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UNIVERSITY OF LOUISVILLE

**I IMPREGNATION AND WOOD PLASTICS
II INVESTIGATIONS IN MARINE GLUING**

A Thesis

**Submitted to the Faculty
of the Graduate School
of the University of Louisville
in Partial Fulfillment
of the Requirements
for the Degree of**

MASTER OF CHEMICAL ENGINEERING

Department of Chemical Engineering

By

Charles F. Seare

1944

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I IMPREGNATION AND WOOD PLASTICS**II INVESTIGATIONS IN MARINE GLUING****Charles F. Soren****Approved by the Examining Committee;****Director** Gordon C. Williams

R. C. Ernst

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W. R. Barnes

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October 1944

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ABSTRACT

This thesis presents results and methods for the production of an artificially grained, hard surfaced material by surface impregnation of wood. The method involves the use of a bladed roller to groove the surface, followed by the impregnation of the surface with a resin and a subsequent cure under pressure. General characteristics are indicated.

The optimum production conditions are given for a plastic to be made from sawdust without the addition of any resin. The procedure involves a controlled moisture content of the sawdust without the use of any treatment previous to the final curing under pressure. Some properties of the plastic are given.

A method is reported for the production of a dense hard material from wood without impregnation. The method is based on the property of the lignin present in the wood to act as a binder when the wood is pressed into a new form. General characteristics are presented.

A method is shown for the determination of degree of cure for phenolic resins by the use of a standard acetone extraction. The optimum curing conditions of several low temperature phenolic resin glues are presented as determined by the acetone extraction method.

The thesis presents the viscosity characteristics of mixed phenolic Cascophen LF-67 adhesives. The viscosity correlations are based on the flow of the material through an orifice.

The plasticity of Cascophen LF-67 resin glue when used with H-18 catalyst is presented. The properties of the glue are determined as affected by temperature.

SECTION I

IMPREGNATION AND WOOD PLASTICS

INTRODUCTION

Commercial processes are available for the production of plastics and impregnated woods which have hardness and other useful properties. These materials have uniform characteristics throughout. A product with these properties on the surface and the properties of untreated wood beneath the surface would make it possible to use wood screws for assembly into furniture. The aim of this research is to produce a substance which has these desirable properties by utilizing a cheap, soft wood as the raw material and impregnating the surface.

A related problem in the wood industry is the utilization of sawdust, chips, and shavings. These substances, as too abundant waste materials, have been used as fuel. Uses of sawdust and other wood waste forms have been the subject of investigation for many years for two reasons. First, the elimination of an undesirable waste material is accomplished. Second, the utilization of this waste material fulfills a useful purpose and results in financial gain. The use of a process which involves the need for costly equipment or complicated procedures must be avoided to attain an economically feasible product. Commercial plastics have been made from wood wastes by use of processes which require extensive treatment of the waste material before it is in a form usable for producing articles of plastic. The purpose of this research is to obtain a method that is simple and direct for the production of such a material from sawdust and other waste wood forms without the use of any resin.

HISTORICAL

There has been considerable experimentation on impregnation of wood but the production of an artificially grained, hard surfaced material by surface impregnation is a new idea. There are several properties in addition to hardness which are important in surface treated wood, wood shrinkage and swelling, and the amount of penetration of the resin.

Stamm and Seborg (14) discuss the effect of various resins on the volume changes of wood. The phenol resins seemed the best impregnating resin for minimizing the tendency for the wood to shrink and swell with changes in moisture content.

Stamm and Seborg (16, 13, 15) discuss the effect of various resins on the volume changes of wood and present results obtained when using resin treated wood in forming plywood.

Erickson, Schmitz, and Gortner (1) have obtained data on the permeability of various woods, and they show that white oak heartwood is one of the least permeable woods.

Nelson (3) points out that there is a greater rate of penetration in the longitudinal direction, which is the direction of the length of the tree trunk. This is found because of the smaller number of cell walls necessary to penetrate when compared with the radial and tangential penetration.

Various methods have been devised for producing a plastic from sawdust or other wood wastes. All of these use the property of lignin in wood to act as a binder or to comprise most of the finished product.

Howard and Sandborn (2) in 1937 patented a process for making a molded, vitreous like, highly-dense product which was very dark to black in color. The process consisted of applying high pressure steam to ligno-cellulose material in a state of coarse subdivision and discharging from the steam chamber into a region of lower pressure causing the material to be explosively disintegrated into tough fiber and solid fines. The fines were separated from the coarse fiber and the moisture content adjusted to about 1 to 11%. The fines plasticized by the water were molded into the product under heat and pressure.

Mason, Boehm, and Koonce (4) produced a molding material by steaming ligno-cellulose under pressure at a temperature above the critical temperature of the material. These conditions were held for a period of time sufficient to alter the fiber structure to a state of plastic flowability for hot pressure molding. In 1937 the first lignin plastic in sheet form for laminating was made by the Masonite Corporation, Laurel, Miss., on the basis of this procedure.

Sherrard (9) devised a method of aniline-hydrolysis of sawdust to produce a molding compound. The sawdust was cooked in a rotating digester with aniline and water, drained free of acid liquor and washed with water until neutral to litmus. It was dried, ground,

and plasticized with furfural to produce a molding powder.

A molding compound was produced by Sherrard (16) using an acid hydrolysis method. The sawdust was cooked in a rotating digester with sulfuric acid and water, drained free of acid-sugar liquor, and washed with water until neutral to litmus. The washed residue was dried, ground, and hot pressed with water alone or with additions of aniline and furfural plasticizer.

Olsen and Plow (6) produced a thermosetting molding compound by mixing a finely divided lignin cellulose material with several times as many parts of dilute sulfuric acid and heating the mixture under a pressure of about 80 to 1000 lbs/in² at a temperature of about 185° C to about 200° C for a period from 2 min. to about 8 min. The material was washed, dried and an organic plasticizer added. The molding compound was capable of being molded to the desired form when heated to between about 140° C and about 200° C and subjected to pressures from about 2000 lbs. to about 7500 lbs/in² in a mold.

A method employed by Scherger (8) produced a product suitable for hot molding under pressure. The method involved depositing on a thermoplastic ligno-cellulose material, such as a woodflour mixture sufficient lignin derived from a solution of lignin in an organic solvent to increase the plasticity of the material being hot molded.

Although most of the work on compressed woods involved the use of impregnating resins, there has been some work which did not use such treatment.

A. S. Olsen (6) produced a compressed wood product using a bath of hot linseed oil containing sodium chloride. The wood was immersed in the bath until saturated and pressed with simultaneous application of heat and pressure. The pressure could be applied longitudinally or transversely of the grain of the wood. The wood was allowed to cool before releasing the pressure and removing from the bath.

Stamm and Seborg (17) give data on treatment of heartwood and sapwood of hickory. Results were obtained using different moisture contents, curing temperatures and curing pressures. The results are presented as a function of the fraction of original thickness after pressing and as percent retained compression upon treatment with pre-compression humidities.

THEORETICAL

Normal wood structure is made up of minute cell groupings, consisting of cavities within and between the cells and the spongy or porous cell walls. The Resinous Products & Chemical Co. (7) present the theory that the most effective type of impregnation requires that the impregant penetrate these spongy cell walls and thus prevent any appreciable shrinking and swelling of the individual cells. This in turn insures dimensional stability to the aggregate of impregnated wood cells, or to the entire piece of wood. The penetration of these cell walls requires a resin impregant characterized by small molecular size, in which the condensation has been arrested at an early stage. It is also important to select a resin impregant that is acceptable to the cellulose of the wood and one that will penetrate as quickly as possible and diffuse with reasonable uniformity throughout the wood structure.

Simmons and Ellis (12) give the structure of wood as consisting of cellulose, lignin, hemicellulose, and sap, the sap containing resins and water soluble substances. The average lignin content of wood is believed to consist of α -lignin about 64% and β -lignin about 36%. Lignin polymerizes to form a chain-like structure. Usually, however, the lignin serves as a binder as it occurs naturally in wood. The lignin bonds in woody raw material are broken and the fibrous material in the wood is bonded into its new form.

Sherrard, Beglinger and Hohf (11) state that the best pressing temperature for lignin is so near the point where it begins

to decompose with heat that it is necessary to use plasticizers to obtain the best results. Such plasticizers improve the plastic properties of the lignin and the water resistance of the final product. Such a pressed product from plasticized lignin alone, although very satisfactory in water resistance, is too brittle for many uses. It needs a "filler" for improving its strength properties. This last situation is rather fortunate than otherwise, since complete removal of all the cellulosic material from wood would be an expensive process and by a comparatively simple treatment enough cellulose can be removed to render the lignin plastic, at the same time leaving enough cellulose to serve as a filler.

EXPERIMENTAL

SURFACE IMPREGNATION

Surface impregnation is a new wood treating procedure devised for the purpose of producing hard or grained surfaces on a soft wood base by the use of resin impregnates. The work was undertaken in this thesis in an attempt to find a new, quick and adaptable procedure.

APPARATUS:

The main piece of apparatus used was a hydraulic press with thermostatically operated, electrically heated platens. This press, made by the Charles E. Francis Company, is hand operated with platens one foot square and a piston area of 80 square inches. The press has a pressure gauge reading from 0 to 2000 lbs. per square inch hydraulic pressure and is graduated in 40 lb. increments. It is possible to obtain up to 80 tons total load on this press.

A hand roller with circular blades was used to score the surface of the blocks of wood. The blades were approximately $7/64$ " apart. These blades were of hardened saw steel and were ground to a fine edge.

PROCEDURE:

Blocks of wood were wetted on the surface and the samples were then scored with the roller to a depth of about $1/16$ ". None of the wood was removed, but the grain was merely separated to allow greater surface for absorption and deeper penetration of the resin solution. This scoring and subsequent impregnation gave an artificial grain to the wood.

Separate scored blocks were soaked for various lengths of time in a 30% solids resin solution but were immersed only to a depth of about 1/4". The surface impregnated blocks were then dried in preparation for curing or polymerization of the resin in the wood. The curing of the sample was accomplished by heating in a hydraulic press at different pressures with a plate temperature of 300° F. on the treated side of the sample. The plate temperature on the untreated side of the sample was allowed to remain at room temperature.

DATA AND RESULTS:

The experimental work was performed using two different phenolic-aldehyde resins - Amberlite PA-25 made by Resinous Products and Chemical Co., Incorporated, and Durez 6666 made by Durez Plastics and Chemicals, Incorporated. Solutions of each resin were 30% solids.

Preliminary experiments indicated that wetting of the surface of the wood before serration facilitated the performance of this operation and resulted in greater resin penetration. It was found that the surface of the samples became ridged if the pressure was released while the sample was still at curing temperature and optimum conditions necessitated cooling to about 200° F. in the press under pressure.

Table I presents results of a series of samples soaked in Amberlite PA-25 solution, air-dried overnight, cured for 1/2 hour at 300° F. and cooled for 1/2 hour in press under pressure.

Table II presents results of a series of samples soaked in Durez 6666 solution, air-dried for a period over 24 hours and dried

TABLE I PROPERTIES OF AMHERLITE PB-22 SURFACE IMPREGNATED WOOD

Soaking Time (Hrs.)	Pressure P.S.I.	Thickness	Penetration	Properties Appearance	Hardness
3	800	15/32"	fair	grooved	fair
3	1000	1/4"	fair	slightly ridged	very good
4	800	15/32"	fair	grooved	fair
4	1000	1/4"	fair	slightly ridged	very good
6-1/2	800	15/32"	excellent	good	very good
6-1/2	1000	1/4"	good	slightly ridged	very good
7-1/2	800	15/32"	very good	good	very good
7-1/2	1000	1/4"	good	badly ridged	very good
* 20	800	15/32"	good	excellent	very good
20	1000	1/4"	good	excellent	excellent

* This sample had no cooling period in press.

TABLE II PROPERTIES OF JUNE 6886 SURFACE IMPREGNATED WOOD

Soaking Time (Hrs.)	Pressure P.S.I.	Thickness	Penetration	Properties Appearance	Hardness
3	1000	12/32"	poor	fair	fair
4	1000	12/32"	good	good	very good
6-1/2	1000	12/32"	fair	fair	good
7-1/2	1000	12/32"	fair	good	very good
* 22	300	12/32"	fair	fair	poor
* 22	1000	8/16"	good	excellent	excellent

* These two samples had no cooling period in press.

at 140° F. for 24 hours. They were cured for 1/2 hour at 300° F. and allowed to cool to 120° F. under pressure.

CONCLUSIONS:

Amberlite PB-25 surface impregnated wood with a 20 hour soaking period had a surface which possessed excellent appearance and hardness with good penetration when cured at 300° F. with 1000 P.S.I. surface pressure. With the same soaking period, and 200 P.S.I. pressure the surface was only slightly softer and the base wood was less dense. The soaking periods of 7 1/2 hours or less tend to have better surface properties at low (200 P.S.I.) curing pressures than at high (1000 P.S.I.).

Durez 6686 surface impregnated wood with a 24 hour soaking period had a surface which possessed excellent appearance and hardness with good penetration when cured at 300° F. with 1000 P.S.I. With the same soaking period and 200 P.S.I. pressure the surface properties were only poor to fair. Satisfactory results can be obtained with a 1000 P.S.I. cure using a soaking period as short as 4 hours.

Satisfactory results can be obtained with a 200 P.S.I. cure using a soaking period as short as 6 1/2 hours; although the finished surface is less hard.

WOOD PLASTIC FROM SAWDUST

Sawdust represents a wasted portion of lumber which is in greater proportion as the size of sections decrease. Gamble Bros., Inc., being a dimension lumber concern, produces an extra large proportion of this waste. This section of the project for utilization of the sawdust in a low grade plastic was instigated in an attempt to help solve the problem.

APPARATUS:

A hydraulic press made by the Adolph I. Buehler Company with accessory molding equipment was used for making small pellets 1 inch in diameter from the sawdust. The accessory equipment included a heating coil which surrounded a cylinder with a ground plug for the bottom and a plunger with a thermometer well for the top.

Some of the samples were prepared using a hydraulic press made by the Charles E. Francis Company. (See Page 12). A mold used with this press produces 3 test blocks each $5" \times \frac{1}{2}" \times \frac{1}{2}"$. The area of the plunger of the molding device was 7.5 sq. in. A cross-section of the mold showing position of samples is shown in Fig. 1. Cubes cut from test blocks made in this mold were tested under specific humidity conditions. The equipment consisted of sealed quart jars containing various solutions maintained at constant temperature in electric ovens. Each jar was provided with a bent glass rod frame which supported a cone of wire screen containing the cubes of plastic.

A constant temperature bath was used to run soaking tests on the sawdust plastic.

FIG. 1 CROSS SECTION OF CURING MOLD
FOR PLASTIC TEST BLOCKS

In all testing of samples for hardness a standard Rockwell Hardness Testing Machine was used with a $\frac{1}{8}$ " steel ball, a 50 kg. major load, and readings made on red scale.

All weighings made for water absorption were made on a standard analytical balance.

Measurements of cubes of sandust plastic were made with vernier calipers and of the pellets of sandust plastic with a 1 inch micrometer.

PROCEDURE:

The various moisture content sandusts were prepared by mixing calculated quantities of moist and dry sandusts of known composition. Duplicate samples for moisture content were taken before and after the preparation of the series of sandust plastic samples.

It was found in preliminary experiments that the only way to obtain a uniform structure with desirable properties was to rinse the mold with water. A 7g sample of sandust was weighed and poured into the mold, the plunger put in the top, and the heating coil placed around it. The entire mold and heating coil were placed in the Buchler Press, the pressure applied, and the heating coil started. The heating coil was turned off at the proper time to give a specified maximum curing temperature and the sample was kept under pressure until a temperature below 200° F had been reached.

The sandust plastic samples were sanded on the top and bottom in preparation for testing. Each sample was measured with a

micrometer, weighed on an analytical balance, tested for hardness on a Standard Rockwell Hardness Testing Machine (see page 17), and placed in a $\frac{1}{2}$ gallon jar of water in a constant temperature bath at 25° C. Readings of thickness and weight were taken at intervals until the samples reached a maximum volume at the end of 3 weeks of soaking. The soaked samples were allowed to air-dry at room temperature for 4 days to reach equilibrium. The dried samples were reweighed, measured, and tested for hardness.

In the production of test blocks from the large press the platens were set at the desired temperature, the mandrel placed in the rinsed mold, the mold placed in the press, and the pressure applied. Two thermometers placed in the mold gave checks on the temperature. As soon as the mold reached the desired temperature cooling water was run through coils in the mold before releasing pressure. Cubes were sawed from some test blocks, sanded, and tested under various humidity and temperature conditions.

For Rockwell Hardness determinations a $\frac{1}{2}$ inch ball penetrator, a minor load of 10 kg. and a major load of 80 kg. was used. This combination is known as the R scale. The major load was removed within 7 to 8 seconds after the handle had been tripped. The readings were taken on the red scale and were recorded as follows: a count was taken of the number of times the needle passed through zero on the red scale on the application of the major load. From this was

subtracted the number of times the needle passed through zero upon the removal of this load. If the difference was zero, the reading was recorded as over 100. If the difference was 1, the reading was recorded as 0 to 100. If the difference was 2, the reading was recorded as negative.

DATA AND RESULTS:

It was found after much preliminary experimentation that sandcast with a range roughly from 11 to 18% moisture could be fused partially by compression at 2000 P.S.I. and heating for 10 minutes at temperatures from 305° to 335° F. The bottom edge of the samples had a tendency to remain unfused because of the rise of moisture from the extreme edges. There was a general trend for the hardness to decrease as the moisture content and the curing temperature rose. The light unfused ring on the bottom edge of the samples was eliminated by rinsing the mold with water before use. This moisture around the lower edge replaced the moisture which migrates toward the top of the sample.

Fig. 2 presents the effect of curing pressure on the hardness of the sandcast plastic. The hardness at 2000 P.S.I. is essentially the same as the hardness at 3000 P.S.I., but below 2000 P.S.I. the hardness drops off rapidly. The structure of the sample cured at 2000 P.S.I. was uniform, the one cured at 3500 P.S.I. had one unfused spot, and as the pressure decreased further the

FIG 2 HARDNESS OF PLASTIC FROM 12.7% MOISTURE SAWDUST
CURING TEMPERATURE, 330°F TIME, 10 MINUTES

unfused portion increased to a completely unfused structure at 500 P.S.I.

In the use of the large press to make sample blocks, it was found impossible to get all three blocks of the same composition. The mold is so constructed (see Fig. 1) that the moisture rises from the bottom block into the two upper blocks leaving the bottom one unfused. Any molding should be as thin as possible in the vertical direction to eliminate the effect of this characteristic of the sandust plastic.

Humidity tests on both a dark colored well fused sample and a light less-fused sample are presented in Tables III and IV. The 30% relative humidity at both 70° F and 130° F was produced using a $\text{HgCl}_2\text{-CH}_2\text{O}$ solution in the sealed jars, the 70% relative humidity using a NaCl solution, and the 100% relative humidity using water.

The data show that even for a poor grade of sandust plastic there is no appreciable change up to a 30% relative humidity at temperatures of 70° F and 130° F.

Data on the final set of samples are presented in Table V.

The graph of Rockwell R Hardness is plotted versus the curing temperature for different moisture contents in Fig. 3. From Fig. 3 it is evident that the hardness goes down as the initial moisture content of the sandust goes up regardless of curing

TABLE III PROPERTIES OF FULLY FUSED SANDUST PLASTIC-11 DAY EXPOSURE

<u>Density</u> <u>g/cc</u>	<u>Rockwell</u> <u>R</u> <u>Hardness</u>	<u>Testing Atmosphere</u> <u>Temp. - °F.</u>	<u>% R.H.</u>	<u>% Vol.</u> <u>Increase</u>	<u>% Wt.</u> <u>Increase</u>
1.24	86	70° F	30	- 7.3	- 0.40
1.25	85	70	70	- 4.2	2.49
1.28	86	70	100	5.1	12.14
1.29	85	120	30	- 4.8	1.43
1.26	81	120	70	2.9	11.80
1.24	86	120	100	10.9	14.78

TABLE IV PROPERTIES OF INCOMPLETELY FUSED SANDUST PLASTIC -
11 DAY EXPOSURE

<u>Density</u> <u>g/cc</u>	<u>Rockwell</u> <u>R</u> <u>Hardness</u>	<u>Testing Atmosphere</u> <u>Temp. - °F.</u>	<u>% R.H.</u>	<u>% Vol.</u> <u>Increase</u>	<u>% Wt.</u> <u>Increase</u>
1.29	87	70° F	30	- 2.6	- 0.22
1.32	88	70	70	- 1.6	2.63
1.31	87	70	100	9.9	13.63
1.31	89	120	30	- 6.2	1.80
1.30	88	120	70	16.1	12.70
1.28	88	120	100	40.9	25.80

**TABLE V EFFECT OF INITIAL MOISTURE CONTENT AND CURING TEMPERATURE
ON THE PROPERTIES OF SANDYST PLASTIC**

<u>% Moisture</u>	<u>Curing Temp. ° F.</u>	<u>Density</u>	<u>Hardness R "R"</u>	<u>After 3 Weeks Water Soak</u>			<u>R "R"</u>	<u>Density</u>
				<u>% Vol. O₂</u>	<u>% Wt. O₂</u>	<u>% H₂O</u>		
11.2	270	1.257	90	10.07	11.92	5.50	69	1.230
12.2	270	1.267	96	10.09	11.76	5.62	74	1.232
13.2	270	1.255	89	10.05	12.22	5.74	65	1.236
14.7	270	1.212	74	8.29	11.57	6.97	43	1.218
16.1	270	1.207	67	9.02	12.20	6.20	29	1.208
16.4	270	1.234	64	10.50	14.28	6.67	28	1.194
11.2	305	1.277	96	11.02	12.08	4.94	78	1.208
12.2	305	1.280	93	10.26	12.12	5.67	64	1.206
13.2	305	1.244	87	10.25	12.62	4.94	55	1.203
14.7	305	1.220	65	9.58	12.92	6.54	55	1.224
16.1	305	1.206	70	10.48	13.08	7.29	55	1.214
16.4	305	1.223	70	9.94	14.28	7.54	40	1.202
11.2	355	1.274	109	10.41	12.42	4.90	84	1.215
12.2	355	1.272	102	10.95	12.90	4.46	81	1.220
13.2	355	1.262	100	10.65	12.20	5.23	78	1.224

TABLE V EFFECT OF INITIAL MOISTURE CONTENT AND CURING TEMPERATURE
ON THE PROPERTIES OF SANDUST PLASTIC (Com'g.)

<u>% Moisture</u>	<u>Curing Temp. ° F.</u>	<u>Density</u>	<u>Hardness R "H"</u>	<u>After 8 Weeks Water Soak</u>			<u>R "H"</u>	<u>Density</u>
				<u>% Vol.C.</u>	<u>% Wt.C.</u>	<u>% Extr.</u>		
14.7	355	1.345	95	9.49	11.48	6.54	77	1.288
16.1	355	1.310	77	10.42	13.83	6.91	61	1.232
16.4	355	1.297	79	10.35	15.20	6.00	52	1.233
11.2	345	1.378	110	14.23	15.65	3.61	80	1.312
12.2	345	1.378	106	17.09	17.98	3.56	71	1.292
13.3	345	1.362	102	11.00	15.02	4.43	82	1.303
14.7	345	1.342	92	11.40	14.88	4.78	69	1.284
16.1	345	1.302	78	10.82	15.44	5.55	52	1.246
16.4	345	1.287	78	11.68	17.78	5.02	48	1.227
11.2	335	1.386	110	16.22	18.69	4.14	65	1.287
12.2	335	1.378	107	15.27	16.56	4.44	76	1.303
13.3	335	1.366	100	13.18	14.87	4.99	78	1.307
14.7	335	1.339	90	11.03	14.82	5.24	72	1.282
16.1	335	1.307	83	11.47	16.83	5.71	64	1.246
16.4	335	1.313	80	11.83	17.24	5.68	59	1.257

temperature. Also, the hardness goes down as the curing temperature rises regardless of moisture content of the sandust.

The graph of Density of plastic is plotted versus the curing temperature in Fig. 4. From Fig. 4 it is seen that the density goes down as the moisture content goes up regardless of curing temperature. The density only drops slightly with increasing curing temperature at the same rate for all moisture contents.

Fig. 5 shows the % volume increase in 3 weeks of soaking. The minimum volume increase is in the range 14% - 16% moisture sandust for all curing temperatures. The 14.7% moisture sandust samples had the lowest volume increase with the 16.1% and the 16.4% moisture sandust the next lowest respectively. The increase in volume becomes smaller as the curing temperature increases.

Fig. 6 shows the % weight increase in 3 weeks of soaking. The minimum weight increase is in the range 13% - 15% moisture sandust. The increase in weight becomes smaller as the curing temperature increases.

Fig. 7 gives the density of the soaked samples after being dried 4 days at room temperature and humidity. The general trend for all moisture content sandust except the 16.1 and the 16.4% moisture is to have the maximum density in those samples which were cured at 350° F. The trend is still to have the greater density as the moisture content decreases.

SAWDUST MOISTURE CONTENT

- 11.2 % X 14.7 %
- 12.2 % Δ 16.1 %
- 17.3 % + 16.9 %

FIG 3 HARDNESS OF SAWDUST PLASTIC

FIG. 4 DENSITY OF SAWDUST PLASTIC

FIG. 5 VOLUME CHANGE OF SAWDUST PLASTIC ON WATER SOAKING
TIME, 3 WEEKS TEMPERATURE, 25 °C.

FIG. 6 WEIGHT CHANGE OF SAWDUST PLASTIC ON WATER SOAKING TIME, 3 WEEKS TEMPERATURE, 25 °C

Fig. 8 gives the Rockwell R Hardness of the soaked samples after being dried 4 days at room temperature and humidity. The general trend is for the maximum hardness to occur in the samples which were cured at 385° F. Also, the general trend still has the lower moisture content samples with the maximum hardness.

During the soaking period of 3 weeks the water becomes darkened with material being extracted from the samples. The amount of material extracted is presented in Fig. 9 and Fig. 10. The amount extracted is based on weights taken after 4 days drying of the soaked sample at room temperature and humidity. The amount of extractable material appears to decrease as the curing temperature decreases to 345° F. at which point it starts to rise again. Also, the extractable material appears to increase as the moisture content rises to 16.1% moisture at which point it starts to rise again. The extractable material seems to be at a maximum in those samples which had minimum expansion and gain in weight. The extractable material is probably the lignin in the smect which is separated from the wood fiber by the high pressure steam produced in the sample while under high pressure and temperature conditions. In other words, the more lignin which is separated from the wood fiber to act as the binder in the new shaped plastic, the higher the material which is extractable.

FIG. 7 DENSITY OF SOAKED SAWDUST PLASTIC AFTER REDRYING
WATER SOAKING TIME, 3 WEEKS TEMPERATURE, 25 °C.
AIR DRYING TIME, 4 DAYS TEMPERATURE, 25 °C.

LEGEND

SAWDUST MOISTURE CONTENT

• 11.2 %	X 14.7 %
○ 12.2 %	△ 16.1 %
□ 13.3 %	+ 16.9 %

FIG. 8 HARDNESS OF SOAKED SAWDUST PLASTIC AFTER REDRYING
 WATER SOAKING TIME, 3 WEEKS TEMPERATURE, 25 °C
 AIR DRYING TIME, 4 DAYS TEMPERATURE, 25 °C

FIG. 9 EFFECT OF CURING TEMPERATURE ON EXTRACTABLE MATERIAL

FIG. 10 EFFECT OF INITIAL SAWDUST MOISTURE CONTENT ON EXTRACTABLE MATERIAL

CONCLUSIONS:

The optimum conditions for production of plastic from sandst is recommended as a moisture content of 14 - 18% for the sandst, a curing temperature between 335° F and 365° F, and an applied pressure of 2500 - 3000 P.S.I. It is also recommended that the article to be molded shall have its minimum dimension in the vertical position; and that the mold be rinsed with water.

WOOD PLASTIC FROM WOOD

After observing the properties of lignin in sawdust to act as a binder to form a plastic, it was considered worthwhile to see if these characteristics would be present if wood blocks were used instead of sawdust. The treatment used to produce this substance should not disintegrate the fibers of the wood.

APPARATUS:

The main piece of apparatus used was a hydraulic press made by the Adolph I. Bushler Company with accessory molding equipment. (See page 17).

A standard Rockwell Hardness Testing Machine was used with a $\frac{1}{2}$ inch steel ball, a 60 kg. major load, and the readings on the red scale.

PROCEDURE:

Cubes of wood were cut and shaped into cylinders with a wood rasp. These cylinders were made 1 inch in diameter and 1 inch high to fit the mold. The cylinders of wood were soaked in water for various lengths of time before putting into the mold. The pressure was applied to the sample in the mold and the heating coil was started. The average time required to reach the desired temperature was 9 minutes. The average cooling time was 30 minutes to cool below 200° F. The sample was tested with a knife to determine the uniformity. The hardness was determined on the Rockwell Hardness Testing Machine as given on page 17 in the procedure for testing

smoked plastics. The density was determined by weighing in air, weighing in water, and calculating on the basis of the volume of water displaced by the sample.

DATA AND RESULTS:

The properties of wood plastics are given in Tables VI, VII, and VIII. The structure seems better when no moisture is added to the wood. When the wood is soaked in water even for a short period of time, the resin in the wood has a tendency to be driven along the grain of the wood toward the center making a distinct band of high resin content.

There appears to be a lower critical temperature and a lower critical pressure below which no fusion takes place.

The best Cottonwood sample is one cured without moisture added at 3000 P.S.I. and 350° F. maximum temperature. The best results obtained from White Oak were with 340° F. cure for 10 minutes at 3000 P.S.I. and 1 minute soak in water before cure. The best results from Red Oak were obtained by curing without added moisture at 4000 P.S.I. and 300° F. maximum curing temperature.

TABLE VI PROPERTIES OF WOOD PLASTICS FROM COTTONWOOD
(Pressed at 2000 P.S.I. at Curing Temperature for 10 Minutes)

<u>Direction of Grain of Wood</u>	<u>Minutes Water- Soaked</u>	<u>Temp. of Cure * F.</u>	<u>Density g/cc</u>	<u>Rockwell "R" Hardness</u>	<u>Structure</u>
Horizontal	1	330	1.38	110	non-uniform
Horizontal	0	330	1.38	106	uniform
Horizontal	5	330	1.38	-	non-uniform
Horizontal	25	330	1.35	88	non-uniform
Horizontal	0	*305	1.36	114	uniform
Horizontal	0	*232	1.15	75	unfused
Vertical	5	330	1.36	83	edge creased-uniform
Vertical	5	330	1.54	84	edge creased-uniform
Vertical	25	330	1.35	84	edge creased-uniform
Vertical	50	330	1.29	58	edge creased-uniform

* Maximum temperature reached with 10 minute cure.

TABLE VII PROPERTIES OF WOOD PLASTICS FROM WHITE OAK
(Pressed at 3000 P.S.I. at Curing Temperature for 10 Minutes.)

<u>Direction of Grain of Wood</u>	<u>Minutes Water- Soaked</u>	<u>Temp. of Cure ° F.</u>	<u>Density g/cc</u>	<u>Rockwell "R" Hardness</u>	<u>Structure</u>
Horizontal	0	•374	1.40	114	unfused lower edge
Horizontal	0	330	1.35	119	unfused lower edge
Horizontal	1/10	340	1.42	113	uniform
Horizontal	1/10	340	1.41	116	uniform
Horizontal	1	340	1.41	116	uniform
Vertical	0	330	1.38	119	edges creased

• Maximum temperature reached without 10 minute cure.

TABLE VIII PROPERTIES OF WOOD PLASTICS FROM RED OAK

<u>Direction of Grain of Wood</u>	<u>Pressure P.S.I.</u>	<u>Minutes Water- Soaked</u>	<u>Max. Temp. of Cure ° F.</u>	<u>Density g/cc</u>	<u>Rockwell "R" Hardness</u>	<u>Structure</u>
Horizontal	3000	0	330	1.40	119	unfused lower edge
Horizontal	3000	2	•340	1.40	114	uniform
Horizontal	3000	1/10	330	1.41	115	uniform
Horizontal	3000	0	280	1.21	101	completely unfused
Horizontal	1000	0	330	0.98	80	completely unfused
Horizontal	4000	0	330	1.44	120	uniform
Horizontal	4000	1/10	364	1.42	116	uniform
Vertical	3000	0	•330	1.30	113	warped grain
Vertical	3000	0	•370	1.33	117	warped grain

• Held at curing temperature for 10 minutes.

SECTION II

INVESTIGATIONS IN MARINE GLUING

INTRODUCTION

A marine gluing project was set up in Louisville for laminating keels, ribs, and other large parts of wooden boats that previously were restricted to use of beams of solid timber. The Chemical Engineering Department of the University of Louisville worked in conjunction with this project. This work consisted of checking types and suitabilities of glues for marine gluing. Certain glues were certified by the Forest Products Laboratory for use in marine gluing. However, experimentation was still required to determine the optimum conditions for the use of these glues and methods for quick testing.

Experiments were started for the determination of optimum curing temperatures and times for various glues in extension of previous work done by J. I. Stevens.

Since the glues were being used in hot rooms in summer, certain experimental work on the pot-life of the glues was requested. This was also included as a portion of the work.

The subject of plasticity of one type of glue became of extreme interest when studies were made of cured and curing laminate timbers at temperatures 190° to 210° F. in a curing chamber. It was desired to determine whether the resin adhesive, after a 24-hour room temperature aging period became liquid, semi-plastic, or retained its room temperature hardness during the heating up cycle. It was

also desired to find the temperature, the minimum allowable shear value, and the nature of the plasticity if softness did result.

HISTORICAL

It has been common practice in the protective coatings industries to follow the polymerization of resin films by a series of solvent extractions. The use of acetone extraction of glue films has been investigated by R. F. Elomquist, Assistant Chemist, Forest Products Laboratory. He presented in tabular and graphical form data which indicate definite correlations between the extractable material and the time and temperature of cure, when curing is done on a surface that is chemically inert.

The resin adhesive most used in marine gluing is Cascophen LF-67, a neutral phenolic-aldehyde partially cured resin which is mixed with a hardener, usually H-18, which is of a para-formaldehyde base. However, another hardener, FH-60, (reported to contain ammonium sulfate) is also used for specific purposes.

The viscosity of these mixed adhesives and slippage in glue bonds with 24-hour pressure were problems which arose in marine gluing at Gamble Bros. and have not been investigated previously.

THEORETICAL

In the determination of the degree of cure for phenolic resins by the use of acetone extraction the degree of cure is a function of the solubility of the powdered glue film in the acetone. The curing of a resin adhesive of the phenol-aldehyde or related type proceeds by the mechanism of condensation with the formation of three dimensional or space polymers of unknown size or completed structure. Acetone has the characteristic of dissolving low and medium molecular weight polymers of the phenol class. This method is more rapid and requires less material and labor than methods involving glue joints and since no wood is used, the method is not subject to variations in the strength of wood.

The mechanism of condensation or growth of molecules is accompanied by a corresponding increase in viscosity of the resin adhesive. By the addition of certain catalysts or "hardeners" this condensation process is speeded up tremendously. The measure of the changing viscosities of the materials give an indication of the catalyst's effectiveness.

Investigations have shown that Cascophen LT-47 with H-18 catalyst is thermoplastic after a pressure of 24-hours at room temperature. That is, the prepared glue film becomes plastic or regains the ability to be formed when heated. However, while an elevated temperature is being reached, the process of condensation

or hardening is being accelerated. Therefore, the question of when a glue bond will shear while being cured after a 24-hour process is a function of the shearing force applied and the temperature of cure.

EXPERIMENTAL

ACETONE EXTRACTION OF GLUE FILMS

Acetone was chosen for the extraction of glue films because it has the characteristic of dissolving the lower molecular weight resins. This property can be used to indicate the percent cure of glue films. The results of this section have been correlated with farther experimentation by the Forest Products Laboratories.

APPARATUS:

The main apparatus used was a battery of Soxhlet extraction units. Each unit consists of a 400 ml. extraction flask, a siphon cup, a Whatman extraction thimble, and a condenser cap. The siphon cup containing the extraction thimble was suspended by fine wire from the condenser cap which rested on the neck of the flask. The units were contained in five batteries of twelve units each. The batteries were in the form of rectangular wooden boxes lined with asbestos paper, and fitted with removable lids in which holes were cut to receive the extraction flasks. The flasks were placed in cylindrical metal reflectors which directed the heat to them from the 75 watt incandescent lamp heaters. Cooling water for the condensers was supplied through an iron pipe manifold and the cooling system of the batteries was connected in series.

Laboratory electric ovens were used to cure the glue films on glass plates. The glue films were ground in a standard porcelain mortar and pestle and passed through a standard 48 mesh screen.

PROCEDURE:

The procedure consisted of preparing the adhesive, spreading glue on glass plates, and curing in oven at prescribed temperatures for designated times. The cured flakes were ground and extracted with acetone.

A double thickness of Whatman filter paper was placed inside a Whatman extraction thimble and extracted with acetone in the extraction batteries for 24 hours. The thimble was then dried at 110° C in an electric oven for several hours. The dried thimble was placed in a desiccator and weighed after coming to equilibrium moisture content. The powdered glue flake was placed in the weighed thimble, reweighed, and extracted in the extraction batteries for 24 hours. The thimble containing extracted powdered glue was then dried as before, placed in a desiccator to attain equilibrium moisture and weighed. The weight extracted was obtained by difference and the percent extracted was calculated.

DATA AND RESULTS AND CONCLUSIONS:

Table IX and Fig. 11 present the acetone extraction of the phenolic-aldehyde Cascophan LT-67 adhesive. This procedure did not give very close checks; however, the broad implications of the work are evident. Acetone solubility of a fully cured LT-67 - N-12 film must be almost negligible as shown by the small negative values obtained on 24-hour films at 200° F.

TABLE IX ACETONE EXTRACTION OF LT-67 ADHESIVE

	Time of Cure	Percent Extractable					
		150° F.	160° F.	170° F.	180° F.	190° F.	200° F.
8 Parts	6 hrs.	28.4	16.4	10.3	11.1	6.8	7.7
N-18		26.8	14.8	11.0	12.8	7.1	9.8
Accelerator			17.3	17.7	15.5	8.8	8.3
Per			19.8		11.1	8.4	2.8
100 Parts			21.6		7.8	11.3	3.0
LT-67					10.1	9.0	
					8.8	7.8	
	8 hrs.				5.0	2.8	
					4.7	3.8	
					9.8		
	10 hrs.				8.3	4.8	
					4.1	6.0	
						8.1	
	24 hrs.						- 4.8
							- 2.8
							- 2.8
Pure	6 hrs.					26.8	
LT-67	8 hrs.					24.8	
	10 hrs.					18.7

FIG. 11 ACETONE EXTRACTION OF LT 67 WITH MIB CATALYST

Films of pure LT-67 without accelerator, cured 6, 8, and 10 hours at 180° F show erratic but relatively high values and indicate a very slow polymerization which is at present outside the time limitations of a production schedule.

The percent extractable curve on a six-hour curing period appears to flatten out above 180° F. and the extractable material does not rapidly diminish even with a twenty degree increase of temperature.

Increasing times of 8 and 10 hours at 180° F. and 190° F. indicate decreasing extractable contents over the 6-hour cure while the effect of increased time appears of more consequence than the effect of temperature at this point.

The formulation for phenolic PR-75-B adhesive is 90 parts of PR-75-B liquid resin, 13.6 parts of P-79 powder accelerator, and 10 parts of commercial ethyl alcohol. The liquid PR-75-B resin had a pH 7.6. The formulation had a pH 7.0.

The visual examination of cured flakes showed an increase in depth of color as time and temperature increased. All flakes at 180° F. and 200° F. showed evidence of bubble formation in the flakes. All flakes were devoid of any phenolic odor, even at lowest times and temperatures. Flakes were increasingly hard as the temperature increased, whereas time seemed to have no significant effect.

Table I and Fig. 12 present the acetone extraction data. An initial set may be obtained at 140° F. in ten hours or less but the resultant adhesive is not insoluble in Soxhlet acetone extraction until a curing temperature of 180° - 200° F. is reached. After 12 hours at any temperature above 140° F. the time of cure is relatively insignificant up to the limits of this experiment. (20 hours cure). The adhesive is a normally neutral phenolic adhesive.

Catabond 880 is a phenolic-aldehyde resin glue marketed as a powder by the Catalin Corporation of America. The formulation is 1 part Catabond 880 powder, 2 parts water by weight.

The visual examination of cured flakes showed firmness, brittleness, and apparent uniform texture. The higher temperature and longer time produce a slightly darker and definitely stronger flake. There was no odor of phenol or formaldehyde in the cured flakes.

The results of the acetone extraction presented in Table II are extremely erratic even though they indicate a definite decrease in acetone solubility with increasing temperature of cure. An examination of the extracted powder furnished some clues and invalidated much of the data in Table II.

The 140° F. cured powder was very soft and during the extraction and drying cycle was hardened and set into a compact ball,

TABLE X ACETONE EXTRACTION OF PR-75-B ADHESIVE

Cure Temp.	% Extractable					
	10 hrs.	12 hrs.	14 hrs.	16 hrs.	18 hrs.	20 hrs.
140° F.	-	11.9	6.1	6.7	11.0	6.8
	-	6.1	6.7	6.8	9.2	-
160° F.	-	8.3	6.6	6.4	7.4	4.8
	-	8.5	4.4	4.4	6.8	-
180° F.	4.4	4.0	6.7	6.3	1.8	-
	1.2	0.9	- 0.8	2.4	1.7	-
200° F.	0.2	- 0.7	- 2.8	0.3	1.4	-
	- 0.6	- 0.8	- 1.0	-	1.4	-

TABLE XI ACETONE EXTRACTION OF CATANED 650

Cure Temp.	% Extractable				
	2 hrs.	4 hrs.	6 hrs.	8 hrs.	10 hrs.
140° F.	11.8	16.5	13.6	17.1	17.6
160° F.	12.6	4.5	3.4	1.9	6.4
180° F.	2.2	4.6	4.2	4.2	5.4
200° F.	2.8	- 1.6	- 2.6	- 2.1	- 1.1

FIG.12 ACETONE EXTRACTION OF PR-75-B RESIN WITH P79 ACCELERATOR

whereas, normal extracted residues remained in their original freely powdered form.

The 180° F. cured powder was medium soft and during the extraction and drying cycle was set into compact balls which were relatively soft and would crumble between the fingers.

The 180° F. and 200° F. cured powders were hard and retained their freely powdered form through the process.

It is highly probable that the 140° F. and 160° F. cured flakes were insufficiently cured and in presence of acetone flowed and formed compact masses hindering the extraction process by having greatly decreased surface exposed to acetone extraction. The other two temperatures indicated a decrease in solubility of cured flakes to a near zero value at 200° F. when 4 or more hours cure was used.

VISCOSITY OF MIXED CASCOPHEN LT-67 ADHESIVE

The pot-life of mixed Cascophen LT-67 adhesives was desired to determine the limitations of the adhesives used in a hot production room. This section was undertaken to determine the pot-life in terms of viscosity change.

APPARATUS:

The apparatus used in this determination was a #4 Ford cup, a Kodak timer, and standard laboratory glassware.

PROCEDURE:

Six 250 ml samples were prepared according to formulation and held at room temperature (80° F.) in pyrex beakers covered with watch glasses for extended periods of time while viscosity tests were made at intervals. The viscosity measurements were made by observing the time in seconds required for a flow of 50 ml. of glue through the orifice of the standard #4 viscosity cup.

DATA AND RESULTS:

Table XII presents the viscosity data for mixed Cascophen LT-67 adhesives. The viscosity of the adhesive containing 8% H-18 catalyst increased the most rapidly, the adhesive containing 8% PH-80 was the next most rapid, and the rate of change viscosity became lower as the amount of catalyst was decreased. (See Fig. 13 and Fig. 14.)

TABLE XII DATA AND RESULTS

VISCOSEITY OF MIXED CASCOFREN LT-67 ADHESIVES
(Temperature Constant at 80° F.)

<u>LT-67</u> <u>5% E-18</u>		<u>LT-67</u> <u>None</u>		<u>LT-67</u> <u>5% FE-80</u>	
<u>Time of</u> <u>Set</u> <u>(Min)</u>	<u>Viscosity</u> <u>in</u> <u>Secs</u>	<u>Time of</u> <u>Set</u> <u>(Hrs)</u>	<u>Viscosity</u> <u>in</u> <u>Secs</u>	<u>Time of</u> <u>Set</u> <u>(Hrs)</u>	<u>Viscosity</u> <u>in</u> <u>Secs</u>
0	120	0	85	0	98
25	137	1	85	1	108
50	143	4	88	2	104
45	152	22	95	2.5	109
60	168	29	95	3	104
75	198	47	100	4	108
90	234	55	101	5	105
105	298	72	129	6	108
120	420	95	155	23	112
135	500	101	152	31	112
150	1065	143	191	48	132
195	Solid	167	223	54	128
				72	157
				95	213
				105	207
				144	267
				165	205

TABLE XII (Con't.)

VISCOSITY OF MIXED GASCOPHEN LT-57 ADHESIVES

LT-57 6% FM-50		LT-57 6% FM-50		LT-57 6% FM-50	
Time of Set (hrs)	Viscosity in Sec. *	Time of Set (hrs)	Viscosity in Sec. *	Time of Set (hrs)	Viscosity in Sec. *
0	122	0	154	0	195
1	133	1	173	1	247
2	128	2.5	179	1.5	257
3	125	3.5	182	4	249
3.5	125	4	191	5	251
4	135	5	195	25	310
5	127	25	194	29	270
6	127	30	195	48	313
22	140	48	226	54	315
31	150	54	224	72	426
48	172	72	232	95	552
54	169	95	257	105	525
72	194	105	276	144	802
95	205	144	400	155	1100
105	275	155	525		
144	340				
155	395				

* Time of flow for 50 ml from #4 standard Ford Viscosity Cup.

FIG. 13 VISCOSITY CHANGES ON LT67 ADHESIVES AT 80°F.
50 ML. THROUGH NO. 4 FORD CUP

FIG. 14 VISCOSITY CHANGES ON LT 67 ADHESIVES AT 80°F.
 50 ML. THROUGH NO. 4 FORD CUP

CONCLUSIONS:

The presence of M-16 catalyst greatly accelerates the tendency of LF-67 to become viscous. At 80° F. under the conditions of test it became too viscous for normal spreading within two hours after mixing (400, #4 Ford cup seconds).

The use of catalyst PM-60 produced an initial fast rise in viscosity, followed by a long period of further slow gain. An 8% catalyst mix reached 400 #4 Ford cup seconds only after about 70 hours, a 6% mix at 115 hours, and a 4% mix at 170 hours. The plain LF-67 which was held as a control started at 85 seconds and even after 170 hours (1 week) only reached 235 seconds.

PLASTICITY OF LX-57, GASCOFENE, GLUE BONDS

The plasticity of the glue bonds became important when it was observed that some of the glued heels and ribs lost their shape slightly on curing supposedly due to softening of bond. This section of the project was designed to determine allowable pressure or friction factors at the curing temperatures.

APPARATUS:

The two main pieces of apparatus were two hydraulic presses. One was a hydraulic press made by the Adolph I. Buchler Company. (See page 17). The other was a hydraulic press made by the Fred S. Carver Company with electrically heated platens. A heating unit was made by wrapping a tin can with asbestos and winding heating element wire around it. Other apparatus included an electric oven, a metal clamp, thermocouples, potentiometer, and a Kodak timer.

PROCEDURE:

Preliminary tests were made by subjecting sharp-cut, dry-cured resin, to elevated temperatures in a rapid manner, e.i. temperature rose to 210° F. in 5 minutes. These were run in constant temperature (dry) electric ovens. Small bits of resin adhesive were placed on a Fisher melting point apparatus and the temperature raised at rates from 1° F./min. to 10° F./min. under dry conditions and also in the presence of a drop of enclosed water.

White oak blocks, $5\frac{1}{2}'' \times 2\frac{1}{2}'' \times \frac{3}{4}''$, were glued with LF-67, H-18 catalyst, in sets of three, the center block extending $\frac{1}{2}''$ beyond the ends of the two outer blocks. A copper-constantan thermocouple was placed in the center of each glued face, the assembly clamped and allowed to pressure for 24 hours at room temperature. (70° F.)

Pressure glued blocks were placed inside the heating element in the Buchler press, the heating element was started, and a predetermined pressure was applied. Temperature readings were taken at five minute intervals until breakage occurred or until 220° F. was reached. The temperature rate was approximately 2° F./min. and the shearing area was 25 square inch.

White oak blocks were also prepared of varying areas and fitted with cardboard spacers on the outer edges to produce glue lines of 0.030" - 0.040". All blocks were pressure in clamps for 24 hours at room temperature and the cardboard stripped edges were planed off. During the gluing operation, thermocouples had been placed in the center of each set for temperature measurement. All blocks were placed between platens held at 200° F. and predetermined pressures were applied. The heating rate was found to be about 5° F. per minute and all samples were held under heat and pressure for five minutes after glue line showed 220° F.

DATA AND RESULTS:

The results of the preliminary tests showed no melting of the sharp cut, day-cured resin, and the sharpness of the edges was

retained on all samples. No deterioration was noted on any edge or section of the solid when tested dry on the Fisher melting point apparatus. However, when the experiment was repeated with a drop of water in the enclosure with the specimen, at around 200° - 210° F. the resin began to swell noticeably, it absorbed water vapor and water, becoming a puffy mass which boiled visably at approximately 212° F. Similar experiments confirmed this observation and yielded the information that the moisture actually produced a soft granulation of the otherwise tough-rubbery mass.

The results of the tests on the shearing blocks is best illustrated in Table XIII and on Fig. 15 as applied unit shear vs. temperature at breaking point.

TABLE XIII STRENGTH OF LY-67 DURING CURE

<u>Applied Shear P.S.I.</u>	<u>Temperature at yield point --- ° F.</u>
23.6	No yield point - sample cured
24.8	No yield point - sample cured
26.0	128° F.
30.7	117° F.
32.9	108° F.
35.4	103° F.
59.0	84° F.
116.0	70° F.

FIG. 15 STRENGTH OF LT6T DURING CURE
24 HOURS ROOM TEMPERATURE PRECURE
HEATING RATE, 2°F. PER MINUTE

It is to be noted that no breaks were found at less than 35 P.S.I. unit shear, but that a slight increase caused failure at 125° F. Increasingly higher shear values caused lower temperatures of failure in an apparent hyperbolic function. This experiment shows a definite softening of the pressured adhesive but indicates that the effect is not excessive when unit shear is the measure.

The results of the compression tests on the blocks with thick glue bonds was as follows:

- #1 Unit compression 400 P.S.I. Glue line bowed slightly upon application of pressure and condensation proceeded slowly for 20 minutes until 200° F. was reached. No further condensation took place but the glue line had bowed out about 3/64".
- #2 Unit compression 200 P.S.I. Slight bowing of glue line when 150° F. was reached.
- #3 Unit compression 150 P.S.I. Slight extension of glue line at isolated spots on upper surface.
- #4 Unit compression 100 P.S.I. Slight extension of glue line at isolated spots on upper surface.

There was no appreciable movement of glue in any specimen but that at 400 P.S.I.

CONCLUSIONS:

The pressuring of LF-67 with E-18 catalyst, for 24 hours at 70° F., produces a condition which becomes only slightly soft with a secondary heating. This pressure will allow for 20 - 25 P.S.I. shear stress design for holding clamps during further cure, if in a dry atmosphere until set. Moisture softens the pressured resin and may cause a lowering of the 20 - 25 P.S.I. limit, although this will apply only to external or thin veneer glue joints. Evidence on the shear specimens indicates that plasticity of the pressured film increases with temperature and reaches a relative high value at about 120° F. This can bear little relation to degree of cure, as this temperature has been found definitely inadequate to produce high acetone insolubility.

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VITA

Charles Francis Saora was born at Louisville, Kentucky, on October 10, 1921, the son of James L. Saora and Ruth E. Fowler. He received his primary and secondary education in the public schools of Louisville. He entered the Speed Scientific School of the University of Louisville in September, 1939, and obtained the degree of Bachelor of Chemical Engineering in March, 1943. He received the degree of Master of Chemical Engineering in October, 1944.

Mr. Saora was awarded a Division of Industrial Research fellowship at the University for the year 1943-1944. In addition to his research he assisted in the Engineering, Science, and Management War Training program at the University of Louisville.

He is a member of Theta Chi Delta, National Honorary Chemistry Fraternity and was a member of the University of Louisville Student Chapter of the American Institute of Chemical Engineers.