Impregnation and wood plastics: II. investigations in marine glueing.

Charles F. Sacra 1921-1985

*University of Louisville*

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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 P. Sear

1964
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I  IMPREGNATION AND WOOD PLASTICS

II  INVESTIGATIONS IN MARINE GLUEING

Charles P. Saern

Approved by the Examining Committee:

Director  Gordon C. Williams

R. C. Ernst

W. R. Barnes

October 1966
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ACKNOWLEDGMENT

The author wishes to acknowledge the kind assistance and helpful guidance of Dr. G. C. Williams who directed this research.
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 Caseophen LF-67 adhesives. The viscosity correlations are based on the flow of the material through an orifice.

The plasticity of Caseophen 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

IMPROVEMENT 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 utilising a cheap, soft wood as the raw material and impregnating the surface.

A related problem in the wood industry is the utilisation 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 utilisation 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.

Staude 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.

Staude and Seborg (14, 15, 16) 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 (5) 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 1.5%. The fines plasticized by the water were molded into the product under heat and pressure.

Mason, Roehm, and Roence (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 Sherard (10) 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 residues was dried, ground, and hot pressed with water alone or with additions of aniline and furfural plasticizer.

Glenn and Plow (8) 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 90 to 1000 lbs/in² at a temperature of about 105⁰ C to about 200⁰ 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 140⁰ C and about 200⁰ C and subjected to pressures from about 2000 lbs. to about 7500 lbs/in² in a mold.

A method employed by Stuhler (5) 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. Clegg (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 Bissellene Products & Chemical Co. (7) present the theory that the most effective type of impregnation requires that the impregnant 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 impregnant characterized by small molecular size, in which the condensation has been arrested at an early stage. It is also important to select a resin impregnant that is acceptable to the celluless of the wood and one that will penetrate as quickly as possible and diffuse with reasonable uniformity throughout the wood structure.

Simonds 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 \(-\text{lignin about 86\% and \(-lignin about 8\%}. \text{ Lignin polymersize 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, Heglinger and Bohf (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.
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 50 square inches. The press has a pressure gauge reading from 0 to 2000 lbs. per square inch hydraulic pressure and is graduated in 50 lb. increments. It is possible to obtain up to 50 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 un-
treated 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 FR-88 made by Resinous Products
and Chemical Co., Incorporated, and DuraS 6053 made by DuraS Plastics
and Chemicals, Incorporated. Solutions of each resin were 30% solids.

Preliminary experiments indicated that wetting of the
surface of the wood before collagen 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 250° F. in the press
under pressure.

Table I presents results of a series of samples soaked in
Amberlite FR-88 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
DuraS 6053 solution, air-dried for a period over 24 hours and dried
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<th>Pressure P.S.I.</th>
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<td>3</td>
<td>800</td>
<td>15/32&quot;</td>
<td>fair</td>
<td>grooved</td>
<td></td>
<td>fair</td>
</tr>
<tr>
<td>3</td>
<td>1000</td>
<td>1/4&quot;</td>
<td>fair</td>
<td>slightly ridged</td>
<td></td>
<td>very good</td>
</tr>
<tr>
<td>4</td>
<td>500</td>
<td>15/32&quot;</td>
<td>fair</td>
<td>grooved</td>
<td></td>
<td>fair</td>
</tr>
<tr>
<td>4</td>
<td>1000</td>
<td>1/4&quot;</td>
<td>fair</td>
<td>slightly ridged</td>
<td></td>
<td>very good</td>
</tr>
<tr>
<td>6-1/2</td>
<td>500</td>
<td>15/32&quot;</td>
<td>excellent</td>
<td>good</td>
<td></td>
<td>very good</td>
</tr>
<tr>
<td>6-1/2</td>
<td>1000</td>
<td>1/4&quot;</td>
<td>good</td>
<td>slightly ridged</td>
<td></td>
<td>very good</td>
</tr>
<tr>
<td>7-1/2</td>
<td>200</td>
<td>15/32&quot;</td>
<td>very good</td>
<td>good</td>
<td></td>
<td>very good</td>
</tr>
<tr>
<td>7-1/2</td>
<td>1000</td>
<td>1/4&quot;</td>
<td>good</td>
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<tr>
<td>20</td>
<td>200</td>
<td>15/32&quot;</td>
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<td>excellent</td>
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<td>very good</td>
</tr>
<tr>
<td>30</td>
<td>1000</td>
<td>1/4&quot;</td>
<td>good</td>
<td>excellent</td>
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<td>excellent</td>
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* This sample had no cooling period in press.
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<th>Soaking Time (Hrs.)</th>
<th>Pressure P.S.I</th>
<th>Thickness</th>
<th>Penetration</th>
<th>Property Appearance</th>
<th>Hardness</th>
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<tr>
<td>3</td>
<td>1000</td>
<td>1/4&quot;</td>
<td>poor</td>
<td>fair</td>
<td>fair</td>
</tr>
<tr>
<td>4</td>
<td>1000</td>
<td>1/4&quot;</td>
<td>good</td>
<td>good</td>
<td>very good</td>
</tr>
<tr>
<td>6-1/2</td>
<td>1000</td>
<td>1/4&quot;</td>
<td>fair</td>
<td>fair</td>
<td>good</td>
</tr>
<tr>
<td>7-1/2</td>
<td>1000</td>
<td>1/4&quot;</td>
<td>fair</td>
<td>good</td>
<td>very good</td>
</tr>
<tr>
<td>22</td>
<td>250</td>
<td>1/8&quot;</td>
<td>fair</td>
<td>fair</td>
<td>poor</td>
</tr>
<tr>
<td>22</td>
<td>1000</td>
<td>5/16&quot;</td>
<td>good</td>
<td>excellent</td>
<td>excellent</td>
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</table>

* These two samples had no cooling period in press.
at 160° F. for 22 hours. They were cured for 1/2 hour at 300° F. and
allowed to cool to 220° F. under pressure.

CONCLUSIONS:

Amberlite MB-23 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 7½ hours or less tend to have
better surface properties at low (200 P.S.I.) curing pressures than
at high (1000 P.S.I.).

Durac 6000 surface impregnated wood with a 22 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 6½ 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. Buchlar 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 8" x 1" x 1". 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 $\frac{1}{2}$" steel ball, a 60 kg.
major load, and readings made on red scales.

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 com-
position. 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 Bushler 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
misometer, 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 \( 25^\circ \) 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
platen were set at the desired temperature, the mixust 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 \( \frac{3}{4} \) inch ball penetra-
tor, a minor load of 10 kg, and a major load of 60 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 core 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
sawdust with a range roughly from 11 to 18% moisture could be fused
partially by compression at 3000 p.s.i. and heating for 10 minutes
at temperatures from 300° to 350° 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 sawdust plastic. The hardness at 3000 p.s.i. is
essentially the same as the hardness at 2000 p.s.i., but below
3000 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 2500 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 300°F.

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 sawdust 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 20% relative humidity at both 70°F and 120°F was produced using a MgCl₂-6H₂O 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 sawdust plastic there is no appreciable change up to a 20% relative humidity at temperatures of 70°F and 120°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. 5. From Fig. 5 it is evident that the hardness goes down as the initial moisture content of the sawdust goes up regardless of curing
### TABLE III  PROPERTIES OF FULLY FUSED SANDUST PLASTIC - 11 DAY EXPOSURE

<table>
<thead>
<tr>
<th>Density g/cc</th>
<th>Rockwell K Hardness</th>
<th>Testing Atmosphere Temp. °F</th>
<th>% R.H.</th>
<th>% Vol. Increase</th>
<th>% Wt. Increase</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.26</td>
<td>86</td>
<td>70</td>
<td>30</td>
<td>- 7.3</td>
<td>- 0.40</td>
</tr>
<tr>
<td>1.25</td>
<td>65</td>
<td>70</td>
<td>70</td>
<td>- 4.8</td>
<td>1.63</td>
</tr>
<tr>
<td>1.23</td>
<td>86</td>
<td>70</td>
<td>100</td>
<td>- 5.1</td>
<td>12.14</td>
</tr>
<tr>
<td>1.29</td>
<td>65</td>
<td>120</td>
<td>20</td>
<td>- 4.3</td>
<td>1.46</td>
</tr>
<tr>
<td>1.26</td>
<td>62</td>
<td>120</td>
<td>70</td>
<td>2.9</td>
<td>11.98</td>
</tr>
<tr>
<td>1.24</td>
<td>66</td>
<td>120</td>
<td>100</td>
<td>10.9</td>
<td>14.75</td>
</tr>
</tbody>
</table>

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

<table>
<thead>
<tr>
<th>Density g/cc</th>
<th>Rockwell K Hardness</th>
<th>Testing Atmosphere Temp. °F</th>
<th>% R.H.</th>
<th>% Vol. Increase</th>
<th>% Wt. Increase</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.29</td>
<td>57</td>
<td>70° F</td>
<td>30</td>
<td>- 8.6</td>
<td>- 0.33</td>
</tr>
<tr>
<td>1.25</td>
<td>65</td>
<td>70</td>
<td>70</td>
<td>- 1.6</td>
<td>2.63</td>
</tr>
<tr>
<td>1.24</td>
<td>57</td>
<td>70</td>
<td>100</td>
<td>9.2</td>
<td>13.65</td>
</tr>
<tr>
<td>1.31</td>
<td>65</td>
<td>120</td>
<td>20</td>
<td>- 6.2</td>
<td>1.60</td>
</tr>
<tr>
<td>1.29</td>
<td>66</td>
<td>120</td>
<td>70</td>
<td>18.1</td>
<td>12.70</td>
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<tr>
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<td>66</td>
<td>120</td>
<td>100</td>
<td>40.9</td>
<td>35.80</td>
</tr>
<tr>
<td>------------</td>
<td>-----------------</td>
<td>---------</td>
<td>---------------</td>
<td>--------------------------</td>
<td>----------</td>
</tr>
<tr>
<td>11.2</td>
<td>370</td>
<td>1.207</td>
<td>90</td>
<td>10.07</td>
<td>11.32</td>
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<tr>
<td>12.2</td>
<td>370</td>
<td>1.207</td>
<td>96</td>
<td>10.49</td>
<td>11.76</td>
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<td>370</td>
<td>1.245</td>
<td>90</td>
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<td>12.82</td>
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<td>370</td>
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<td>76</td>
<td>8.99</td>
<td>11.57</td>
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<td>15.1</td>
<td>370</td>
<td>1.297</td>
<td>87</td>
<td>9.68</td>
<td>12.20</td>
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<td>370</td>
<td>1.324</td>
<td>64</td>
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<td>1.207</td>
<td>96</td>
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<td>12.93</td>
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<td>95</td>
<td>10.89</td>
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<tr>
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<td>365</td>
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<td>97</td>
<td>10.59</td>
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<td>8.98</td>
<td>12.93</td>
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<td>365</td>
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<td>70</td>
<td>10.48</td>
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<tr>
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<td>365</td>
<td>1.323</td>
<td>70</td>
<td>9.84</td>
<td>14.32</td>
</tr>
<tr>
<td>11.2</td>
<td>355</td>
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<td>10.61</td>
<td>12.42</td>
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<tr>
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<td>355</td>
<td>1.572</td>
<td>102</td>
<td>10.95</td>
<td>12.90</td>
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<tr>
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<td>355</td>
<td>1.563</td>
<td>100</td>
<td>10.55</td>
<td>12.90</td>
</tr>
<tr>
<td>% Moisture</td>
<td>Curing Temp. ° F.</td>
<td>Density</td>
<td>Hardness</td>
<td>% Vol.C.</td>
<td>% Wt.C.</td>
</tr>
<tr>
<td>------------</td>
<td>------------------</td>
<td>---------</td>
<td>-----------</td>
<td>----------</td>
<td>---------</td>
</tr>
<tr>
<td>14.7</td>
<td>325</td>
<td>1.345</td>
<td>95</td>
<td>9.49</td>
<td>11.49</td>
</tr>
<tr>
<td>14.1</td>
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<td>1.310</td>
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</tr>
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<td>16.6</td>
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<td>78</td>
<td>10.35</td>
<td>15.20</td>
</tr>
<tr>
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<td>345</td>
<td>1.378</td>
<td>110</td>
<td>14.23</td>
<td>15.65</td>
</tr>
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<td>17.09</td>
<td>17.38</td>
</tr>
<tr>
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<td>14.94</td>
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<td>345</td>
<td>1.302</td>
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<td>10.82</td>
<td>15.44</td>
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<tr>
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<td>345</td>
<td>1.297</td>
<td>78</td>
<td>11.66</td>
<td>17.75</td>
</tr>
<tr>
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<td>355</td>
<td>1.386</td>
<td>110</td>
<td>16.22</td>
<td>18.09</td>
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<tr>
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<td>355</td>
<td>1.378</td>
<td>107</td>
<td>16.57</td>
<td>16.86</td>
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<tr>
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<td>355</td>
<td>1.366</td>
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<td>16.16</td>
<td>14.87</td>
</tr>
<tr>
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<td>1.339</td>
<td>90</td>
<td>11.03</td>
<td>14.33</td>
</tr>
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<td>355</td>
<td>1.307</td>
<td>84</td>
<td>11.47</td>
<td>16.88</td>
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<td>355</td>
<td>1.313</td>
<td>80</td>
<td>11.88</td>
<td>17.24</td>
</tr>
</tbody>
</table>
temperature. Also, the hardness goes down as the curing temperature rises regardless of moisture content of the sand.ust.

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 16% - 18% moisture content for all curing temperatures. The 16.7% moisture content samples had the lowest volume increase with the 16.1% and the 16.4% moisture content 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 15% - 18% moisture content. 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 samples except the 16.1 and the 16.4% moisture is to have the maximum density in those samples which were cured at 230°F. The trend is still to have the greater density as the moisture content decreases.
FIG 3 HARDNESS OF SAWDUST PLASTIC
**LEGEND**
Sawdust Moisture Content
- • 11.2%  X 14.7%
- O 12.2%  A 16.1%
- □ 13.3%  + 16.9%

**FIG 4** Density of Sawdust Plastic

Curing Temperature - °F
LEGEND

CURING TEMPERATURES

+ 335 °F
0 345 °F
8 355 °F
x 365 °F
D 370 °F

VOLUME CHANGE - % INCREASE

INITIAL SAWDUST MOISTURE CONTENT - % WATER

FIG. 5 VOLUME CHANGE OF SAWDUST PLASTIC ON WATER SOAKING TIME, 3 WEEKS TEMPERATURE, 25°C.
LEGEND
CURING TEMPERATURES
+
335°F.
O
345°F.
D
355°F.
X
365°F.
Δ
370°F.

WEIGHT CHANGE - % INCREASE

INITIAL SAWDUST MOISTURE CONTENT - % WATER

Fig. 6 Weight change of sawdust plastic on water soaking time, 3 weeks temperature, 25°C
Fig. 8 gives the Rockwell $E$ 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 became 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 wood dust 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
SAW DUST MOISTURE CONTENT
• 11.2 %  X 14.7 %
○ 12.2 %  Δ 16.1 %
□ 13.3 %  + 16.9 %

FIG 8 HARDNESS OF SOAKED SAWDUST PLASTIC AFTER RE DRYING
WATER SOAKING TIME, 3 WEEKS  TEMPERATURE, 25 °C
AIR DRYING TIME, 4 DAYS  TEMPERATURE, 26 °C
CURING TEMPERATURE - °F.
Fig. 9 Effect of curing temperature on extractable material.
LEGEND

CURING TEMPERATURES:

+ 335°F.

O 345°F.

@ 355°F.

x 365°F.

A 370°F.

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

INITIAL SAWDUST MOISTURE CONTENT - % WATER
CONCLUSIONS:

The optimum conditions for production of plastic from
sandlot is recommended as a moisture content of 14 - 18% for the
sandlot, a curing temperature between 325°F and 350°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 ½ 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.
sandust 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 250° F. maximum temperature. The best results obtained from White Oak were with 360° 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 350° F. maximum curing temperature.
### TABLE VI  PROPERTIES OF WOOD PLASTICS FROM COTTONWOOD

(Pressed at 3000 P.S.I. at Curing Temperature for 10 Minutes)

<table>
<thead>
<tr>
<th>Direction of Grain of Wood</th>
<th>Minutes Water-Soaked</th>
<th>Temp. of Cure °F</th>
<th>Density g/cc</th>
<th>Rockwell &quot;R&quot; Hardness</th>
<th>Structure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Horizontal</td>
<td>1</td>
<td>320</td>
<td>1.30</td>
<td>110</td>
<td>non-uniform</td>
</tr>
<tr>
<td>Horizontal</td>
<td>0</td>
<td>320</td>
<td>1.36</td>
<td>106</td>
<td>uniform</td>
</tr>
<tr>
<td>Horizontal</td>
<td>5</td>
<td>320</td>
<td>1.36</td>
<td>-</td>
<td>non-uniform</td>
</tr>
<tr>
<td>Horizontal</td>
<td>23</td>
<td>320</td>
<td>1.36</td>
<td>26</td>
<td>non-uniform</td>
</tr>
<tr>
<td>Horizontal</td>
<td>0</td>
<td>&lt;385</td>
<td>1.36</td>
<td>114</td>
<td>uniform</td>
</tr>
<tr>
<td>Horizontal</td>
<td>0</td>
<td>&lt;222</td>
<td>1.16</td>
<td>76</td>
<td>unfused</td>
</tr>
<tr>
<td>Vertical</td>
<td>3</td>
<td>320</td>
<td>1.38</td>
<td>53</td>
<td>edge creased-uniform</td>
</tr>
<tr>
<td>Vertical</td>
<td>5</td>
<td>320</td>
<td>1.54</td>
<td>56</td>
<td>edge creased-uniform</td>
</tr>
<tr>
<td>Vertical</td>
<td>23</td>
<td>320</td>
<td>1.53</td>
<td>34</td>
<td>edge creased-uniform</td>
</tr>
<tr>
<td>Vertical</td>
<td>30</td>
<td>320</td>
<td>1.29</td>
<td>33</td>
<td>edge creased-uniform</td>
</tr>
</tbody>
</table>

* 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.)

<table>
<thead>
<tr>
<th>Direction of Grain of Wood</th>
<th>Minutes Water-Soaked</th>
<th>Temp. of Cure ° F.</th>
<th>Density g/cc</th>
<th>Rockwell &quot;H&quot; Hardness</th>
<th>Structure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Horizontal</td>
<td>0</td>
<td>576</td>
<td>1.40</td>
<td>114</td>
<td>unfused lower edge</td>
</tr>
<tr>
<td>Horizontal</td>
<td>0</td>
<td>350</td>
<td>1.50</td>
<td>119</td>
<td>unfused lower edge</td>
</tr>
<tr>
<td>Horizontal</td>
<td>1/10</td>
<td>340</td>
<td>1.42</td>
<td>113</td>
<td>uniform</td>
</tr>
<tr>
<td>Horizontal</td>
<td>1/10</td>
<td>340</td>
<td>1.61</td>
<td>116</td>
<td>uniform</td>
</tr>
<tr>
<td>Horizontal</td>
<td>1</td>
<td>340</td>
<td>1.41</td>
<td>118</td>
<td>uniform</td>
</tr>
<tr>
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<td>350</td>
<td>1.38</td>
<td>119</td>
<td>edges creased</td>
</tr>
</tbody>
</table>

* Maximum temperature reached without 10 minute cure.
<table>
<thead>
<tr>
<th>Direction of Grain of Wood</th>
<th>Pressure (P.S.I.)</th>
<th>Minutes Water-Soaked</th>
<th>Max. Temp. of Cure (°F)</th>
<th>Density (g/cc)</th>
<th>Rockwell &quot;N&quot; Hardness</th>
<th>Structure</th>
</tr>
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<tbody>
<tr>
<td>Horizontal</td>
<td>3000</td>
<td>0</td>
<td>320</td>
<td>1.40</td>
<td>119</td>
<td>unfused lower edge</td>
</tr>
<tr>
<td>Horizontal</td>
<td>3000</td>
<td>3</td>
<td>340</td>
<td>1.40</td>
<td>114</td>
<td>uniform</td>
</tr>
<tr>
<td>Horizontal</td>
<td>3000</td>
<td>1/10</td>
<td>360</td>
<td>1.41</td>
<td>115</td>
<td>uniform</td>
</tr>
<tr>
<td>Horizontal</td>
<td>3000</td>
<td>0</td>
<td>360</td>
<td>1.51</td>
<td>101</td>
<td>completely unfused</td>
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<td>0</td>
<td>350</td>
<td>0.86</td>
<td>90</td>
<td>completely unfused</td>
</tr>
<tr>
<td>Horizontal</td>
<td>4000</td>
<td>0</td>
<td>360</td>
<td>1.44</td>
<td>120</td>
<td>uniform</td>
</tr>
<tr>
<td>Horizontal</td>
<td>4000</td>
<td>1/10</td>
<td>366</td>
<td>1.43</td>
<td>116</td>
<td>uniform</td>
</tr>
<tr>
<td>Vertical</td>
<td>3000</td>
<td>0</td>
<td>350</td>
<td>1.50</td>
<td>113</td>
<td>warped grain</td>
</tr>
<tr>
<td>Vertical</td>
<td>3000</td>
<td>0</td>
<td>370</td>
<td>1.58</td>
<td>117</td>
<td>warped grain</td>
</tr>
</tbody>
</table>

* Held at curing temperature for 10 minutes.
SECTION II

INVESTIGATIONS IN MARINE GLUBING

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. L. 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 230° 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.
It has been common practice in the protective coatings industries to follow the polymerisation of resin films by a series of solvent extractions. The use of acetone extraction of glue films has been investigated by R. F. Blomquist, 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 Cassophen LT-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, PH-80, (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.
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-aldehydes 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 Casco phen L7-37 with H-18 catalyst is thermoplastic after a pressure of 25-hours at room temperature. That is, the pressured 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

glass bond will shear while being cured after a 24-hour pre-cure 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 further experimentation by the Forest Products Laboratories.

APPARATUS:

The main apparatus used was a battery of solvent extraction units. Each unit consists of a 600 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-alddehyde Cascophen 12-67 adhesive. This procedure did not give very close checks; however, the broad implications of the work are evident. Acetone solubility of a fully cured 12-67 - M-18 film must be almost negligible as shown by the small negative values obtained on 24-hour films at 200° F.
<table>
<thead>
<tr>
<th>Sample</th>
<th>Time of Cure</th>
<th>150°F</th>
<th>160°F</th>
<th>170°F</th>
<th>180°F</th>
<th>190°F</th>
<th>200°F</th>
</tr>
</thead>
<tbody>
<tr>
<td>8 Parts</td>
<td>6 hrs.</td>
<td>22.6</td>
<td>16.4</td>
<td>10.3</td>
<td>11.1</td>
<td>6.6</td>
<td>7.7</td>
</tr>
<tr>
<td>M-15 Accelerator</td>
<td>6 hrs.</td>
<td>28.6</td>
<td>14.8</td>
<td>11.0</td>
<td>12.8</td>
<td>7.1</td>
<td>9.8</td>
</tr>
<tr>
<td>Per</td>
<td>6 hrs.</td>
<td>17.3</td>
<td>17.7</td>
<td>18.5</td>
<td>8.8</td>
<td>6.3</td>
<td></td>
</tr>
<tr>
<td>100 Parts</td>
<td>6 hrs.</td>
<td>19.8</td>
<td></td>
<td>11.1</td>
<td>8.4</td>
<td>2.8</td>
<td></td>
</tr>
<tr>
<td>LT-67</td>
<td>6 hrs.</td>
<td>21.6</td>
<td></td>
<td>7.3</td>
<td>11.3</td>
<td>3.0</td>
<td></td>
</tr>
<tr>
<td></td>
<td>10 hrs.</td>
<td></td>
<td>10.1</td>
<td>9.8</td>
<td>8.9</td>
<td>7.6</td>
<td></td>
</tr>
<tr>
<td></td>
<td>24 hrs.</td>
<td></td>
<td>5.0</td>
<td>2.3</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pure</td>
<td>6 hrs.</td>
<td></td>
<td>4.7</td>
<td>3.8</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>10 hrs.</td>
<td></td>
<td>9.8</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>LT-67</td>
<td>6 hrs.</td>
<td></td>
<td>3.5</td>
<td>4.8</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>8 hrs.</td>
<td></td>
<td>4.4</td>
<td>6.0</td>
<td>8.1</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>10 hrs.</td>
<td></td>
<td>4.1</td>
<td>6.1</td>
<td>8.1</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

TABLE IX  ACETONE EXTRACTION OF LT-67 ADHESIVE
LEGEND
CURING TIME:

○ 6 HOURS - (CURVE)
× 8 HOURS
△ 10 HOURS

FIG. 11 ACETONE EXTRACTION OF LT 67 WITH MB CATALYST
Films of pure LF-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-75-3 adhesive is 90 parts of PR-75-3 liquid resin, 12.5 parts of P-79 powder accelerator, and 10 parts of commercial ethyl alcohol. The liquid PR-75-3 resin had a pH 7.0. 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 X 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 160° F., the time of cure is relatively insignificant up to the limits of this experiment. (80 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 XI 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 XI.

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 FR-75-B ADHESIVE

<table>
<thead>
<tr>
<th>Cure Temp.</th>
<th>% Extractable</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10 hrs.</td>
</tr>
<tr>
<td>140° F.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>-</td>
</tr>
<tr>
<td>150° F.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>-</td>
</tr>
<tr>
<td>160° F.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>-</td>
</tr>
<tr>
<td>180° F.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.4</td>
</tr>
<tr>
<td>200° F.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.2</td>
</tr>
<tr>
<td></td>
<td>0.2</td>
</tr>
<tr>
<td></td>
<td>-0.6</td>
</tr>
</tbody>
</table>

### TABLE XI ACETONE EXTRACTION OF CATAPOD 060

<table>
<thead>
<tr>
<th>Cure Temp.</th>
<th>% Extractable</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>3 hrs.</td>
</tr>
<tr>
<td>140° F.</td>
<td>11.8</td>
</tr>
<tr>
<td>150° F.</td>
<td>12.6</td>
</tr>
<tr>
<td>160° F.</td>
<td>2.2</td>
</tr>
<tr>
<td>180° F.</td>
<td>2.9</td>
</tr>
<tr>
<td>200° F.</td>
<td>2.3</td>
</tr>
</tbody>
</table>
whereas, normal extracted residues remained in their original freely powdered form.

The 160° 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 160° F. and 180° 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 CASEPHEN LE-67 ADHESIVE

The pot-life of mixed Casephen LE-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 64 Ford cup, a Kodak timer, and standard laboratory glassware.

PROCEDURE:

Six 250 ml samples were prepared according to formulation and held at room temperature (60°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 60 ml. of glue through the orifice of the standard 64 viscosity cup.

DATA AND RESULTS:

Table XIII presents the viscosity data for mixed Casephen LE-67 adhesives. The viscosity of the adhesive containing 3% H-10 catalyst increased the most rapidly, the adhesive containing 6% RH-40 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 XIII  DATA AND RESULTS

VISSCOSITY OF MIXED CASCOPHEN LY-67 ADHESIVES
(Temperature Constant at 80°F.)

<table>
<thead>
<tr>
<th>LT-67</th>
<th>LT-67</th>
<th>LT-67</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>% N-II</td>
<td></td>
</tr>
<tr>
<td>Time of Set (Min)</td>
<td>Viscosity in Sec.</td>
<td>Time of Set (Hrs)</td>
</tr>
<tr>
<td>0</td>
<td>120</td>
<td>0</td>
</tr>
<tr>
<td>25</td>
<td>137</td>
<td>1</td>
</tr>
<tr>
<td>50</td>
<td>143</td>
<td>4</td>
</tr>
<tr>
<td>45</td>
<td>153</td>
<td>23</td>
</tr>
<tr>
<td>60</td>
<td>165</td>
<td>26</td>
</tr>
<tr>
<td>75</td>
<td>193</td>
<td>47</td>
</tr>
<tr>
<td>90</td>
<td>224</td>
<td>65</td>
</tr>
<tr>
<td>105</td>
<td>233</td>
<td>75</td>
</tr>
<tr>
<td>120</td>
<td>420</td>
<td>95</td>
</tr>
<tr>
<td>135</td>
<td>500</td>
<td>101</td>
</tr>
<tr>
<td>150</td>
<td>1069</td>
<td>143</td>
</tr>
<tr>
<td>180</td>
<td>solid</td>
<td>167</td>
</tr>
<tr>
<td></td>
<td></td>
<td>72</td>
</tr>
<tr>
<td></td>
<td></td>
<td>96</td>
</tr>
<tr>
<td></td>
<td></td>
<td>108</td>
</tr>
<tr>
<td></td>
<td></td>
<td>144</td>
</tr>
<tr>
<td></td>
<td></td>
<td>168</td>
</tr>
</tbody>
</table>
### TABLE XIII (Cont'd.)

**Viscosity of Mixed Gascohen LR-67 Adhesives**

<table>
<thead>
<tr>
<th>LR-67</th>
<th>LR-67</th>
<th>LR-67</th>
</tr>
</thead>
<tbody>
<tr>
<td>6% FH-50</td>
<td>6% FH-50</td>
<td>6% FH-50</td>
</tr>
<tr>
<td>0</td>
<td>122</td>
<td>0</td>
</tr>
<tr>
<td>1</td>
<td>135</td>
<td>1</td>
</tr>
<tr>
<td>2</td>
<td>155</td>
<td>1.5</td>
</tr>
<tr>
<td>3</td>
<td>155</td>
<td>3.5</td>
</tr>
<tr>
<td>4</td>
<td>155</td>
<td>6</td>
</tr>
<tr>
<td>5</td>
<td>157</td>
<td>10</td>
</tr>
<tr>
<td>6</td>
<td>127</td>
<td>20</td>
</tr>
<tr>
<td>6</td>
<td>126</td>
<td>42</td>
</tr>
<tr>
<td>51</td>
<td>180</td>
<td>54</td>
</tr>
<tr>
<td>46</td>
<td>172</td>
<td>72</td>
</tr>
<tr>
<td>84</td>
<td>180</td>
<td>90</td>
</tr>
<tr>
<td>72</td>
<td>194</td>
<td>109</td>
</tr>
<tr>
<td>95</td>
<td>288</td>
<td>146</td>
</tr>
<tr>
<td>103</td>
<td>278</td>
<td>160</td>
</tr>
<tr>
<td>144</td>
<td>340</td>
<td></td>
</tr>
<tr>
<td>195</td>
<td>393</td>
<td></td>
</tr>
</tbody>
</table>

* Time of flow for 50 ml from 4" standard Ford Viscosity Cup.
LEGEND
CURVES:
A  LT67 + 8% H-18
B  LT67 + 8% FM50
C  LT67 + 6% FM50
D  LT67 + 4% FM50
E  LT67 + 2% FM50
F  LT67 PLAIN

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

TIME - HOURS

VISCOITY - NO. 4 FORD CUP SECONDS
LEGEND

X LT67 PLAIN
O LT67 AND 8% M/10
□ LT67 AND 8% FM50

VISCOITY - NO. 4 FORD CUP SECONDS

TIME - MINUTES

FIG. 14. VISCOITY CHANGES ON LT67 ADHESIVES AT 80°F. 30 ML. THROUGH NO. 4 FORD CUP
CONCLUSIONS:

The presence of H-16 catalyst greatly accelerates the tendency of LT-67 to become viscous. At 80°F, under the conditions of test it became too viscous for normal spreading within two hours after mixing (600, 4 Ford cup seconds).

The use of catalyst EF-50 produced an initial fast rise in viscosity, followed by a long period of further slow gain. An 8% catalyst mix reached 600 4 Ford cup seconds only after about 70 hours, a 6% mix at 118 hours, and a 4% mix at 170 hours. The plain LT-67 which was held as a control started at 85 seconds and even after 170 hours (1 week) only reached 225 seconds.
PLASTICITY OF LX-67, GASCOPHEN, 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 L. 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.g., 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 unsaturated water.
White oak blocks, 5/8" x 5/8" x 3/4", were glued with 12-67, H-16 catalyst, in sets of three, the center block extending 1/8" 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.)

Pressed glued blocks were placed inside the heating elements in the Bushler 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 5°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 0.002" - 0.006". All blocks were pressed 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 250°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 visibly at approximately 215° 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 LF-67 DURING CURE**

<table>
<thead>
<tr>
<th>Applied Shear P.S.I.</th>
<th>Temperature at yield point — ° F.</th>
</tr>
</thead>
<tbody>
<tr>
<td>25.6</td>
<td>No yield point – sample cured</td>
</tr>
<tr>
<td>26.8</td>
<td>No yield point – sample cured</td>
</tr>
<tr>
<td>28.0</td>
<td>125° F.</td>
</tr>
<tr>
<td>30.7</td>
<td>117° F.</td>
</tr>
<tr>
<td>32.9</td>
<td>108° F.</td>
</tr>
<tr>
<td>36.4</td>
<td>106° F.</td>
</tr>
<tr>
<td>69.0</td>
<td>94° F.</td>
</tr>
<tr>
<td>115.0</td>
<td>70° F.</td>
</tr>
</tbody>
</table>
FIG. 15  STRENGTH OF LT67 DURING CURE
24 HOURS ROOM TEMPERATURE PRECURSE
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 120° F. Increasingly higher shear values caused lower temperatures of failure in an apparent hyperbolic function. This experiment shows a definite softening of the pressed 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 bands was as follows:

§1 Unit compression 400 P.S.I. Glue line bowed slightly upon application of pressure and expansion proceeded slowly for 20 minutes until 200° F. was reached. No further expansion took place but the glue line had bowed out about 1/34".

§2 Unit compression 200 P.S.I. Slight bowing of glue line when 180° F. was reached.

§3 Unit compression 100 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 LP-87 with H-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 30 - 35 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 30 - 35 P.S.I. limit, although this will apply only to external or thin walled 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 130° 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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