J., Rubber Chem. and Technol. 33:1323(1960).
7. Landel, R. F., Rheology Bulletin 33:9(1964).


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APPENDIX I

DUPLICATION OF SPECIMEN CUTTING

To check the reproducibility of cutting tabs from disks, six disks were cut into tabs, as shown in Fig. 2. The results are listed in Table VI.

TABLE VI
GLASS FIBER DISK AND TAB WEIGHTS

Disk Weight, g.	Disk, ^a g./cm. ²	Weight of Tabs, g.				Average	Calculated ^a Weight of 2.371 cm. ²	Ratio of Av. Tab Wt. to Calcd. Wt., %
		Tab 1	Tab 2	Tab 3	Tab 4			
0.1597	0.00672	0.0154	0.0151	0.0159	0.0156	0.0155	0.01593	97.3
0.1648	0.00694	0.0153	0.0165	0.0162	0.0157	0.01593	0.01644	96.9
0.1704	0.00717	0.0163	0.0171	0.0160	0.0171	0.01663	0.01699	97.9
0.1682	0.00718	0.0163	0.0161	0.0164	0.0156	0.0161	0.01702	94.6
0.1658	0.00698	0.0160	0.0162	0.0157	0.0154	0.0158	0.01654	95.5
0.1660	0.00699	0.0166	0.0161	0.0162	0.0166	0.01638	0.01657	<u>98.2</u>
								96.9

^aDisk area = $(1.5)^2(3.1417) = 23.7591 \text{ cm.}^2$; tab area = $(0.5 \times 0.735 \text{ inch})(2.54)^2 = 2.371 \text{ cm.}^2$

APPENDIX II

BASIC DATA OF THE TENSILE AND VIBRATING REED MEASUREMENTS

The weights of the disk before adhesive addition, and after adhesive addition and cure, are presented in Table VII. See p. 3 for the complete description of the adhesives in the first set and p. 19, Table III, for those in the second set.

The weight, caliper, and tensile data of the tabs are listed in Table VIII.

The frequency and amplitude data of the reeds from the first adhesive set are given in Table IX. In Table X are listed the resonance frequency (ω_0), band width at $1/\sqrt{2}$ of maximum amplitude ($\Delta\omega$), and the loss tangent of the vibrating reeds.

The original data from the recorder of the Instron Tensile Tester is filed with this report in the Central Files of The Institute of Paper Chemistry.

TABLE VII

DISK AND ADHESIVE WEIGHTS

Adhesive ^a	Disk Identification ^b	Disk Weight, g.		Adhesive Weight, g.
		Before	After	
None	N-6	0.1697	0.1690	(-0.0007)
	N-7	0.1836	0.1831	(-0.0005)
	N-8	0.1771	0.1762	(-0.0009)
	N-9	0.1801	0.1790	(-0.0011)
	N-10	0.1650	0.1642	(-0.0008)
First Set of Adhesives				
R-44 concd.	C-1	0.1699	0.5154	0.3455
	C-2	0.1748	0.5307	0.3559
	C-3	0.1817	0.4562	0.2745
	C-4	0.1674	0.3986	0.2312
	C-5	0.1785	0.5094	0.3309
Phenol-formaldehyde	P-1	0.1620	--	--
	P-2	0.1681	--	--
	P-3	0.1650	0.2460	0.0810
	P-4	0.1622	0.2600	0.0978
	P-5	0.1654	0.2440	0.0786
R-2	66-1	0.1639	0.2356	0.0717
	66-2	0.1623	0.2365	0.0742
	66-3	0.1641	0.2353	0.0712
	66-4	0.1654	0.2288	0.0634
	66-5	0.1642	0.2350	0.0708
R-44	67-6	0.1611	0.2098	0.0487
	67-7	0.1625	0.2031	0.0406
	67-8	0.1616	0.2096	0.0480
	67-9	0.1685	0.2037	0.0352
	67-10	0.1619	0.2009	0.0390
R-44 and glycerin	67-11	0.1653	0.2000	0.0347
	67-12	0.1606	0.1880	0.0274
	67-13	0.1653	0.1936	0.0283
	67-14	0.1608	0.1870	0.0262
	67-15	0.1666	0.1880	0.0214
R-44 and hexamine	67-16	0.1638	0.2128	0.0490
	67-17	0.1623	0.2131	0.0508
	67-18	0.1648	0.2175	0.0527
	67-19	0.1629	0.2098	0.0469
	67-20	0.1641	0.2126	0.0485
R-44, ferric chloride and acrylamide	67-21	0.1623	0.1779	0.0156
	67-22	0.1653	0.1803	0.0150
	67-23	0.1582	0.2104	0.0522
	67-24	0.1643	0.1938	0.0295
	67-25	0.1653	0.2176	0.0523
Methanol-soluble fraction	67-26	0.1634	0.2180	0.0546
	67-27	0.1626	0.2229	0.0603
	67-28	0.1632	0.2220	0.0588
	67-29	0.1646	0.2266	0.0620
	67-30	0.1606	0.2255	0.0649
Methanol-insoluble fraction	67-31	0.1636	0.2331	0.0695
	67-32	0.1650	0.2358	0.0708
	67-33	0.1641	0.2426	0.0785
	67-34	0.1656	0.2443	0.0787
	67-35	0.1621	0.2282	0.0661

See end of table for footnotes.

TABLE VII (Continued)
DISK AND ADHESIVE WEIGHTS

Adhesive ^a	Disk Identification ^b	Disk Weight, g.		Adhesive Weight, g.
		Before	After	
Second Set of Adhesives (Wood Bonding Studies)				
R-38	106-36	0.1740	0.2521	0.0781
	106-37	0.1819	0.2690	0.0871
	106-38	0.1790	0.2627	0.0837
	106-39	0.1692	0.2466	0.0774
	106-40	0.1763	0.2706	0.0943
R-38A (autoclaved)	106-41	0.1675	0.2755	0.1080
	106-42	0.1857	0.2691	0.0834
	106-43	0.1862	0.2830	0.0968
	106-44	0.1676	0.2715	0.1039
	106-45	0.1801	0.2786	0.0985
R-38A X-200	106-46	0.1868	0.3190	0.1322
	106-47	0.1691	0.3051	0.1360
	106-48	0.1765	0.3181	0.1416
	106-49	0.1842	0.3170	0.1328
	106-50	0.1668	0.3129	0.1461
R-38A hexamine X-200	106-51	0.1798	0.3484	0.1686
	106-52	0.1817	0.3769	0.1952
	106-53	0.1831	0.3868	0.2037
	106-54	0.1659	0.3345	0.1686
	106-55	0.1766	0.3844	0.2078
R-38A hexamine glycerin X-200	106-56	0.1817	0.3869	0.2055
	106-57	0.1639	0.3968	0.2329
	106-58	0.1844	0.3990	0.2146
	106-59	0.1646	0.3702	0.2056
	106-60	0.1772	0.4151	0.2379
R-38F (formulated adhesive)	107-61	0.1763	0.5069	0.3306
	107-62	0.1805	0.5315	0.3510
	107-63	0.1682	0.4720	0.3038
	107-64	0.1788	0.4940	0.3152
	107-65	0.1736	0.5223	0.3487
R38-F X-200	107-66	0.1688	0.4966	0.3278
	107-67	0.1810	0.5299	0.3489
	107-68	0.1838	0.4969	0.3131
	107-69	0.1779	0.4755	0.2976
	107-70	0.1668	0.5040	0.3372
R38-F 12-P-6	107-71	0.1822	0.4752	0.2930
	107-72	0.1681	0.4797	0.3116
	107-73	0.1769	0.4999	0.3230
	107-74	0.1837	0.5175	0.3344
	107-75	0.1655	0.4949	0.3294

^aFor complete description of the adhesives see p. 3 for the first adhesive set and p. 19, Table III, for the second set.

^bThe disks are identified by the page number in Notebook 2742 on which they are first described (i.e., 66-1 is disk No. 1 on p. 66 in Notebook 2742).

TABLE VIII
THE WEIGHT, CALIPER, AND TENSILE DATA OF THE TABS

Tab Identification ^a	Tab Weight, 10 ² x g.	Adhesive Weight, 10 ³ x g.	Tensile		Elongation to Break, 10 ³ x in.	Slope		Average ^c Caliper, 10 ³ x in.
			lb.	lb./g. adhesive		lb./0.001 in.	lb./0.001 in./g. adhesive	
Glass mat	1.61	0	(-1.0)	--	0	0	--	9.5
First Set of Adhesives								
R-44 concd.	4.76	0.0315	6.2	197	2.8	3.82	121	16.0
Phenol-formaldehyde	10.49	0.00888	7.1	807	3.2	2.62	295	6.0
66-2-A	2.28	7.00	4.45	636	0.93	2.40	343	6.9
-B	2.10	6.59	4.50	683	1.00	2.30	349	6.0
-C	2.28	7.15	5.79	809	1.26	2.30	322	6.7
-D	2.29	7.15	5.48	763	1.13	2.50	348	6.0
66-3-A	2.06	6.23	4.73	759	1.02	2.40	385	5.3
-B	2.23	6.75	5.16	764	1.14	2.44	361	5.0
-C	2.13	6.45	--	--	--	--	--	5.0
-D	2.45	7.41	5.59	759	1.20	2.55	344	6.1
66-4-A	2.03	5.62	4.02	715	0.92	2.27	404	5.3
-B	2.15	5.96	--	--	--	--	--	5.1
-C	2.20	6.40	4.25	697	1.02	2.43	398	5.4
-D	2.18	6.04	5.02	831	1.14	2.40	347	5.1
67-6-A	2.08	4.83	3.75	776	0.90	1.80	373	8.2
-B	1.98	4.60	3.58	779	0.88	1.90	413	6.8
-C	2.00	4.64	3.78	815	1.11	1.90	409	5.9
-D	2.02	4.69	3.62	772	1.02	1.83	390	6.6
67-7-A	2.02	4.04	3.29	814	0.75	2.20	545	5.5
-B	1.99	3.98	--	--	--	--	--	5.4
-C	1.92	3.84	--	--	--	--	--	5.3
-D	1.94	3.88	2.99	771	0.72	2.25	580	5.3
67-8-A	1.98	4.53	3.29	726	1.10	1.65	364	5.8
-B	2.02	4.63	2.90	626	0.88	1.80	389	5.3
-C	2.11	4.83	4.20	870	1.11	2.00	414	6.9
-D	2.08	4.76	1.80	375	0.50	1.76	370	4.8
67-16-A	2.09	4.81	3.40	707	1.30	1.47	306	9.3
-B	1.99	4.58	3.74	817	1.52	1.48	323	8.8
-C	2.02	4.65	4.30	930	1.60	1.63	351	9.0
-D	2.00	4.60	2.45	533	1.00	1.28	278	9.0
67-17-A	2.09	5.70	4.49	790	1.70	1.70	298	9.0
-B	2.01	4.79	3.79	791	1.30	1.67	349	8.1
-C	2.11	5.03	4.29	853	1.66	1.70	338	9.4
-D	2.05	4.89	3.89	796	1.32	1.70	348	8.7
67-18-A	2.07	5.02	2.94	586	1.11	1.70	339	8.2
-B	2.09	5.06	--	--	--	--	--	--
-C	2.03	4.92	3.30	671	1.30	1.48	301	8.9
-D	2.05	4.97	3.60	724	1.16	1.60	322	9.0
67-26-A	2.01	5.03	1.82	362	2.90 ^d	0.74	147	10.4
-B	1.99	4.98	1.40	281	2.50	0.63	129	9.4
-C	2.25	5.63	2.80	497	3.90	0.84	140	11.8
-D	2.01	5.03	1.52	302	3.10	0.60	119	9.0
67-27-A	2.25	6.87	1.72	250	3.70 ^d	0.70	102	12.0
-B	2.51	7.66	2.42	316	5.60	0.50	65	15.6
-C	2.05	6.26	1.32	211	3.80	0.30	48	11.9
-D	2.00	6.11	1.70	278	4.20	0.48	79	12.7
67-30-A	2.35	6.76	2.05	303	4.80 ^d	0.30	44	13.8
-B	2.08	5.00	1.48	277	1.20	0.27	37	12.0
-C	2.05	5.90	1.61	273	4.50	0.48	81	12.0
-D	1.91	5.50	1.65	300	3.60	0.50	91	11.9
67-32-A	2.21	6.64	5.80	874	1.40	2.44	367	6.9
-B	2.10	6.30	2.60	413	0.72	2.05	325	6.1
-C	2.34	7.03	5.85	832	1.48	2.16	307	9.2
-D	2.03	6.08	4.19	685	1.08	2.07	340	6.7
67-34-A	2.15	6.93	4.98	719	1.23	2.25	325	6.0
-B	2.15	6.93	3.25	468	0.82	1.90	278	6.2
-C	2.22	7.15	4.10	573	0.96	2.28	313	6.1
-D	2.43	7.83	5.95	760	1.58	2.13	272	9.8

See end of table for footnotes.

TABLE VIII (Continued)
THE WEIGHT, CALIPER, AND TENSILE DATA OF THE TABS

Tab Identification ^a	Tab Weight, 10 ² x g.	Adhesive ^b Weight, 10 ³ x g.	Tensile		Elongation to Break, 10 ³ x in.	Slope		Average ^c Caliper, 10 ³ x in.
			lb.	lb./g. adhesive		lb./0.001 in.	lb./0.001 in./g. adhesive	
First Set of Adhesives (Cont'd.)								
67-35-A	2.20	6.37	4.92	772	1.08	2.43	381	6.4
-B	2.20	6.37	5.70	894	1.72	2.60	408	5.3
-C	2.11	6.11	5.38	881	1.32	2.50	409	5.3
-D	2.16	6.26	4.20	671	0.92	2.50	399	4.9
67-11-A	1.98	3.43	4.18	1210	1.28	2.00	583	7.2
-B	2.03	3.52	3.82	1085	1.06	2.20	625	9.0
-C	1.81	3.14	2.82	898	0.88	1.90	541	5.4
-D	1.81	3.14	2.68	854	0.80	2.00	637	4.9
67-12-A	1.56	2.71	2.00	736	0.68	1.70	627	5.5
-B	1.86	2.71	2.22	818	0.87	1.68	620	5.8
-C	1.87	2.72	1.40	515	0.86	1.17	430	6.7
-D	1.81	2.64	2.02	765	0.82	1.60	606	4.8
67-23-A	1.96	4.86	1.20	247	0.68	0.88	181	6.8
-B	2.00	4.96	2.46	496	0.72	1.95	393	5.3
-C	2.36	5.85	4.00	684	1.28	1.65	282	8.9
-D	2.01	4.89	3.00	601	0.80	1.90	389	5.0
67-29-A	2.18	5.24	4.66	889	1.30	1.90	363	7.8
-B	--	--	--	--	--	--	--	--
-C	2.10	5.05	4.55	901	1.41	1.80	356	7.8
-D	--	--	--	--	--	--	--	--
Second Set of Adhesives (Wood Bonding Study)								
106-36-A	2.35	7.3	4.60	630	1.9	2.20	301	--
-B	2.28	7.1	4.35	613	2.0	2.00	282	--
-C	2.25	7.0	5.35	764	2.5	2.20	314	--
-D	2.22	6.9	4.95	718	2.3	2.46	357	--
106-37-A	2.54	8.2	3.85	470	2.7	2.00	244	--
-B	2.44	7.9	3.90	494	2.5	1.87	237	--
-C	2.34	7.6	4.65	612	2.3	2.40	316	--
-D	2.48	7.0	4.50	643	2.6	1.90	271	--
106-38-A	2.49	7.2	4.95	688	2.2	2.62	364	--
-B	2.22	7.1	4.85	683	2.2	2.50	352	--
-C	2.43	7.7	5.30	688	2.6	2.38	309	--
-D	2.39	7.6	5.11	672	2.6	2.25	296	--
106-41-A	2.46	9.6	1.92	200	1.9	1.64	171	--
-B	2.40	9.4	4.68	498	2.3	2.35	250	--
-C	2.56	10.0	4.45	445	2.4	2.13	213	--
-D	2.57	10.1	4.10	406	2.3	2.62	259	--
106-42-A	2.44	7.6	6.28	826	2.9	2.08	274	--
-B	2.62	8.1	5.50	679	2.3	2.48	306	--
-C	2.29	7.1	5.33	751	2.7	2.29	323	--
-D	2.48	7.7	6.62	860	2.7	2.73	355	--
106-43-A	2.66	9.1	4.60	506	2.5	2.18	240	--
-B	2.61	8.9	4.35	489	1.9	2.21	248	--
-C	2.60	8.9	4.25	478	2.2	2.41	271	--
-D	2.53	8.6	4.49	522	2.5	2.29	266	--
106-46-A	3.01	12.0	5.69	474	2.2	2.93	244	--
-B	3.12	12.4	4.59	370	2.0	3.19	257	--
-C	2.99	11.9	4.45	374	2.1	2.65	223	--
-D	3.01	12.0	6.00	500	2.2	2.86	238	--
106-47-A	2.77	12.3	6.65	541	2.7	2.86	233	--
-B	2.70	12.0	6.30	525	2.4	2.73	228	--
-C	3.07	13.7	3.60	263	1.5	2.41	200	--
-D	3.01	13.4	5.35	399	2.1	2.78	207	--
106-48-A	2.94	13.1	6.65	508	2.3	3.00	229	--
-B	3.02	13.4	5.10	381	2.0	2.87	214	--
-C	2.96	13.2	5.69	425	2.1	3.06	232	--
-D	3.06	13.6	6.66	490	2.5	3.05	224	--
106-51-A	3.44	16.6	5.60	337	2.3	2.64	159	--
-B	3.05	14.8	5.88	397	2.3	2.31	156	--
-C	3.13	15.1	5.45	361	2.4	2.29	152	--
-D	3.17	15.3	6.09	398	2.2	2.92	191	--

See end of table for footnotes.

TABLE IX
 FREQUENCY VERSUS AMPLITUDE DATA

Freq., c.p.s.	Amp., cm.	Freq., c.p.s.	Amp., cm.	Freq., c.p.s.	Amp., cm.
Glass Fiber Mat					
11.3	0.1580	10.38	0.1497	10.68	0.1966
11.2	0.1664	9.88	0.0979	10.50	0.1881
11.0	0.1415	9.38	0.0975	10.40	0.1611
10.8	0.1217	10.20	0.1168	10.30	0.1532
10.6	0.1047	10.30	0.1390	10.20	0.1617
10.3	0.1018	10.45	0.1212	10.00	0.1560
11.4	0.1586	10.50	0.1380	9.70	0.1484
11.5	0.1575	10.60	0.1424	9.20	0.1295
11.7	0.1438	10.70	0.1406	10.90	0.2040
12.0	0.1371	10.80	0.1335	11.00	0.1934
12.2	0.1227	10.90	0.1199	11.10	0.1944
12.4	0.1092	11.00	0.1280	11.30	0.1569
12.8	0.1057	11.10	0.1179	11.50	0.1578
		11.50	0.0917	11.80	0.1653
		11.80	0.0984	12.20	0.1223
				12.70	0.1154
Phenol Formaldehyde					
25.3	0.2117	25.8	0.1645	26.8	0.1657
24.8	0.1780	25.3	0.1326	26.5	0.1607
24.3	0.1074	24.8	0.0719	26.2	0.1351
23.8	0.0734	24.3	0.0536	25.9	0.0953
23.3	0.0620	23.8	0.0483	25.6	0.0735
22.8	0.0554	25.0	0.0653	25.0	0.0662
25.3	0.2150	25.7	0.1525	26.7	0.1585
25.8	0.1580	26.0	0.1493	27.0	0.1461
26.3	0.0985	26.3	0.1062	27.3	0.1173
26.8	0.0723	26.6	0.0857	27.6	0.0921
27.3	0.0605	26.9	0.0669	28.0	0.0640
27.8	0.0588	27.5	0.0440	28.5	0.0562
R-44 (Concentrated)					
62.4	0.1356	52.5	0.1110	56.2	0.1748
61.9	0.1527	52.0	0.1029	55.7	0.1761
61.4	0.1367	51.5	0.0836	55.2	0.1386
60.9	0.1283	51.0	0.0681	54.7	0.0981
60.4	0.0830	50.5	0.0710	54.2	0.0736
59.9	0.0791	53.0	0.1047	56.2	0.1755
59.4	0.0679	53.5	0.1079	56.7	0.1522
62.9	0.1366	54.0	0.0950	57.2	0.1171
63.4	0.1171	54.5	0.0889	57.7	0.0978
63.9	0.1010	55.0	0.0870	58.2	0.0891
64.4	0.0820			58.7	0.0661
64.9	0.0880				

TABLE IX (Continued)
FREQUENCY VERSUS AMPLITUDE DATA

Freq. ⁻¹ , 10 ³ x sec.	Amp., cm.	Freq. ⁻¹ , 10 ³ x sec.	Amp., cm.	Freq. ⁻¹ , 10 ³ x sec.	Amp., cm.
R-2					
66-2-1		66-3-2		66-4-1	
38.29	0.1698	45.42	0.1568	45.41	0.1890
38.78	0.1248	45.67	0.1844	45.78	0.1812
39.40	0.0775	46.36	0.1595	46.10	0.1440
39.90	0.0715	46.78	0.1206	46.58	0.1304
40.34	0.0494	47.18	0.1065	46.98	0.1056
38.18	0.1640	48.10	0.0780	48.08	0.0794
37.96	0.1559	49.21	0.0667	45.20	0.1793
37.57	0.1296	45.00	0.1343	44.82	0.1523
37.30	0.1021	44.64	0.1107	44.44	0.1300
36.86	0.0826	44.26	0.0962	44.07	0.1086
36.22	0.0587	43.87	0.0857	43.69	0.0467
35.43	0.0450	43.14	0.0685	42.92	0.0775
R-44					
67-6-2		67-7-1		67-8-2	
44.61	0.1700	44.83	0.1666	43.71	0.1809
44.26	0.1722	45.15	0.1572	43.89	0.1831
43.89	0.1587	45.48	0.1389	44.14	0.1670
43.47	0.1290	45.97	0.1046	44.54	0.1361
43.14	0.1082	46.60	0.0850	45.14	0.1124
42.19	0.0790	48.16	0.0630	46.66	0.0804
44.88	0.1515	44.47	0.1613	43.31	0.1744
45.21	0.1178	44.06	0.1435	42.95	0.1457
45.62	0.1011	43.72	0.1274	42.60	0.1220
46.09	0.0840	43.32	0.1098	42.22	0.1044
46.50	0.0771	42.79	0.0906	41.70	0.0885
47.83	0.0592	41.55	0.0712	40.52	0.0687
R-44 Hexamine					
67-16-2		67-17-1		67-18-1	
29.38	0.1726	29.62	0.1759	32.13	0.1480
29.56	0.1580	29.67	0.1673	32.15	0.1582
29.71	0.1479	30.05	0.1440	32.42	0.1634
30.05	0.1112	30.57	0.1022	32.45	0.1553
31.15	0.0829	31.09	0.0796	32.89	0.1450
31.67	0.0713	31.63	0.0660	32.97	0.1166
29.66	0.1226	29.65	0.1745	32.66	0.0809
29.58	0.1500	29.40	0.1752	34.14	0.0713
29.18	0.1736	28.61	0.1254	32.18	0.1532
28.78	0.1505	28.42	0.0949	31.82	0.1391
28.72	0.1515	27.98	0.0718	31.29	0.1076
28.32	0.1105	27.28	0.0547	30.07	0.0702
27.86	0.0898				

TABLE IX (Continued)
FREQUENCY VERSUS AMPLITUDE DATA

Freq. ⁻¹ , 10 ³ x sec.	Amp., cm.	Freq. ⁻¹ , 10 ³ x sec.	Amp., cm.	Freq. ⁻¹ , 10 ³ x sec.	Amp., cm.
Methanol Soluble					
67-26-2		67-27-1		67-30-2	
27.80	0.1400	31.03	0.1613	26.14	0.1591
27.85	0.1347	31.07	0.1664	26.53	0.1376
27.99	0.1224	31.44	0.1364	26.87	0.1251
28.19	0.1057	31.63	0.1267	27.20	0.1121
29.03	0.0818	32.19	0.0971	27.64	0.0942
29.56	0.0618	32.79	0.0798	29.00	0.0670
27.47	0.1523	30.60	0.1576	26.20	0.1528
27.39	0.1505	30.61	0.1570	25.46	0.0977
27.00	0.1331	30.42	0.1431	25.19	0.0868
26.85	0.1179	30.15	0.1282	26.06	0.1566
26.34	0.0927	29.72	0.1036	25.87	0.1495
26.07	0.0697	28.88	0.0855	25.67	0.1053
Methanol Insoluble					
67-32-1		67-34-1		67-35-1	
33.46	0.1862	34.13	0.2066	33.58	0.1767
33.65	0.1794	34.45	0.1879	33.84	0.1936
33.86	0.1552	34.81	0.1419	34.28	0.1622
34.10	0.1264	35.23	0.1070	34.62	0.1234
34.50	0.1012	38.72	0.0802	35.23	0.0842
35.48	0.0725	36.46	0.0670	36.50	0.0618
33.20	0.1686	34.26	0.2007	33.68	0.1825
32.88	0.1391	33.80	0.1730	33.37	0.1522
32.23	0.1048	33.40	0.1305	33.03	0.1156
31.70	0.0889	33.02	0.1018	32.26	0.0753
31.20	0.0697	32.29	0.0761	30.96	0.0567
R-44 Glycerin					
67-11-1		67-12-1			
28.69	0.1957	48.57	0.1407		
29.10	0.1704	49.07	0.1337		
29.48	0.1229	49.41	0.1104		
29.65	0.1080	50.03	0.0806		
30.46	0.0772	50.56	0.0720		
31.12	0.0706	51.31	0.0591		
28.67	0.1860	48.33	0.1387		
28.46	0.1797	47.83	0.1159		
28.01	0.1171	47.13	0.0904		
28.25	0.1524	46.49	0.0787		
27.62	0.1004	45.56	0.0613		
26.96	0.0723	47.43	0.1068		
R-44 FeCl ₃ -Acrylamide					
67-23-1		67-25-1			
36.32	0.1684	33.15	0.1523		
36.83	0.1435	33.34	0.1503		
37.13	0.1166	33.50	0.1239		
37.39	0.0972	33.75	0.1078		
37.79	0.0774	34.28	0.0754		
39.08	0.0597	35.04	0.0578		
36.11	0.1526	32.76	0.1595		
35.82	0.1309	32.59	0.1344		
35.45	0.1046	32.30	0.1142		
35.08	0.0805	31.94	0.0870		
34.60	0.0703	32.37	0.0739		
33.31	0.0539	30.33	0.0443		

TABLE X

VIBRATING REED DATA

Reed Identification ^a	Maximum Amplitude, cm.	Resonance Frequency, ω_0 , c.p.s.	Band Width, $\Delta\omega$, c.p.s.	Loss Tangent, $\Delta\omega/\omega_0$	Average Caliper, $10^3 \times$ in.
Glass fiber mat	0.0530	10.4	0.53	0.051	9.8
	0.0810	10.9	0.80	0.073	
	0.0640	11.2	0.62	0.055	
First Set of Adhesives					
R-44 concentrated	0.0360	52.5	1.77	0.0337	16.0
	0.0793	61.9	1.97	0.0318	
	0.1050	56.0	1.56	0.0278	
Phenol formaldehyde	0.1580	25.3	1.01	0.0399	6.0
	0.1185	25.8	0.94	0.0365	
	0.1048	26.8	0.93	0.0348	
66-2-1	0.1187	26.116	0.699	0.0268	6.4
66-3-2	0.1187	21.896	0.561	0.0256	5.4
66-4-1	0.1087	22.022	0.533	0.0242	5.2
67-6-2	0.1249	22.584	0.751	0.0332	6.9
67-7-1	0.1074	22.308	0.826	0.0370	5.4
67-8-2	0.0978	22.784	0.651	0.0286	5.7
67-16-2	0.1138	34.270	1.379	0.0401	9.0
67-17-1	0.1051	33.761	1.283	0.0380	8.8
67-18-1	0.0971	30.845	1.087	0.0353	8.7
67-26-2	0.0950	36.403	1.397	0.0384	10.2
67-27-1	0.0889	32.185	1.071	0.0333	13.1
67-30-2	0.1064	38.256	1.478	0.0385	12.5
67-32-1	0.1125	29.886	0.724	0.0242	7.2
67-34-1	0.1444	29.300	0.785	0.0268	7.0
67-35-1	0.1296	29.551	0.817	0.0277	5.5
67-11-1	0.1260	34.855	1.081	0.0310	6.6
67-12-1	0.0826	20.589	0.626	0.0304	5.7
67-23-1	0.1052	27.533	0.750	0.0272	6.5
67-25-1	0.0956	30.525	0.832	0.0273	7.8
Second Set of Adhesives (Wood Bonding Studies)					
106-36-1	--	29.160	1.028	0.0352	7.5
106-37-1	--	28.689	1.067	0.0363	9.0
106-37-2	--	30.983	0.994	0.0321	9.2
106-38-1	--	28.686	0.971	0.0338	7.8
106-38-2	--	32.631	1.067	0.0327	8.0
106-41-1	--	32.131	1.105	0.0344	9.0
106-41-2	--	32.521	1.054	0.0324	9.2
106-42-1	--	37.443	1.237	0.0330	9.4
106-42-2	--	37.483	1.264	0.0337	9.7
106-43-1	--	32.343	1.031	0.0319	8.9
106-43-2	--	31.949	1.080	0.0338	9.0

TABLE X (Continued)

VIBRATING REED DATA

Reed Identification ^a	Maximum Amplitude, cm.	Resonance Frequency, ω_0 , c.p.s.	Band Width, $\Delta\omega$, c.p.s.	Loss Tangent, $\Delta\omega/\omega_0$	Average Caliper, $10^3 \times \text{in.}$
Second Set of Adhesives (Wood Bonding Studies) (Cont'd.)					
106-46-1	--	33.192	1.051	0.0317	7.8
106-46-2	--	33.196	1.080	0.0310	7.7
106-47-1	--	29.628	0.944	0.0319	8.0
106-47-2	--	28.458	0.851	0.0311	7.8
106-48-2	--	33.141	0.984	0.0297	8.7
106-51-1	--	34.180	1.036	0.0303	8.6
106-51-2	--	35.876	1.178	0.0328	9.2
106-52-1	--	39.236	1.222	0.0312	10.5
106-55-1	--	34.595	1.190	0.0344	10.2
106-55-2	--	39.415	1.258	0.0318	10.7
106-57-1	--	39.839	1.346	0.0338	10.5
106-57-2	--	38.238	1.334	0.0349	10.8
106-58-1	--	40.383	1.327	0.0329	10.2
106-58-2	--	38.886	1.350	0.0347	10.4
106-59-1	--	39.041	1.356	0.0348	10.0
106-59-2	--	38.108	1.205	0.0332	10.0
107-61-1	--	33.783	1.404	0.0416	9.2
107-63-1	--	41.501	1.175	0.0283	9.3
107-65-2	--	45.251	0.986	0.0218	11.5
107-66-1	--	48.716	1.807	0.0375	11.4
107-67-1	--	37.393	1.730	0.0463	10.5
107-67-2	--	39.760	1.473	0.0376	10.6
107-70-1	--	45.045	1.232	0.0273	10.5
107-70-2	--	42.054	1.506	0.0358	10.7
107-71-1	--	29.422	1.141	0.0388	10.8
107-73-1	--	43.497	1.299	0.0299	10.6
107-73-2	--	41.329	1.506	0.0365	10.5
107-75-1	--	51.374	1.430	0.0278	11.2
107-75-2	--	50.156	1.428	0.0285	11.3

^aThe reed is identified by the disk (see Table VII) and the location (see Fig. 2).