



angle, and dissipation of the glued assembly placed between its electrodes. Capacitance was found to be the property that was most sensitive to changes in the wood-adhesive composite during the cure process.

The dielectric spectrometer was able to accurately and consistently measure the capacitance of oven dried wood. Dielectric measurements were very sensitive to the presence of water, and the increase in capacitance with an increase in moisture content was reproducible. Problems were encountered when using the dielectric spectrometer to monitor cure of a resin in wood composites. The lack of repeatability in capacitance measurements led to the conclusion that the Audrey II dielectric spectrometer could not measure cure of a highly caustic phenolic adhesive or a resorcinol-formaldehyde laminating adhesive in Douglas-fir gluelines.

Components of a phenol-formaldehyde resin were examined to determine what caused the lack of reproducibility. The capacitance was reproducible with either water or aqueous caustic on the veneers. However, the presence of either phenol or formalin on the veneers resulted in a total lack of repeatability in capacitance. Both phenol and formalin are excellent swelling agents for wood and as wood swells, its microstructure is opened and plasticized. This would increase the capacitance. Other factors that would influence variation in swelling and capacitance would include depth of lathe checks, wood damage, porosity, density, and natural anatomical variation that influences the rate of penetration of chemicals into wood. Even considering the swelling effects and natural variation

of wood, the variability in capacitance of the wood-adhesive composite is not readily understood.

Measuring Adhesive Cure by Dielectric Analysis

by

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Dedicated to my wife, Patty, who provided encouragement and support throughout the preparation of this thesis.

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## Measuring Adhesive Cure by Dielectric Analysis

### I. INTRODUCTION

In recent years, the consumer has increasingly demanded reliable, long lasting products. Failing to meet these expectations often resulted in costly court battles and lost markets for wood products.

The durability of plywood panels is determined mostly by the quality of the glueline which bonds the composite together. The more commonly used methods of evaluating glueline quality include gel time of the adhesive, its water solubility, thermal softening of the glueline, press time studies, and glueline microscopy. These methods all estimate completeness of glueline cure, but, as explained in the following sections, are not adequate in either production or laboratory situations.

#### Gel Time

Studying adhesives by the gel time method is an attempt to simulate glueline gelling in a laboratory environment. The Monsanto Chemical Company (Anon., 1958) developed a laboratory technique similar to many for determining adhesive gel time. In summary, test tubes filled with a small quantity of liquid adhesive are immersed in a hot water bath and the time to solidification is measured. Some questions can be answered objectively by the effect adhesive variables have on gel time. Most commonly, studies are conducted to test the effect of varying the temperature, catalyst, percent of solids, or resin formulation. The gel time for a glueline is presumed to

approximate the gel time of a test sample under the same conditions. But adhesive cure in the glueline involves water loss as well as chemical crosslinking. Because little water is lost from a gel tube, no variable affecting water loss from gluelines can be evaluated by the gel time method.

### Water Solubility

The water solubility technique, reported by Chow and Hancock (1969), is an accurate way to determine the degree of cure of a phenol-formaldehyde resin on some species. This method, also known as the spectrophotometric method, involves scraping the glueline from a panel after it has been removed from the hot press and dispersing the phenolic residue in distilled water.

"The degree of cure of a glue or neat resin is related to its solubility in distilled water, the solubility being measured from the absorbance ratios of bands at specific wave lengths in the ultraviolet spectrum."<sup>1</sup>

This method can be used only if extractive materials in the wood do not interfere with the ultraviolet analysis, thus limiting the method's use with many species.

### Thermal Softening

The degree of resin cure can be determined by the thermal softening method as stated by Rosenberg (1978) and Chow (1973). This method also involves analysis of a sample taken from a cured glue-line. The flow properties of this glue sample are measured as the

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<sup>1</sup>Chow and Hancock, 1969, p. 21.

sample is heated using a linear temperature programmer. Resin that is not completely crosslinked (cured) will have a greater ability to melt and flow at any particular temperature. Thus, an empirical correlation between resin flow and resin cure can be derived. But this correlation still requires some other reliable method of measuring resin cure. And the flow characteristics of a resin sample will vary depending on the amount of wood fiber imbedded in the cured resin.

### Press Time Studies

Press time studies are extensively used throughout the plywood industry to determine plywood bond quality. Panels are formed and pressed for varying lengths of time. Test specimens are cut from the plywood panels and subjected to accelerated aging tests as specified by the U.S. Product Standard PS 1-74 (National Bureau of Standards, 1974). The aged specimens are then sheared and the gluelines are examined to estimate percent wood failure and, occasionally, bond strength. Unfortunately the results of such tests are not available until one-to-three days after the plywood has been made. Such delays prohibit process changes that would correct production problems in a timely manner.

### Glueline Microscopy

Examining plywood under a low power microscope is still another method of studying glue bonds as reported by Hancock and Northcott (1961). Two key features, glue migration into lathe checks and

discontinuities in the glueline, can be estimated but plywood bond quality depends on much more.

The primary disadvantage with all of these methods is that the wood composite must be removed from the press and examined using time consuming techniques, allowing only a sample of the composite to be examined. Destructive testing of a product prohibits large scale sampling and becomes very expensive for the manufacturers. Also, such "hit and miss" techniques only allow adjustments on future production. The real need is for non-destructive techniques that can be used "in-process."

#### In-Process Methods

Monitoring the glueline while the wood composite is still in the press would provide many advantages over existing methods. Such a system would provide greater control over the degree of cure, helping to eliminate variable quality in the glue bond. "In-process" methods for monitoring glueline cure have not been overlooked. Historically, much attention has been focused on developing methods and techniques for examining adhesive cure in-process.

Previous studies cited by Chow, Troughton, Hancock, and Mukai (1973) have shown that bond quality correlates well with inner glue-line temperatures, providing none of the other process variables that affect gluing are out of control. Although this test method can provide limited, but valuable, in-process data, the insertion of a thermocouple wire into the inner glueline presents obvious production problems and its practical use is restricted to laboratory experiments.

A laboratory technique for measuring glueline viscosity was developed by Freeman and Kreibich (1968). This technique measured the torque of a single open glueline placed on a hardboard substrate which was mounted on a rotating hot plate. The glueline viscosity was found to provide valuable insights into plywood bond quality, but does not allow imitation of "real world" situations.

Pillar (1966) used a dynamic mechanical test to study the curing properties of an adhesive in contact with wood. A device was constructed to vibrate and measure the mechanical strain of a thin sheet of veneer covered on both sides with a room temperature curing glue. At different time intervals the dampening constant and flexural modulus were measured. As the adhesive cured the veneer composite became less flexible. The mechanical properties were highly correlated to the extent of cure of the adhesive.

Although all of these methods measure in-process changes of a wood composite they are highly unsuited for industrial applications. They require closely controlled laboratory conditions, time consuming operating procedures and in-process "baby sitting".

The ideal method for monitoring resin glues in-process would be characterized by the following traits:

1. is interfaced with the production process,
2. provides immediate and continuous feedback,
3. is non-destructive to the sample,
4. can operate in a noisy environment, and
5. is easy and reliable to use.

One alternative that may have these traits is to monitor the dielectric properties of the composite as the adhesive cures.

## Dielectric Monitoring

By using the hot press as the plates of a condenser separated by a composite panel as a dielectric medium, dielectric properties (e.g. capacitance, dissipation, etc.) of the composite panel can be measured as the adhesive cures, enabling estimates of the percent of cure. Such techniques have been explored by Miller and Cole (1957), Yalof (1975a, 1975b, 1975c, 1976, 1977) and others. Electronic instruments can be interfaced easily with production equipment and rapidly yield continuous data from measurements that are non-destructive to the sample.

Recently, Tetrahedron Associates, Inc.<sup>2</sup> began advertising the Audrey II dielectric spectrometer for use as a dielectric monitor in many commercial gluing processes including those used in the wood industry. However, the Audrey II has never been used to monitor a wood gluing process. Before the wood industry invests in such equipment, verification is needed that the Audrey II will work in an industrial wood gluing process.

## Objectives

The following major objectives were established for this investigation:

1. Determine if the Audrey II dielectric spectrometer can measure cure of a highly caustic phenolic plywood adhesive in Douglas-fir gluelines under conditions analogous to mill

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<sup>2</sup>Tetrahedron Associates, Inc., San Diego, Ca.

production (i.e. 150°C and 200 psi press temperature and pressure).

2. Determine if the Audrey II dielectric spectrometer can measure cure of a resorcinol-formaldehyde laminating adhesive in a Douglas-fir glueline at ambient temperature and pressure of 200 psi.

Both objectives were satisfactorily completed during the course of this investigation.

## II. REVIEW OF LITERATURE

Dielectric spectroscopy of gluelines involves measuring selected dielectric properties of the composite. This chapter will explain what a dielectric material is, describe its electric properties and review previous research related to the dielectric properties of gluelines.

### Dielectric Concepts

For the past couple of decades high frequency electromagnetic radiation has been used in the forest products industry as a means of drying wood and curing adhesives. In drying, this method increases the internal temperature of the wood by molecular friction and causes drying from the "inside out". The underlying principle is the same for any dielectric material (e.g. wood, adhesives) placed between two electrodes receiving an alternating current. The alternating current induces internal vibration of the molecules in the dielectric, producing heat from the molecular friction.

Most dielectric materials contain polar molecules. Each polar molecule is made up of oppositely charged segments localized at a site. These polar segments of molecules are called dipoles. The electrical properties of a dielectric are contingent upon the dipoles' ability to respond to alternating current. "Dipoles within a dielectric attempt to stay in alignment with an applied electric field."<sup>3</sup> If no external electric field is applied the dipoles may be randomly oriented as shown in Figure 1-2. If an external electric

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<sup>3</sup>Yalof, S. and D. Brisbin, 1973, p. 65.

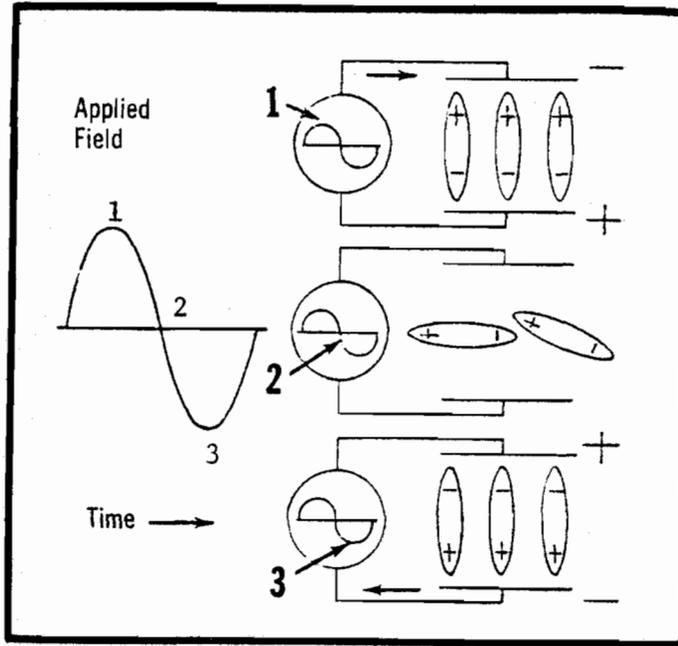


Figure 1. Alignment of dipoles within a dielectric.

field is applied the dipolar molecules tend to align themselves with the electric field as shown in Figure 1-1. The positive pole of the dipole will be attracted to the negative terminal and vice versa. If the polarity of the electric field is reversed the dipoles will re-align to oppose the electric orientation of the terminal as shown in Figure 1-3.

When the dipolar molecules re-align with the electric orientation, most of the energy supplied from the alternating current is stored in the dipolar molecules as potential energy. The remaining portion of the energy supplied is dissipated as heat in raising the energy levels of adjacent molecules due to molecular friction.

Dielectric monitoring is the opposite of dielectric heating. If a very weak, high frequency signal is applied across a dielectric, one can measure selected dielectric properties as estimates of the molecular response of the dipoles to the alternating current. The properties measured are voltage, capacitance, dielectric constant, dissipation, and phase angle.

Voltage. The difference in electrical potential energy between two condenser plates is the voltage, whether in an alternating current or a direct current system.

Capacitance. The quantity of electricity being stored in a condenser is its capacitance.

When a voltage (E) is applied to a parallel plate condenser electrons flow from one plate to the other. The flow continues until a sufficient number of electrons has moved from the one plate to the other to cause a difference in electrical potential equal to the voltage. The quantity of electricity stored as the excess of

electrons on one plate is the electrical charge  $Q$ . The ratio of the charge  $Q$  to the voltage  $E$  is the capacitance  $C$  of the condenser (Equation 1) when  $C$ ,  $Q$ , and  $E$  are expressed in farads, coulombs, and volts, respectively (Brown, Panshin, and Forsaith, 1952).

$$C = \frac{Q}{E} \quad (1)$$

The capacitance of a condenser depends on the dielectric placed between the condenser plates. As illustrated in Figure 2, the molecules of a dielectric such as wood orient to neutralize part of the charge on the condenser plates. As a result additional electrons flow from plate 1 to plate 2 until the difference in electric potential again equals the voltage  $E$ . The more polarized the dielectric, the greater the capacitance of the condenser.

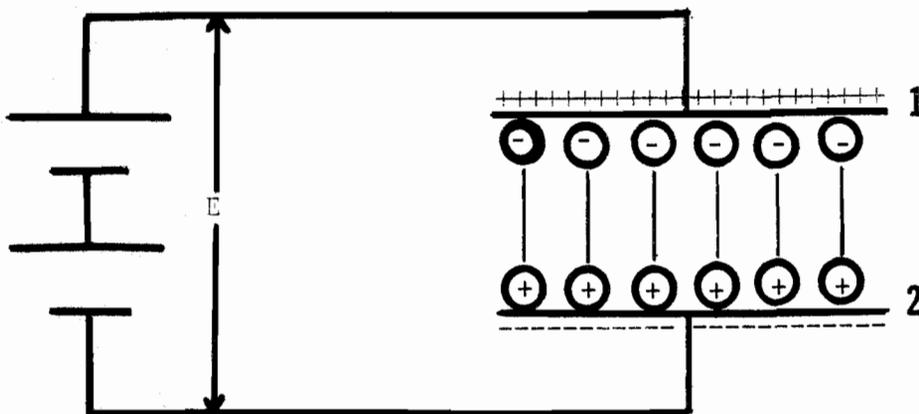


Figure 2. The increase in the electrical charges on condenser plates to neutralize the charges on the wood molecules.

Dielectric Constant. The increased capacitance of a condenser with a dipolar material (compared to a vacuum) is a property of the dielectric material. The ratio of the capacitance of a condenser with a dielectric to the capacitance of the same condenser in a vacuum is the dielectric constant (Equation 2) (Brown, Panshin, and Forsaith, 1952):

$$\epsilon = \frac{C_d}{C_o} \quad (2)$$

where  $\epsilon$  is the dielectric constant,  $C_d$  is the capacitance of a condenser with a dielectric and  $C_o$  is the capacitance of the same condenser in a vacuum.

Dissipation. The dielectric dissipation in a dielectric material is the ratio of the electrical energy dissipated as heat per cycle of oscillation in a condenser, to the total electrical energy stored in the condenser during the cycle.

Phase Angle. In a capacitor, the voltage increases so long as current flows into the capacitor; conversely, voltage decreases when current flows out of the capacitor. Figure 3 displays this relationship between current I and voltage E in a capacitor. The phase angle is that angle by which the current leads the voltage in a capacitor. In a perfect capacitor the phase angle is  $90^\circ$ .

### Dielectric Properties of Wood

Both the wood and the adhesive in a glueline are dielectric materials having each of the properties described in the previous section. To estimate adhesive cure from dielectric properties

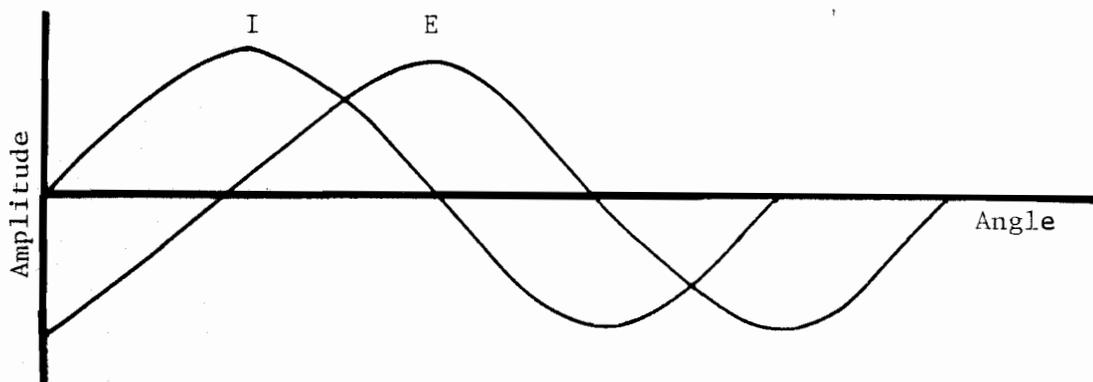


Figure 3. The relationship between current (I) and voltage (E) in a circuit.

requires that the change in dielectric properties during adhesive cure be distinguishable from the dielectric response of the wood.

Lin (1965, 1967a, 1967b) has made detailed reviews of the electric and dielectric properties of wood. The results show that the dielectric properties of wood are strongly dependent on moisture content and frequency and to a lesser degree on temperature. Lin concludes that the dielectric properties of wood increase with increasing moisture content, increase with increasing temperature, increase with increasing density and decrease with increasing frequency.

James (1975, 1977) has focused on the responses of wood at different moisture levels, temperatures, and times. James' research confirms Lin's findings (Table 1) and also states that wood with very high moisture content displays similar dielectric properties to that of water.

TABLE 1. SUMMARY OF DIELECTRIC PROPERTIES OF WOOD.

Properties	Increase			
	Temp.	M.C.	Freq.	Wood Density
Capacitance	increase	increase	decrease	increase
Dissipation	increase	increase	decrease	increase
Dielectric Constant	increase	increase	decrease	increase
Voltage	----	----	----	----
Phase Angle	----	----	----	----

Brown, Panshin and Forsaith (1952) state that the dielectric constant of water is approximately 81 at frequencies less than 1000 megahertz. They also state that oven-dry wood with a density of  $0.5 \text{ gm/cm}^3$  has a dielectric constant of approximately two. As wood increases in moisture content, its dielectric constant increases rapidly, as shown in Figure 4. Fortunately, the dielectric properties of a curing adhesive are reported to change more than these properties change for wood, making possible the dielectric monitoring of adhesive cure.

#### Dielectric Monitoring of Adhesives

Based on dielectric theory we would expect to observe several trends in the dielectric properties of an adhesive as it cures. At the time of adhesive application the adhesive liquid is low in degree of polymerization and it is most capable of responding to the alternating current at frequencies within the radio frequency range. During the early stage of resin cure, there should be substantial alignment and heat loss (dissipation) generated by the rotating molecules. High capacitance and dissipation readings would be

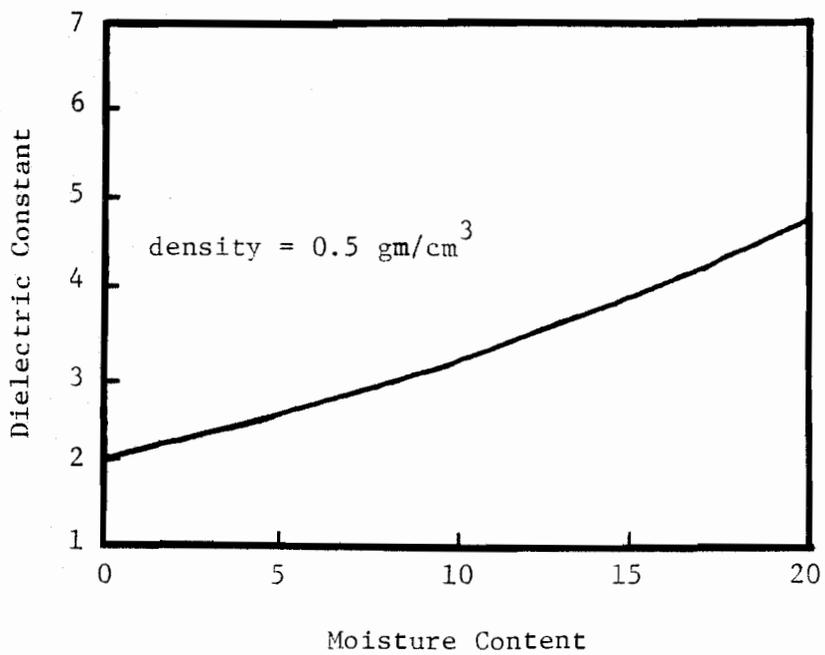


Figure 4. The relationship between the dielectric constant of wood and its moisture content (Brown, Panshin, and Forsaith, 1952).

observed. As the adhesive advances in molecular size, its ability to align with an alternating current should decrease. This lack of response should first be detected as a lowering in the capacitance and dissipation at the higher frequencies and followed by similar observations at the lower frequencies. At the point of complete cure there should be no further increases in molecular size of the polymer. This should be represented by a stabilization of the dielectric properties. The capacitance and dissipation should dip to a low and remain constant.

Miller and Cole (1957) were pioneers in dielectric monitoring when they measured the dielectric properties of resin glues for wood. Their objectives were similar to the ones outlined in this study--to dielectrically monitor adhesive behavior to full cure. A coaxial-type sample holder was used as a test cell for the glue samples. This provided a shielded environment that could be externally heated. The equipment used for their experiment consisted of an impedance measuring circuit, bridge oscillator, receiver and an oscilloscope.

Miller and Cole measured the conductivity and dielectric constant at the megahertz ( $10^6$ ) frequency range and concluded that the conductivity and dielectric constant showed no significant changes with cure time, temperature or frequency. Perhaps this lack of dielectric response can be attributed to monitoring the glue at excessively high frequencies. The dielectric response associated with curing resin has been reported (Yalof, 1975d) to occur normally at much lower frequencies ( $10^2 - 10^3$  hertz). Although Miller and Cole were unable to track resin curing using "state of the art" electronic equipment,

they made many valuable contributions for future research efforts in this field.

Advances in developing new electronic equipment have enlarged the scope of recent research efforts. Yalof (1975a, 1975b, 1975c) has been instrumental in designing equipment to non-destructively measure specific dielectric properties with ease and accuracy. Yalof and Brisbin (1973) have shown both theoretically and experimentally that mechanical and dielectric properties are interrelated. Yalof and Zika (1975) compared dielectric spectroscopy to other types of spectroscopy and were able to reach similar conclusions with the different methods. Yalof (1975d) used an electronic temperature programmer to slowly increase the temperature, controlling the rate of cure, and dielectrically monitoring cure of a wide range of non-aqueous polymers such as: epoxies, polyesters, pyriones, silicones, phenolics, sheet molding compounds, polyamides, and polyvinyl chlorides.

Successful monitoring of an adhesive cure has been reported by measuring capacitance, dissipation, temperature and frequency. Jemal (1974) used an epoxy resin that cured in five minutes as shown in Figure 5. In approximately five minutes both the capacitance and dissipation leveled off, indicating little dielectric change in the adhesive after the cure had been completed.

The Air Force Materials Laboratory (Arvay and Centers, 1974) used an automatic dielectrometer to monitor the cure of a structural adhesive during an in-process bonding operation. The press system required considerable development in insulating and shielding to obtain relatively noise free dielectrometer traces while operating

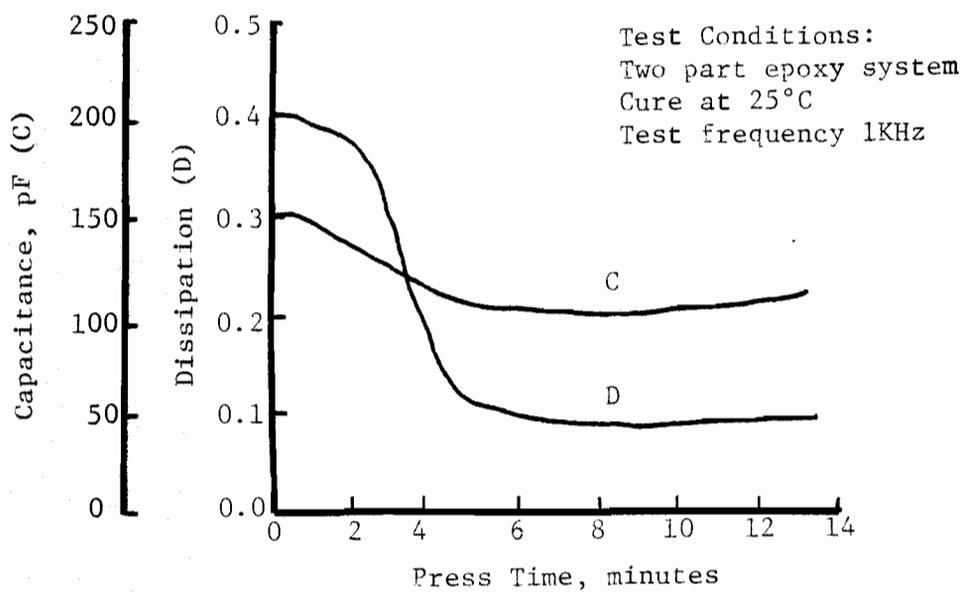


Figure 5. Audrey analysis for five minute epoxy.

in the electronically noisy environment of a heated platen, hydraulic press. Conclusive curves were obtained on both an epoxy type and a polyamide type adhesive system under a constantly increasing temperature cure cycle.

A wide range of thermosetting polymers has been examined by dielectric spectroscopy. Headvig (1977) used a carefully isolated test cell and a linear "up and down" temperature program to follow the adhesive reaction until the resin became completely rigid. His present efforts are in developing new methods to detect adhesive cure by measuring the dielectric properties and the total conductivity as a function of temperature at different stages of cure. The adhesives studied are non-aqueous in nature.

Numerous accounts of research in the aerospace industry (Arvay and Centers, 1974; Carpenter, 1976; Crabtree, 1977; Fritzen, Wereta, and Arvay, 1977; May, 1975; May, Whearty, and Fritzen, 1976) have successfully used dielectric analysis to monitor resin cure in process. Specially designed test environments and the use of non-aqueous resins have contributed to their success.

Yalof and Wrasidlo (1972) used dielectric thermal analysis (DTA) to monitor adhesive behavior. This technique uses temperature cycling, i.e., heating the polymer to a particular temperature and allowing it to cool. The polymers are then tested in a specially designed dielectric-DTA cell.

"Each cycle produces a loop and the circumscribed area of each loop decreases with each succeeding cycle, until finally the enclosed area drops to zero. At this point, the resin is fully cured up to a particular temperature. By cycling to a higher temperature, further curing reactions are

produced and we again generate a circle loop circumscribing a certain area."<sup>4</sup>

The resin may be considered fully cured if there is no circle loop produced when the temperature is elevated. An example of the dielectric thermal analysis is shown in Figure 6.

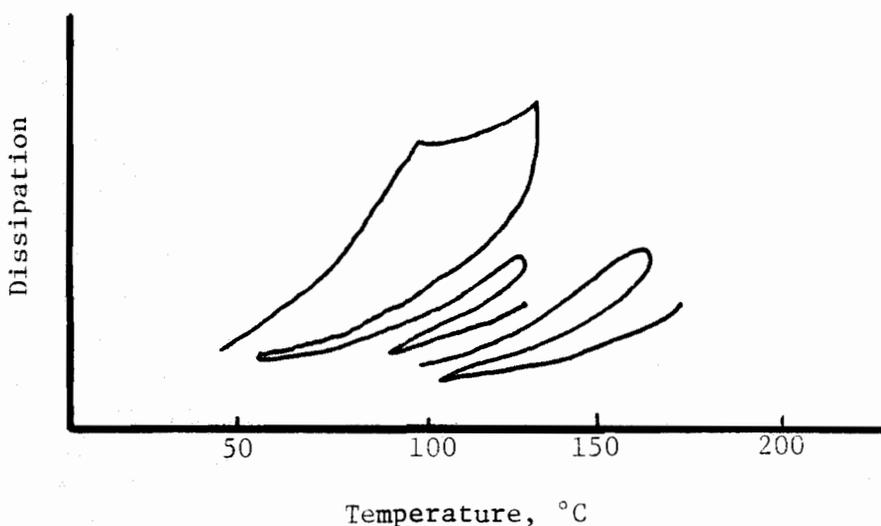


Figure 6. Curing loop for phenolic resin.

An Audrey dielectrometer was used by Fritzen, Wereta, and Arvay (1977) to investigate adhesive cure. Capacitance, dissipation, phase angle, and voltage were measured and the trends examined. Aluminum adherends, graphite/epoxy laminates and titanium adherends were studied in an electrically isolated environment. Test conditions consisted of using aluminum foil electrodes, adjusting the frequency (100-1000 Hz) to optimize the response of a particular adhesive, and carefully controlling the temperature. Favorable results were

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<sup>4</sup>Yalof and Wrasidlo, 1972, p. 2163.

reported for monitoring the adhesive bonding process with both the dielectrometer and the phasemeter.

Few experiments have involved dielectric tracking of aqueous adhesives. Headvig (1977) conceded that water in polymeric systems affects the ability to monitor dielectric properties. In contrast Yalof (1975a, 1975b, 1975d) claims that the dielectric spectrum from water is in a higher frequency range than that used for polymers, allowing monitoring of aqueous adhesives.

In summary, previous research has focused primarily on bonding non-porous adherends with non-aqueous resins in carefully designed test cells. Although dielectric spectrometers are now manufactured and recommended to the wood industry for monitoring aqueous adhesives, their applicability has never been established in the published literature. This study tested adhesives common in the plywood and laminated beam industries. These adhesives generally range in water content from 25 to 45 percent. Wood, a porous material, was used as the adherend and a miniature production hot press was used as the sample holder. Thus this study was the first systematic evaluation of dielectric monitoring of aqueous adhesives in a wood gluing system.

### III. MATERIALS AND METHODS

#### Equipment

A schematic drawing of the experimental setup is shown in Figure 7. Basically, the equipment included the Audrey II dielectric spectrometer to measure dielectric responses, a recorder, a hot press, and a millivolt potentiometer used as an external power source to drive the recorder at the desired chart speed.

The Audrey II dielectric spectrometer was designed to monitor dielectric properties as related to cure of a polymer. Its features are as follows.

#### Dielectric Measuring Ranges:

Capacitance---50 to 500 picofarads.

Dissipation---0.0 to 1.0 on the linear scale.  
0.0 to infinity on the non-linear scale.  
Measurements on the non-linear scale are  
non-linear and non-calibrated.

Voltage ---0.0 to 1.0 volts A.C.

Phase Angle---  $-90^\circ$  to  $+90^\circ$ .

Frequency Range: 100 to 1000 Hz from an internal oscillator.

Internal Thermocouple (TC) Amplifier.

The Audrey II spectrometer was interfaced with a multi-channel recorder. Four channels could be in operation simultaneously. Two of the channels were always monitoring temperature and frequency; the other two channels monitored either capacitance and dissipation or voltage and phase angle. By the design of the Audrey II equipment it was not possible to monitor more than two dielectric properties at a time. The lowest internal speed on the recorder produced graphs

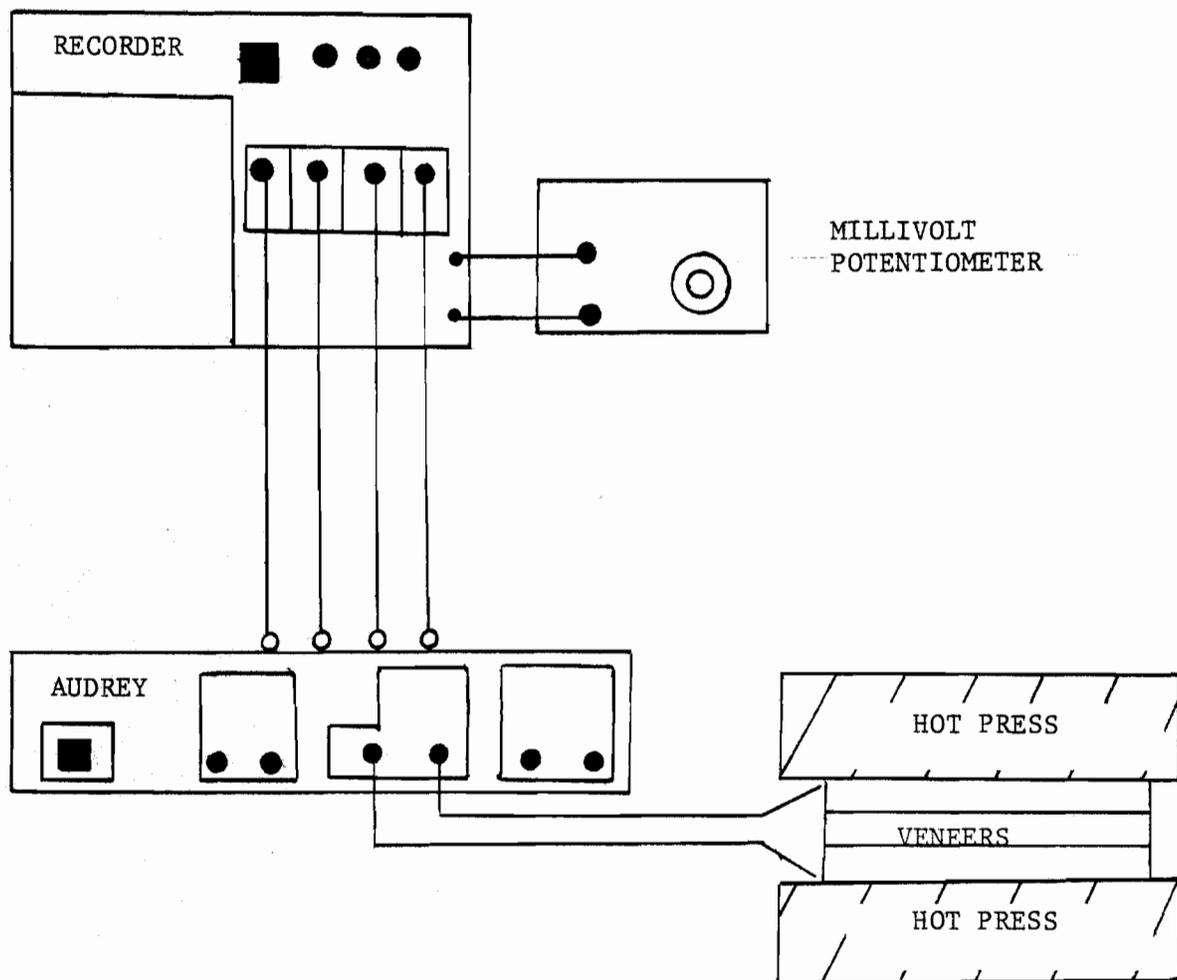


Figure 7. Schematic diagram of equipment setup.

much longer than desired. A millivolt potentiometer was used as an external power source to drive the recorder at the desired rate (13mm/minute) for purposes of condensing the size of the graphs. Operating at this low speed often caused paper friction and was responsible for occasional variation in the distance between gradations on the time axis.

### Electrodes

An isolated system was important when making dielectric measurements. Isolation of the plywood panel was achieved by multiple-layers of electrically resistant materials between the electrodes and metal plates of the hot press. This sequence of layering can be viewed in Figure 8. Immediately next to the hot press plates were two sheets of asbestos followed by a film of kapton. Then the aluminum foil electrodes were placed on top and bottom of the wood composite.

The two opposing electrodes were visually aligned. The area that was overlapped by the two opposing electrodes was the area being monitored. A copper wire was placed on each electrode, 25 mm from the edge on alternate edges, to provide an attachment point for the cables from the Audrey dielectrometer. Because the electrodes were on the outside of the wood laminate the dielectric measurements were an average of the changes that occur in the panel. A thermocouple (TC) wire was placed in one of the glue lines to monitor glue line temperature.

Other electrode configurations were used but these systems will be mentioned specifically when they are pertinent to individual experiments.

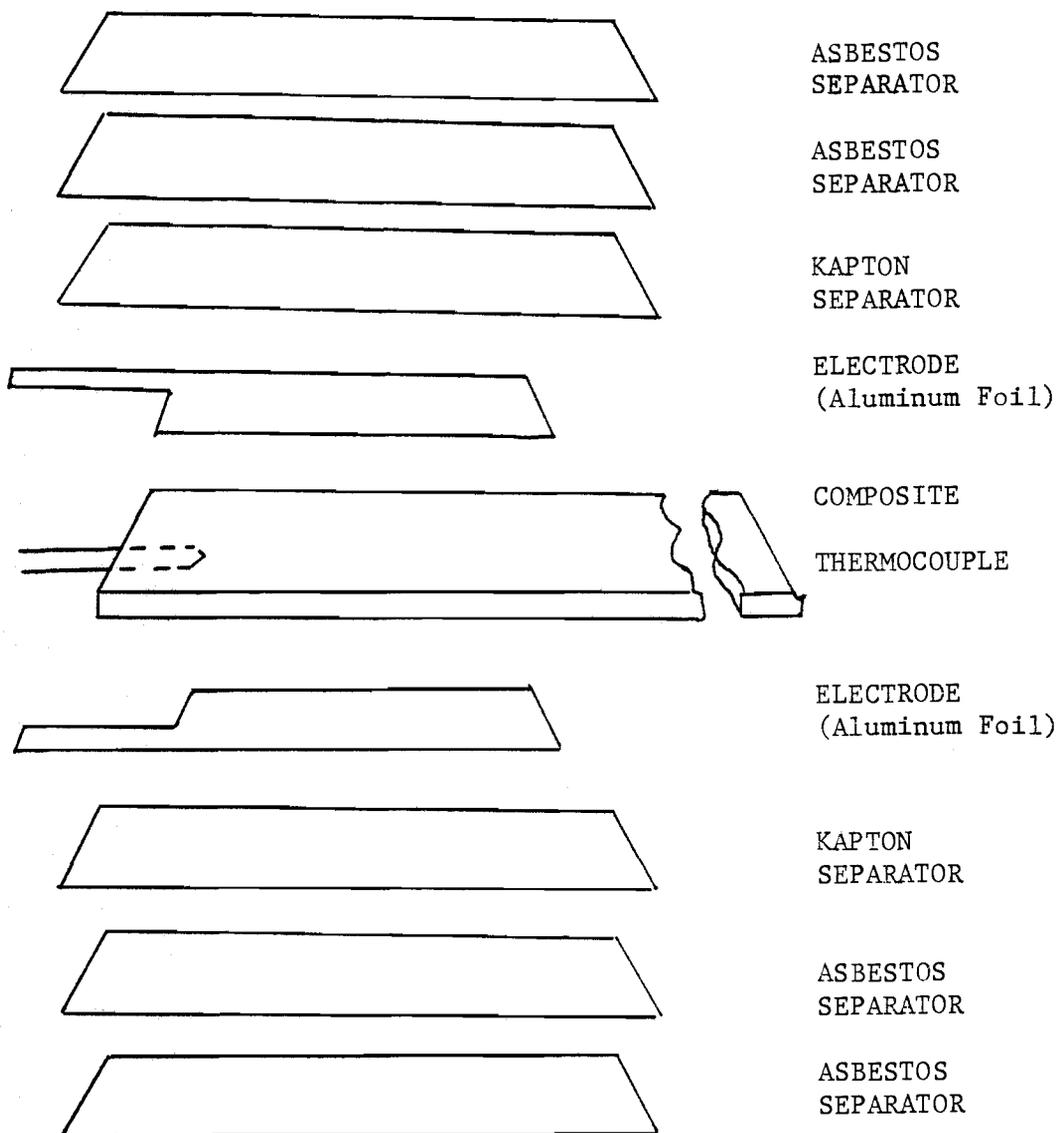


Figure 8. Capacitive pair electrode.

The size of the electrode varied with the type of the adhesive being monitored. Each adhesive had a unique blend of ingredients that gave each mix its own specific dielectric properties. Electrode sizes and shapes were experimentally determined for each polymer studied. A 191mm X 292mm electrode was used for the phenolic and resorcinol resins, and a 178mm X 178mm electrode was required for urea resins. All electrodes were rectangular except when copper wires were used as electrodes to monitor resin behavior within the same glue-line.

### Wood

Grade N Douglas-fir [Pseudotsuga menziesii (Mirb.) Franco] was used. Veneers were selected to minimize the effect of variation associated with wood. A 0.61m X 0.61m sheet of 2.5mm thick veneer was quartered, producing four samples. These sheets were labeled for future matching and conditioning to the desired moisture content (M.C.%). Veneers were conditioned for approximately four weeks in one of two conditioning chambers--an equilibrium moisture content (EMC) of 6% or an EMC of 10%. The formula used for determining the moisture content was as follows (Panshin and de Zeeuw, 1970):

$$\text{M.C.\%} = \frac{\text{weight at test} - \text{weight oven dry}}{\text{weight oven dry}} \times 100 \quad (3)$$

Wood conditioned to 6% M.C. was used for all experiments except when otherwise specified.

Phenolic and urea resins were supplied by various manufacturers. Adhesives were prepared from these resins by adding fillers immediately prior to gluing and according to the manufacturer's

specifications. Table 2 is a summary of the adhesive information.

TABLE 2. PROPERTIES OF RESINS AND ADHESIVES USED FOR DIELECTRIC MONITORING.

Systems	Viscosity (centipoise)	pH	Solids (%)	Mfg. and Number
Phenolic resin	800	12.1	42	Monsanto PF-541
Urea resin	2500	7.8	67	Bordon CR-5H
Phenolic adhesive	1400	11.3	45	Monsanto PF-3102
Resorcinol adhesive	5500	7.5	--	Monsanto RF-900
Phenolic dry film	--	--	100	Reichhold 42-200

#### Adhesive Application and Pressing

A variety of methods were utilized to apply the resins and adhesives. The viscosity of the system dictated which methods were used. An air pressure spray gun was used to apply the phenolic and urea resins. The spread weights were 377 grams per square meter of double glueline for both of these resin systems. Phenolic adhesives were applied by a rubber roll glue spreader. Spread weights were consistently at 398 grams per square meter of double glueline. Resorcinol adhesives were applied by hand using a scraper. This resin system used 366 grams per square meter of single glueline. This technique was developed because of the extremely high adhesive viscosity. The dry film phenolic resins needed only to be placed

between the sheets of veneer. The average weight of a resin sheet was 75 grams per square meter.

A 15 minute assembly time (time between glue application and pressing) was used for all studies involving wood composites except when examining the effect of varying assembly time.

All panels were pressed at a pressure of 200 psi for sufficient time to cure the adhesive. The specific hotpress times and temperatures are indicated as results are presented.

### Panels

Two, three and five ply composites were prepared. The two ply construction was made of parallel veneers. The three and five ply constructions were assembled by alternating the grain 90 degrees in adjacent layers. Unless otherwise specified, three ply panels were used for the dielectric experiments.

#### IV. RESULTS AND DISCUSSION

Many factors greatly influence measurements of dielectric properties. The most influential factors include the following:

1. Electrode size
2. Distance between electrodes
3. Dielectric materials
4. Moisture content of the dielectric

All of these variables needed to be determined experimentally, in light of the machine limitations, before pursuing the primary objectives of this study.

##### Equipment Checkout

To assure that the Audrey instrument recorded reliable data, it was necessary to measure the electrical properties of some knowns. Much research was available on the dielectric constant of wood over a complete range of moisture contents. The Audrey machine was demonstrated to be in calibration by comparing the literature values shown in Figure 4 to experimental values at three different moisture contents; zero, six, and ten percent (see Table 3).

The dielectric constant was calculated for the test specimens using the following equation (Brown, Panshin, and Forsaith, 1952):

$$E = \frac{C_d}{C_o} \quad (4)$$

where E is the dielectric constant and  $C_d$  and  $C_o$  are the capacitance for wood, and a vacuum dielectric respectively. The capacitance for wood was measured with the Audrey dielectric spectrometer. The

capacitance for the vacuum dielectric was calculated as follows (Brown, Panshin, and Forsaith, 1952):

$$C_o = \frac{A}{11.3d} \quad (5)$$

where A was the average area of the condenser plates in square centimeters and d was the distance between condenser plates in centimeters. A sample calculation follows.

Two sheets of 2.5mm thick veneer were held under pressure of 14.1 Kg/cm<sup>2</sup>. Assuming a 25%<sup>5</sup> reduction in veneer thickness while the veneer was under pressure, the distance between the electrodes was (2 X 2.5mm X .75) or 0.38 centimeters. Electrode size was 191mm X 292mm or 556 square centimeters. Substituting these into Equation 5 yields:

$$C_o = \frac{556}{11.3 \times 0.38} = 129 \text{ picofarads}$$

The capacitance for dry wood was 265 picofarads. Substituting the wood and vacuum capacitances in the Equation 4:

$$E = \frac{265}{129} = 2.0 \text{ for wood at M.C. equals zero}$$

TABLE 3. COMPARISON OF THE DIELECTRIC CONSTANTS OF WOOD AT THREE MOISTURE CONTENTS.

M.C. %	Dielectric Constant		
	Figure 6	Audrey Dielectrometer	Wheatstone Bridge Capacitor
0	2.0	2.0	1.9
6	2.5	2.6	2.7
10	3.3	3.2	3.2

<sup>5</sup>Wellons, 1978, (Unpublished results of plywood compression study). Oregon State University, Corvallis, Oregon.

Experiments on dry wood or wood conditioned to less than 15% M.C. resulted in reproducible data that agreed well with literature values. However, this was not the case when adhesives were monitored.

#### Monoprobe vs. Direct

The Audrey dielectric spectrometer is equipped with two electrode systems. Each electrode system is capable of interfacing with the wood composites used in this study. The first electrode system used one of the condenser plates as a ground while the other terminal (wire electrode) was inserted in the glue line. The second system used two electrodes that were opposite each other with the veneer composite between. These two electrode systems are called monoprobe and direct, respectively.

Since only one electrode system could be operable at a time, a choice between the two systems had to be made. Yalof's research (1975a, 1975b, 1975c, 1976, 1977) has largely been conducted with the Audrey electrode system in the direct mode. Other researchers (Fritzen, Wereta, and Arvay, 1977; Jemal, 1974; Kouchinsky, 1977; May, 1975; May, Whearty, and Fritzen, 1976) using the Audrey equipment also preferred this methodology. However, a few people (Fritzen, Wereta, and Arvay, 1977; Yalof, 1975d) have conducted their research using the monoprobe technique.

A series of experiments were conducted to test which electrode system was best suited to monitor the type of material being studied.

On these preliminary studies and all subsequent ones, graphs of dielectric properties were recorded as a function of time at various frequencies. Each graph reported will have two contour lines tracing

the dielectric response of one of the properties. The lines that are labeled 100 and 1000 are the dielectric measurements at the 100 and 1000 Hz frequencies, respectively. For capacitance values less than 50 or greater than 500 picofarads the response is nonlinear and non-calibrated.

A preliminary study demonstrated good compatibility between the two electrode systems when measuring capacitance and dissipation of conditioned wood. The capacitance readings were  $150 \pm 15$  picofarads with a frequency sweep of 100-1000 Hz. Similar results were obtained with the application of 5-10 grams of water to the conditioned veneer. Differences in the electrical measurements became noticeable with the monitoring of phenolic gluelines. The monoprobe technique recorded extremely high capacitance and remained off-scale for three to ten minutes (see Figures 9-1, 9-2). Also replicate experiments were very dissimilar. Polymer crosslinking began shortly after the panel was put in the press and the glueline should have been completely cured in  $2\frac{1}{2}$ -3 minutes. None of the changes in dielectric properties indicated this cure. Consequently, the monoprobe technique had limitations that made studying phenolic gluelines impossible.

The direct electrode technique provided some sensible observations. In Figure 9-3, the capacitance was initially off scale but was completely on scale after 90 seconds. The capacitance at 100 Hz showed the expected response during the period of interest. Figure 9-4 remained on scale throughout the entire time of measurement, but did not reproduce Figure 9-3 as well as had been expected. In spite of this lack of reproducibility, the direct electrode system showed

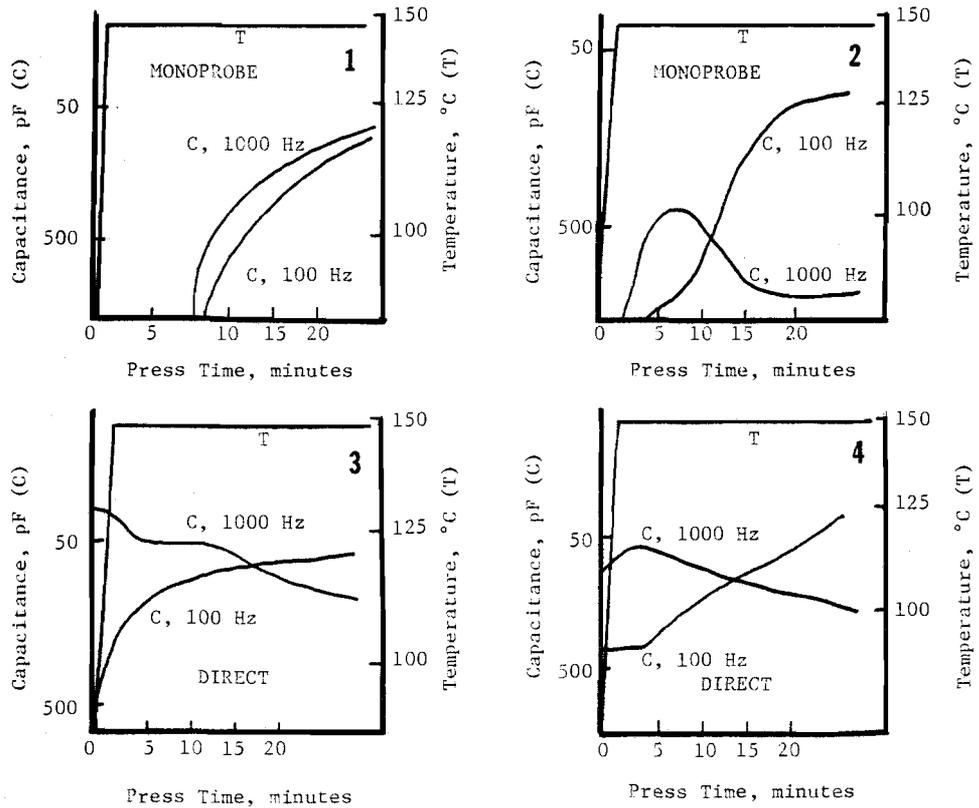


Figure 9. Comparison of the monoprobe and direct electrode systems as a function of time after adding phenolic adhesive to the veneer surface.

some promise, and was chosen as the electrode system used for this research.

### Comparing Different Electrical Properties

Cure of a phenolic plywood adhesive in a three ply panel using 2.5mm veneers at press temperatures of 150°C and a pressure of 12.3 Kg/cm<sup>2</sup> normally occurs in 2½-3 minutes (Anon., 1977). It is essential that the electrical properties be able to monitor the adhesive during this rapidly changing state. Because it is not possible to monitor all of the electrical properties simultaneously, a particular electrical property needed to be selected.

Previous researchers have chosen many different properties by which to monitor a composite's dielectric behavior. James (1975) used dielectric constant and loss tangent to study circular specimens of 5mm thick Douglas-fir. Miller and Cole (1957) measured the conductivity of a sample of resin glue. Yalof (1973, 1975a, 1975b, 1977) successfully tracked many adhesive systems using the electrical properties that are measured by the Audrey II.

All four of the Audrey's electrical properties were tested and evaluated using a phenolic adhesive. Typical response curves for the different properties are shown in Figure 10.

The graphs of voltage (Figure 10-1) and dissipation (Figure 10-3) during the cure time consisted of two parallel lines, one for the 1000 Hz frequency and the other for the 100 Hz frequency. No changes were observed in the electrical properties during the entire period. The graph of phase angle versus cure time (Figure 10-2) likewise showed no change until after approximately five minutes. At this

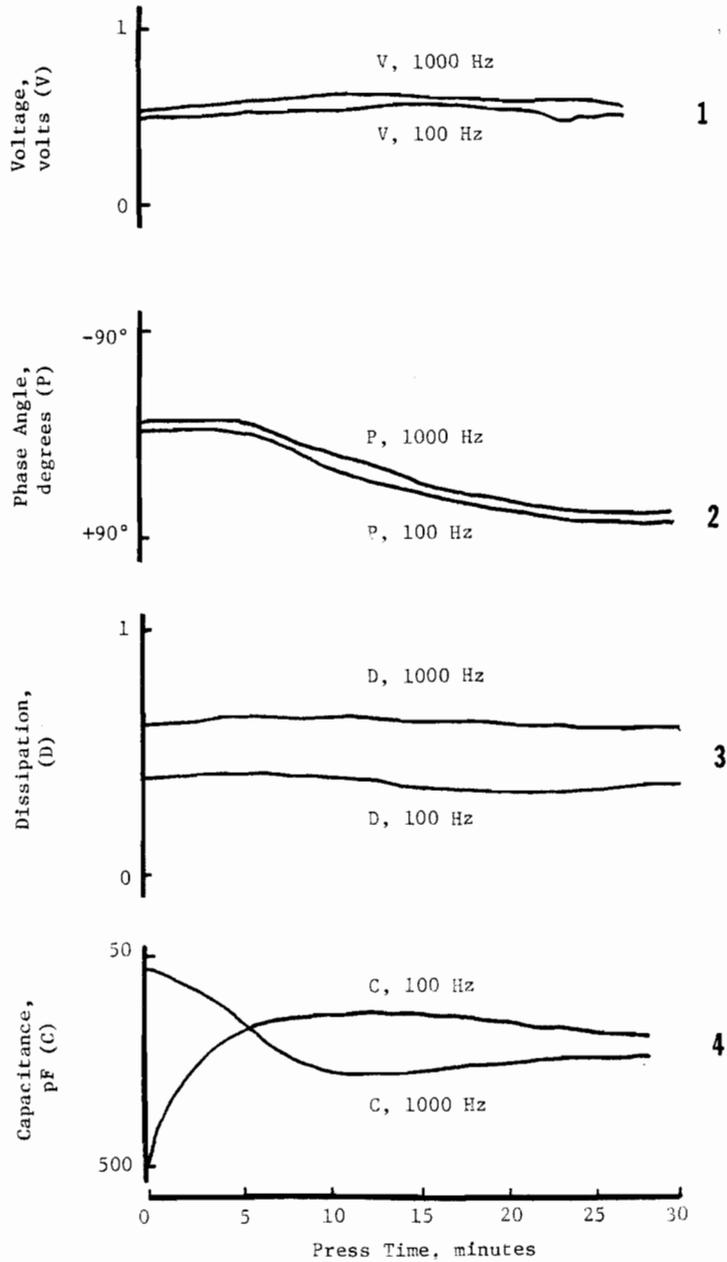


Figure 10. Comparison of different electrical properties during the cure of a phenolic adhesive.

point, the phase angle began to increase steadily for 20-30 minutes, but the values of 100 and 1000 Hz continued to parallel one another. The slight changes in the phase angle occurred after expected adhesive cure. The capacitance curve showed much change in the time frame important to adhesive cure, especially at 100 Hz. The curves of capacitance versus time (Figure 10-4) at the two frequencies moved towards one another, crossed, and then spread apart. Because changes in capacitance occurred within the time frame significant to adhesive cure, capacitance was chosen as the electrical property to be used to monitor adhesive cure.

#### Electrode Location

The placement of electrodes had not been adequately addressed in previous literature primarily because past research in the area of dielectric monitoring had used specially designed test cells (Miller and Cole, 1957; Jemal, 1974; Yalof and Brisbin, 1973; Yalof, 1975a; Yalof, 1975b; Yalof, 1975d; Yalof, 1977). These test cells were well isolated from the environment and had built-in electrodes.

The location of the electrodes had to be experimentally derived. Three different electrode locations were examined: 1) both electrodes outside of the panel, 2) one electrode outside the panel and one electrode in the glueline, 3) both electrodes in the same glueline (see Figure 11). Figure 12 shows typical graphs of capacitance versus cure time of a phenolic adhesive using the three electrode locations.

By placing both electrodes in the same glueline (Figure 11-3) it was our intent to monitor the changes of that glueline as the resin cured. Apparently, the electric field between the two

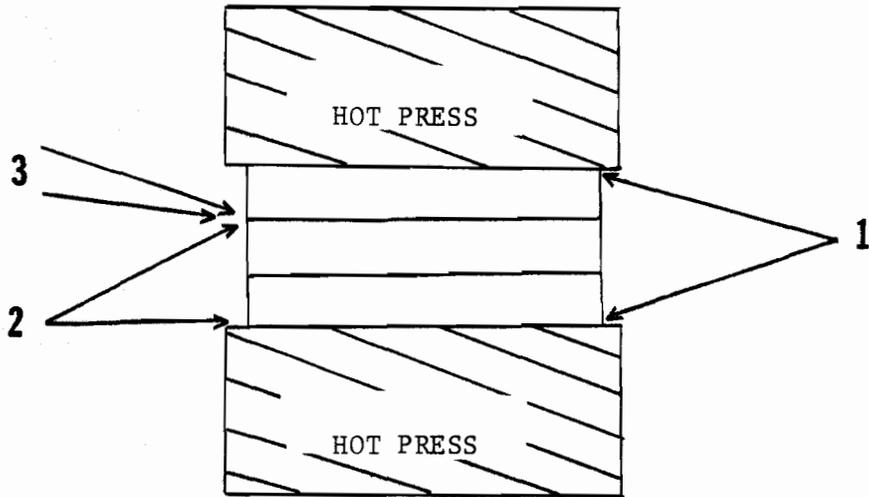


Figure 11. Electrode positions.

- 1 BOTH ELECTRODES OUTSIDE PANEL
- 2 ONE ELECTRODE OUTSIDE PANEL  
ONE ELECTRODE IN GLUELINE
- 3 BOTH ELECTRODES IN SAME GLUELINE

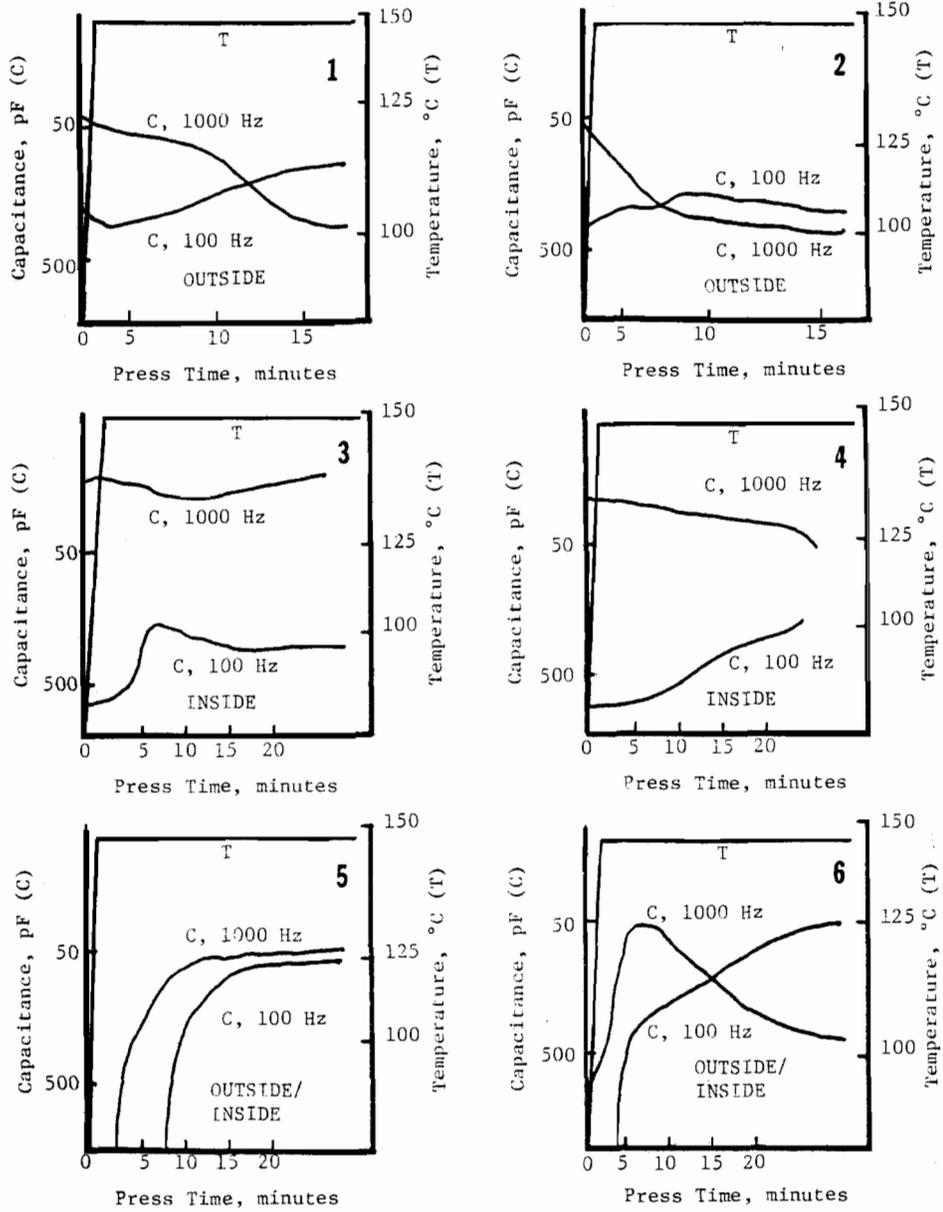


Figure 12. Determination of electrode location using a phenol-formaldehyde resin.

electrodes was influenced by an exchange of ions and caused an electrical short. Attempts to remedy this problem were made by coating the electrode with an electrically resistant material. However, coating the electrodes did not prevent electrical shorting. Aluminum foil electrodes were replaced by 24-gauge copper wire in an attempt to minimize the surface area of the electrode and to reduce the sensitivity of the electrical measurements. This electrode modification also failed to monitor the adhesive system. Figures 12-3 and 12-4 display the results of this electrode placement. The fact that all dielectric properties were off scale verified the electrical short circuit.

One electrode inside the glueline (Figure 11-2) created problems similar to those incurred when using the monoprobe electrode system-- capacitance readings that were off scale but changed rapidly with time when on scale. The curves in Figures 12-5 and 12-6 began off scale (high capacitance) but after a few minutes they came into the range of the Audrey dielectrometer and within minutes went off scale due to low capacitance. The results were very non-reproducible.

The third alternative for the electrode location consisted of having both electrodes outside the panel, Figure 11-1. This method measured an average change in the electrical properties for the entire composite, that is, changes in the wood as well as changes within the adhesive. Locating the electrode outside proved to be manageable for monitoring adhesive cure in wood composite samples. By adjusting electrode size the capacitance could be kept within the range of the Audrey. The curves in Figures 12-1 and 12-2 are on scale to start

with and remain on scale throughout the observation period. This electrode placement also produced graphs that showed dielectric response within the time frame significant to adhesive cure.

#### Determination of Electrode Size

Zeroing in on the correct electrode size was essential for keeping the capacitance within the calibrated range of the Audrey II. "Capacitance is proportional to the electrode area and inversely proportional to the distance between electrodes and sensitive to the quantity of dielectric material between the electrodes."<sup>6</sup> Too small or too large an electrode resulted in capacitance measurements off scale (less than 50 or greater than 500 picofarads). For a given experiment, the dielectric material (wood, glue) and the distance between electrodes remained constant so only the electrode area (size) needed to be determined experimentally.

To illustrate the effect different electrode areas had on the capacitance, five grams of water was applied to each side of the core veneer of a three-ply panel and capacitance was measured as a function of time. Figure 13 shows the graphs using three different electrode sizes. The 25mm X 70mm electrode (Figure 13-1) did not have enough capacitance to be on scale; the electrode area was too small. Increasing the size of the electrode to 102mm X 102mm (Figure 13-2) resulted in a measurable capacitance but again the effect was negligible. The 191mm X 292mm electrode (Figure 13-3) resulted in capacitance within the range of the Audrey II. Further enlargement

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<sup>6</sup>Allen, J. D., 1975, p. 109.

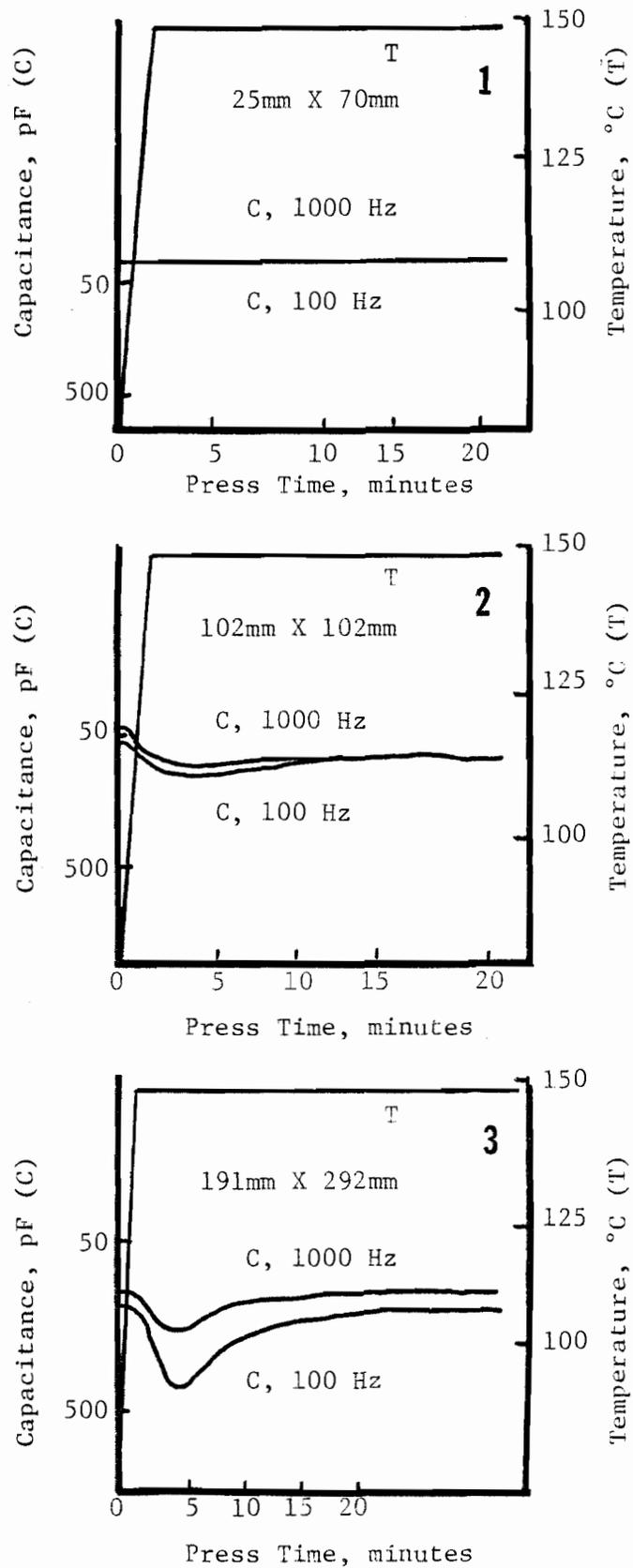


Figure 13. The effect of electrode size on capacitance of wetted veneers.

of the electrode area resulted in capacitance that exceeded the range of the instrument.

A similar approach was used for determining electrode size for the different resin systems. A summary of the electrode sizes for each adhesive system can be found in Table 4.

TABLE 4. ELECTRODE SIZE FOR EACH ADHESIVE SYSTEM.

	Electrode Size (millimeters)	Electrode Area (sq. mm)
Phenolic resin	191 X 292	55,600
Urea resin	178 X 178	31,600
Phenolic adhesive	191 X 292	55,600
Resorcinol adhesive	191 X 292	55,600
Phenolic dry film	191 X 292	55,600

#### Replication of Adhesive Systems

Five different adhesive systems were studied, all of which are common to the forest products industry.

Phenolic adhesives are primarily important to the softwood plywood industry. Their benefit is that they form water proof gluelines. Urea resins are used in the plywood industry and their glue bonds are considered only to be water resistant and application is limited to interior grade plywood. Because the price difference between urea (natural gas derivative) and phenol (petroleum derivative) has narrowed in recent years, phenolic adhesives have dominated the softwood plywood industry. If this trend reverses, urea resins could recapture the interior plywood market. Resorcinol adhesives

have special applications to the laminated beam and furniture industries. Phenolic dry film adhesive is very expensive and has a limited use in the overlay industry.

Each resin or adhesive was tested several times holding all other variables constant. These replications seldom resembled one another. This result was surprising because consistent results were obtained with both wet and dry veneers. Figure 14 shows three typical replications for a phenol-formaldehyde resin. The range in capacitance varied greatly. Figure 14-1 shows curves that initially are off scale (high capacitance) for both frequencies and gradually return to approximately 250 picofarads. In Figure 14-2 the curve for 1000 Hz starts out of range (low capacitance) while the curve for the 100 Hz frequency records a capacitance of about 450 picofarads. The capacitance readings at 1000 and 100 Hz become more similar with time until they cross at about eight minutes. In Figure 14-3 the curves remain on scale during the entire observation period and the response curves for 100 Hz and 1000 Hz frequencies cross at about seven minutes.

By adding extenders (wheat flour and shell flour) to the resin (to make an adhesive) we encountered similar trends in variation. Figure 15 displays a wide range of capacitance values. The capacitance curves at 100 and 1000 Hz cross at times ranging from seven minutes to 15 minutes.

Resorcinol is chemically similar to phenol. Figure 16 illustrates the chemical structures of phenol, resorcinol, and urea. Resorcinol unlike phenol is able to cure at room temperature, but heat catalyzes the reaction. Figure 17 shows three replications of resorcinol-formaldehyde adhesive holding all variables constant. The capacitance

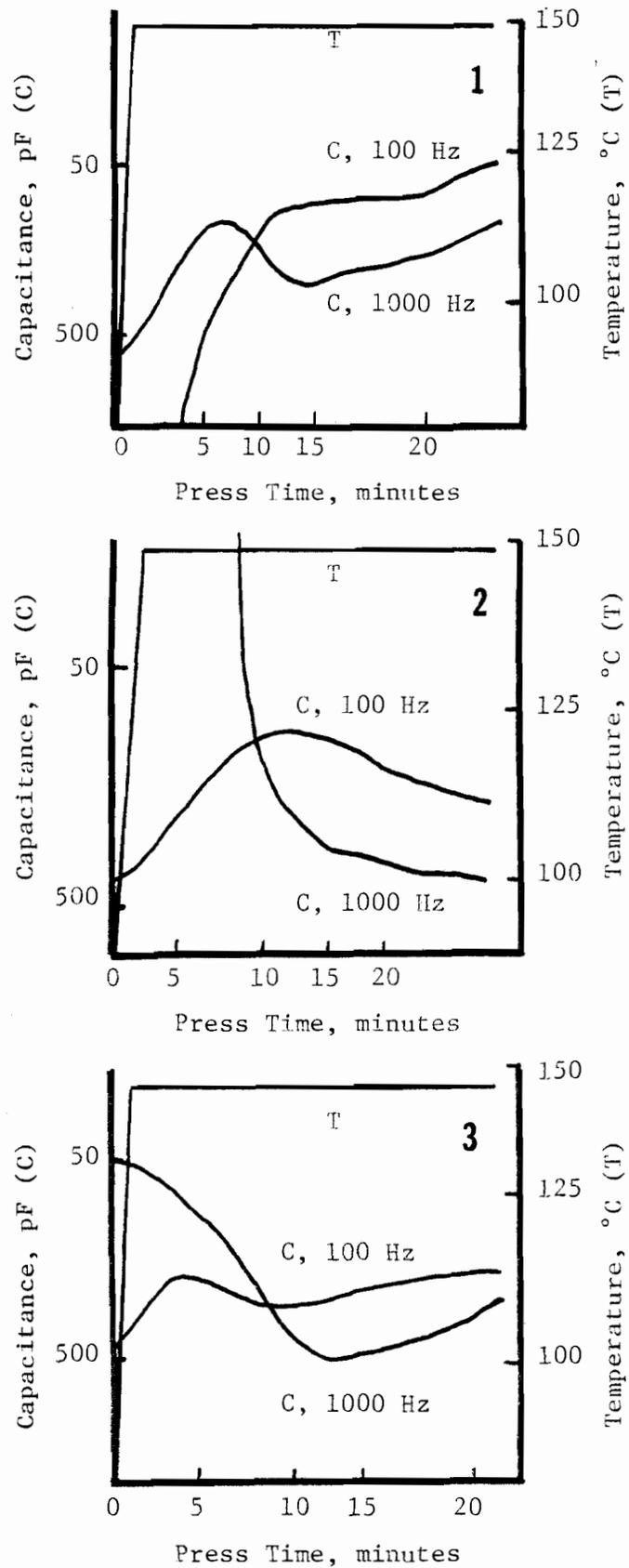


Figure 14. Replications of the change in capacitance of a phenol-formaldehyde resin as a function of cure time.

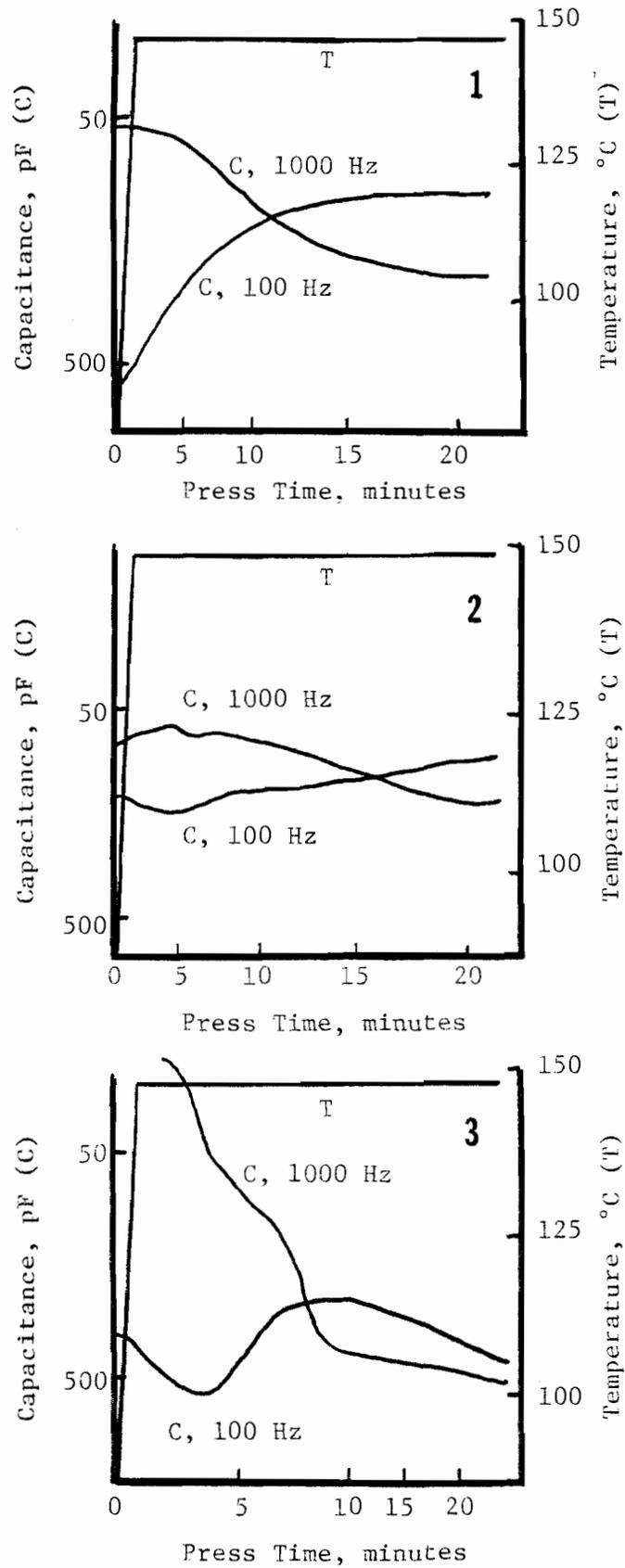
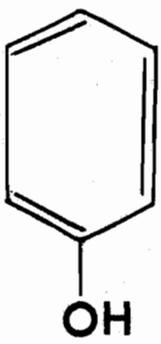
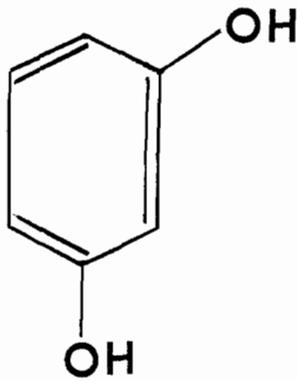


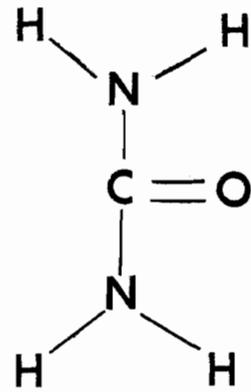
Figure 15. Replications of the change in capacitance of a phenol-formaldehyde adhesive as a function of cure time.



1 Phenol



2 Resorcinol



3 Urea

Figure 16. Chemical structure of plywood resin building blocks.

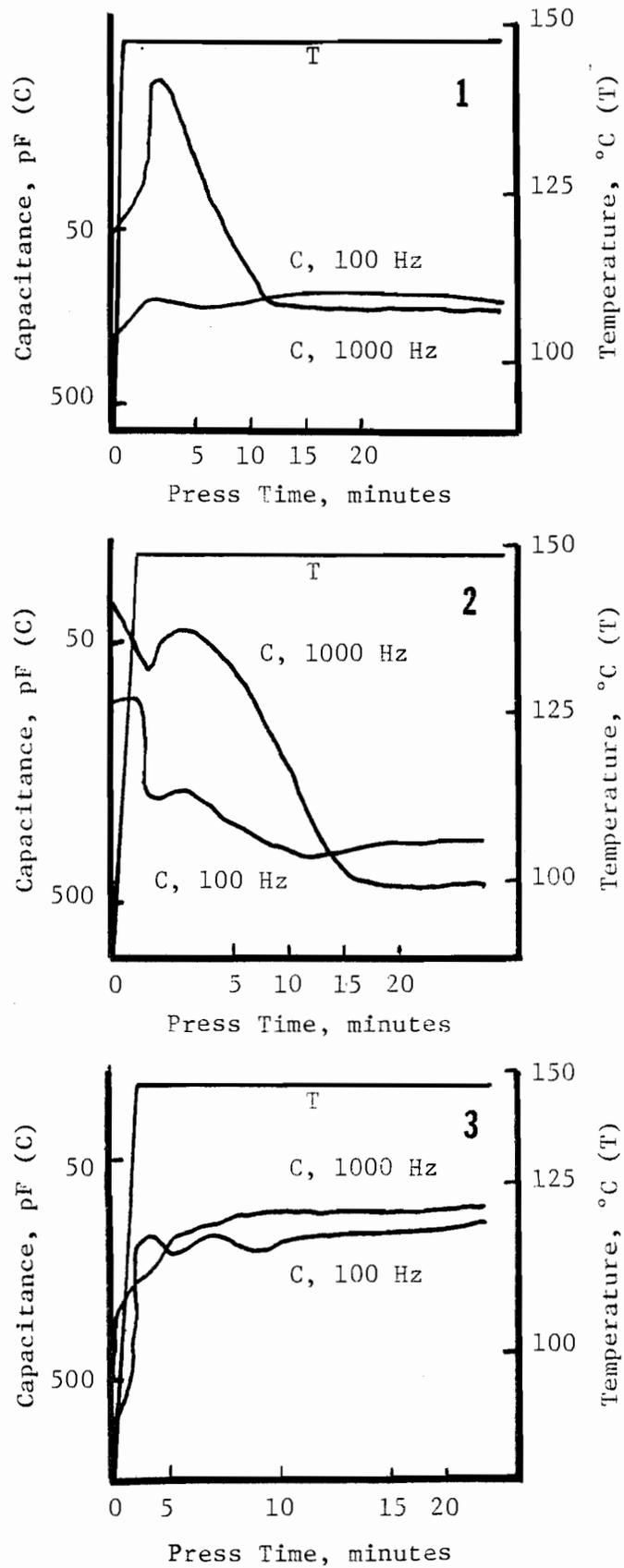


Figure 17. Replications of the change in capacitance of a resorcinol-formaldehyde resin as a function of cure time.

values ranged from off scale due to low capacitance to off scale due to high capacitance. The shapes of the graphs were also variable. The time at which the 100 and 1000 Hz curves cross ranges from two to 15 minutes. No predictable curing trends were observed for the resorcinol adhesive.

Urea resin is not chemically similar to either phenol or resorcinol. Complete cure of urea-formaldehyde resin used in this study can be expected in two to three minutes. Figure 18 shows three replications of urea resin cure. During the time of expected resin cure the capacitance changed little for a given sample, but capacitance changed considerably from sample to sample.

None of the adhesive responses could be reproduced. Some capacitance responses hovered between 50 and 500 pF, other responses exceeded the lower limit of the capacitance scale while others exceeded the upper limit of this scale. The graphs were different in shape and time at which the 100 and 1000 Hz curves crossed. Originally, it was thought that the time of the crossing might have some significance to adhesive cure. This idea was refuted by observing that some pairs of graphs (Figure 20-1) did not cross, while other pairs (Figure 17-3) crossed twice. In addition most graphs crossed long after the expected cure time.

The Audrey II dielectrometer was unable to monitor the dielectrical behavior of resins and adhesives that are key to the forest products industry. To better understand the variability encountered, water was examined as a cause for variable capacitance. Previous literature emphasized that the presence of water is a major concern when making dielectric measurements (Headvig, 1977). This research

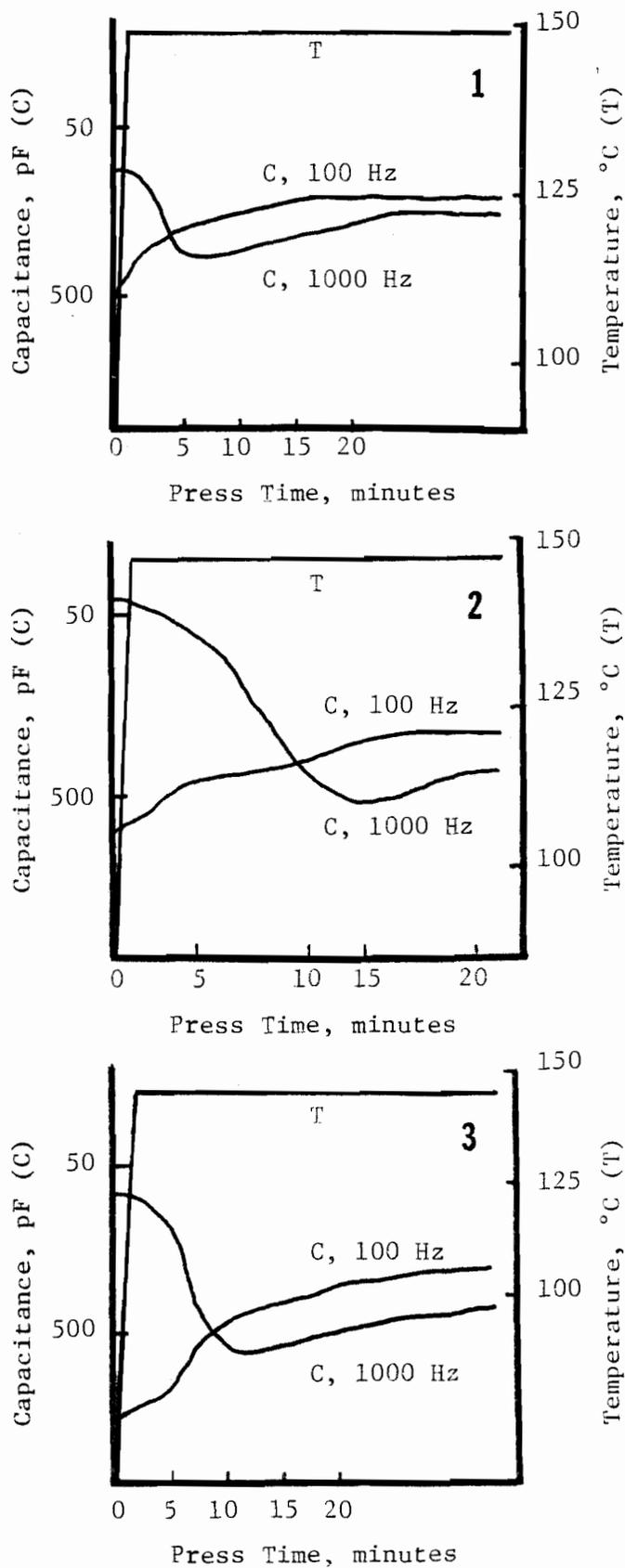


Figure 18. Replications of the change in capacitance of a urea-formaldehyde resin as a function of cure time.

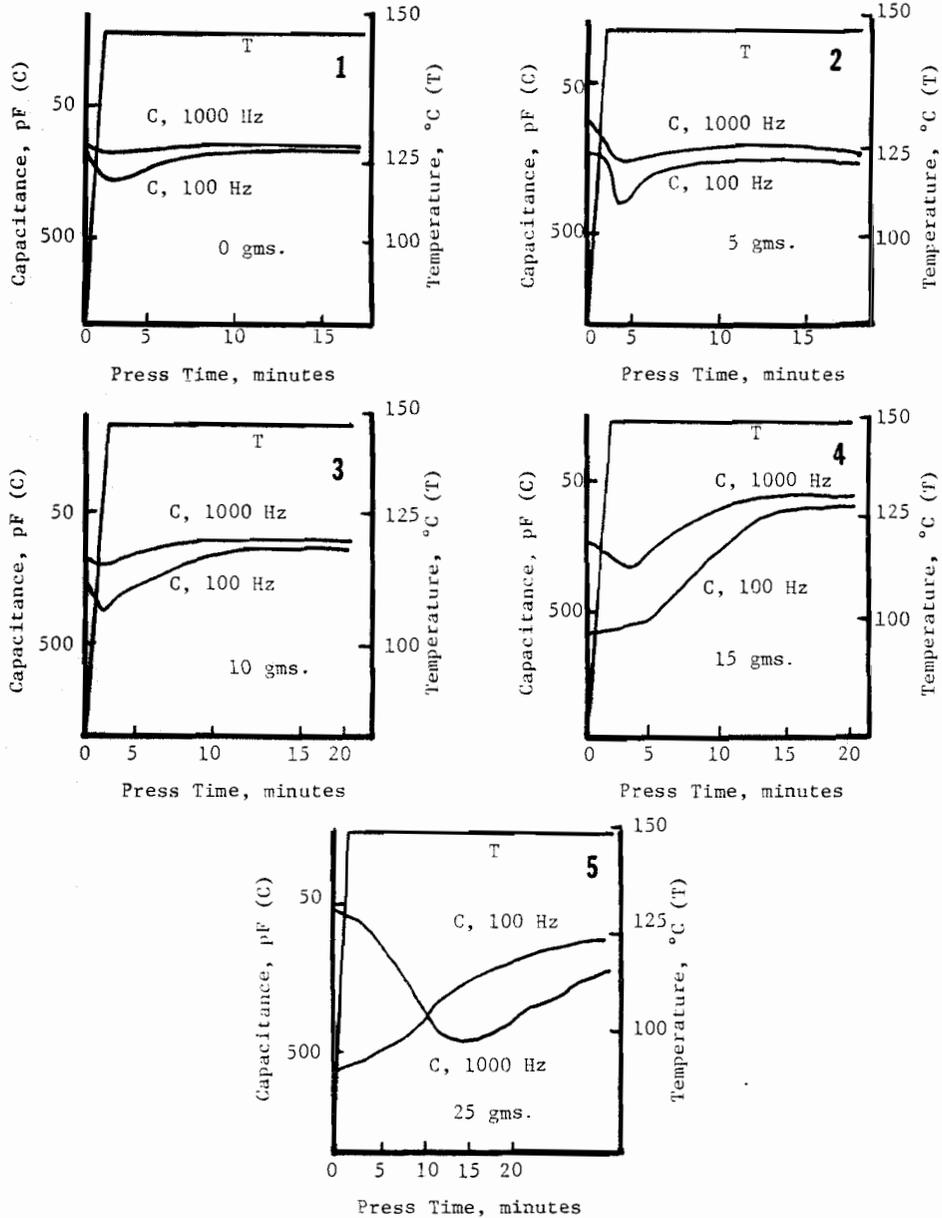


Figure 19. Effect of applying water to veneer surface on capacitance of wood conditioned to 6% M.C.

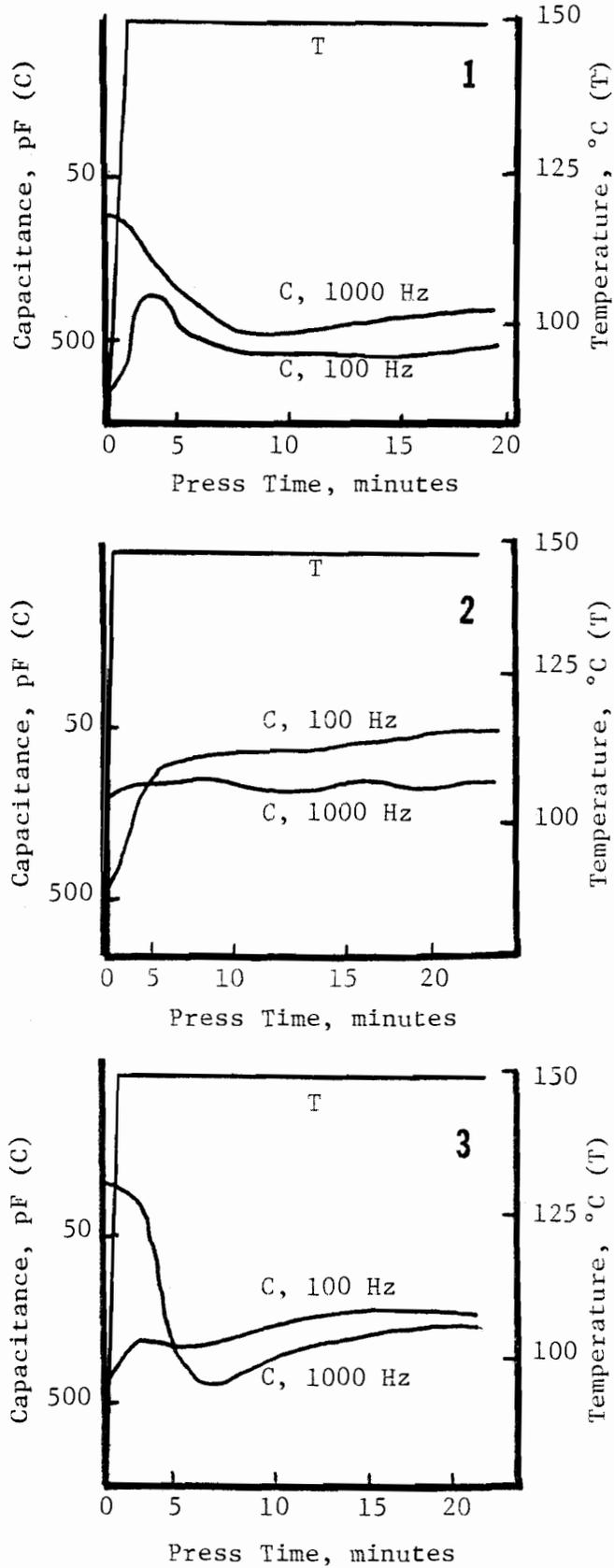


Figure 20. Replications of the change in capacitance of a dry phenolic adhesive film as a function of cure time.

has shown that under carefully controlled conditions the presence of water creates no serious problems in monitoring capacitance (Figure 19). The shape of the capacitance curve does depend on the amount of water present but the effect is reproducible and changes systematically as the water content changes.

To further eliminate water as a source of variation a number of replications were made using a nonaqueous phenolic adhesive film (Figure 20). Nonaqueous adhesives have shorter cure times than aqueous adhesives; full cure of the film adhesive would be expected to occur in approximately two minutes. Again much variation was observed in the capacitance values. An obvious trend is that the graphs show much less change in capacitance than those with aqueous phenolic adhesives. Figure 20-1 showed rapidly changing capacitance for the first three minutes of cure followed by a constant capacitance. Figures 20-2 and 20-3 report graphs of capacitance response in opposing directions. The problem of repeatability still existed in the absence of water in the glueline; therefore, the source of variation must be due to the ingredients of the adhesive.

#### Phenol-Formaldehyde Resin Components

Because the dielectric response of the resins could not be successfully interpreted, it became important to isolate the individual resin components to determine which component(s) were responsible for the lack of repeatability. Although the problem occurred with all of the adhesive systems, only phenol-formaldehyde was chosen for detailed analysis.

A typical formulation used for a phenol-formaldehyde resin is as follows: 56% water, 7% catalyst (NaOH), 15% formaldehyde, and 22% phenol. When examining the influence on capacitance of individual resin components, the resin components were applied to two ply panels in proportions that would be equivalent to the amount of that component included in a normal resin application. The amounts of each component applied to a veneer are summarized in Table 5. Figures 21, 22, and 23 graphically describe the effect of those resin components on capacitance.

TABLE 5. PERCENT OF PHENOL-FORMALDEHYDE RESIN COMPONENTS APPLIED TO VENEER.

Components	Percent of Normal Resin Components Applied	Actual Weight Applied Per Glueline (gms/meter <sup>2</sup> )
Water (56%)	56	109
Water (56%), catalyst (7%)	63	122
Water (56%), formalin (15%)	71	138
Water (56%), phenol (22%)	78	152
Water (56%), catalyst (7%), formalin (15%)	78	152
Water (56%), formalin (15%), phenol (22%)	85	165
Water (56%), catalyst (7%), formalin (15%), and phenol (22%)	100	194

Wood. Wood conditioned (Figure 21-1) to 6% M.C. served as a control. Graphs of capacitance versus heating time were reproducible and showed little change in capacitance. The graphs of capacitance

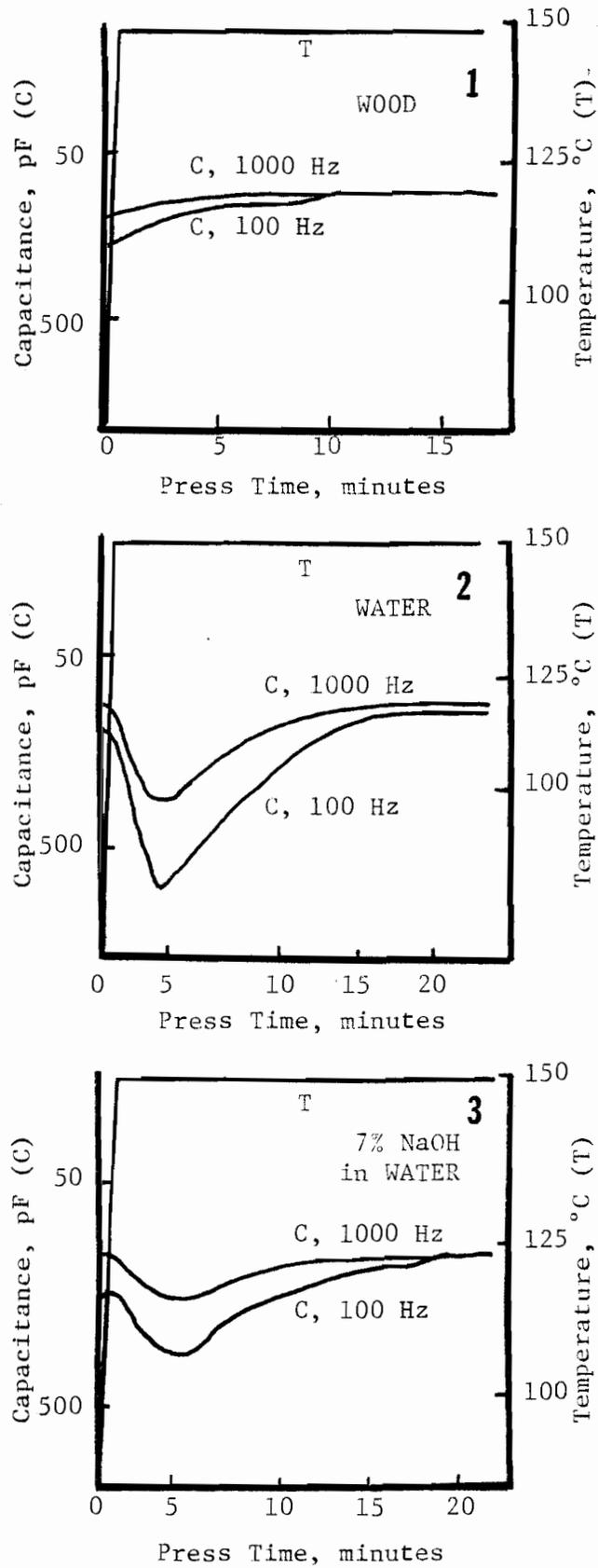


Figure 21. The effect of water and caustic on capacitance.

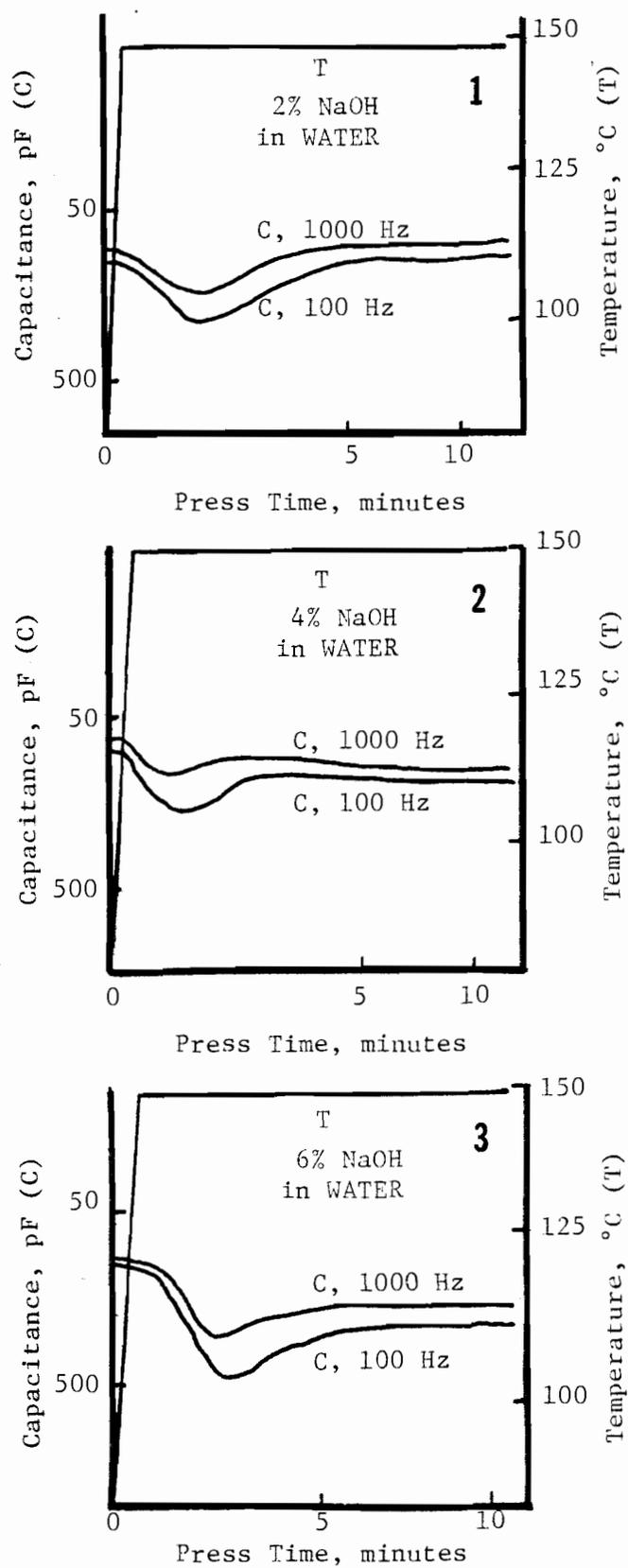


Figure 22. The effect of caustic concentration on capacitance.

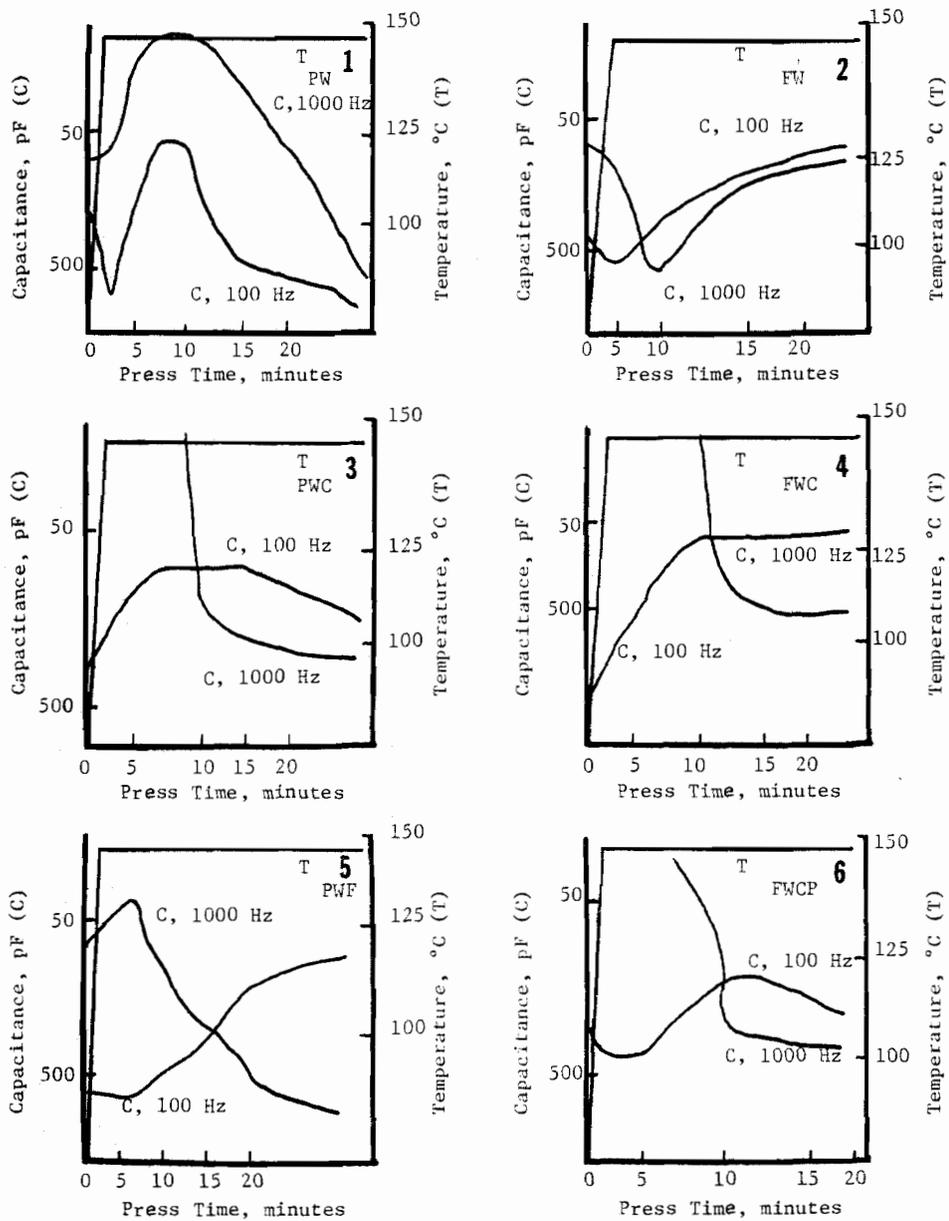


Figure 23. The effect of phenol and formaldehyde on capacitance.  
 LEGEND: W=water, C=caustic, P=phenol, F=formalin

at 100 and 1000 Hz did not cross and the capacitance varied only 25-30 picofarads in going from 100 and 1000 Hz.

Water. With an increase in temperature and moisture, wood fiber softens and molecular mobility increases. Panels were measured for capacitance after applying the same amount of water found in a normal phenol-formaldehyde resin glueline and heating the panel. These graphs were consistent and repeatable. Figure 21-2 shows a typical graph. The initial capacitance is typical of that of conditioned wood. As the heat and moisture penetrated the wood, the capacitance increased and the difference in capacitance between 100 and 1000 Hz also increased. As the water reached its boiling point (100°C), it moved through the wood as water vapor (Anon., 1970) until it reached the edges of the veneer and evaporated into the atmosphere. With the loss of moisture the capacitance gradually decreased and stabilized to that of dry wood.

Catalyst. Sodium hydroxide (NaOH) is the catalyst used in the adhesive being studied. Variation in catalyst concentration was considered to be a potential source of variability in capacitance. NaOH is an electrolyte and may cause electrical shorting.

Because adhesive formulations vary slightly from one batch to another this experiment was intended to define any differences that might be associated with a change in the NaOH concentrations. The percent of sodium hydroxide in water was studied over the range from 2-7%. The NaOH solution was applied to one surface of a two ply composite. Figures 21-3, 22-1, 22-2, and 22-3 show graphically the results of this experiment. The capacitance increased slightly with an increase in NaOH. The shapes of the graphs were very similar

to each other and very similar to ones with equivalent amounts of water. These graphs could be reproduced, so it was concluded that normal variations in the NaOH concentrations from one batch of adhesive to another did not adversely affect the reproducibility of the capacitance measurements.

Phenol or Formalin. Uncontrollable variation in capacitance resulted with the application of phenol or formalin to the veneers. (See Figure 23.) Phenol and formalin are considered to be excellent swelling agents for wood (Stamm, 1964). Phenol swells wood about 23% more than water while formalin swells it about 9% more than water (see Figure 24). As wood swells, the wood's microstructure is opened and plasticized. This would increase the capacitance. Other factors that would influence capacitance variation would include depth of lathe checks, wood damage, porosity, density, and natural anatomical variation that influences the rate of penetration of these chemicals into wood. Although these may be some of the reasons for variation in capacitance, the exact reason is not known. Even considering the swelling effects and the natural variation of wood, the variability in capacitance of the wood-adhesive composite is not readily understandable.

#### Varying Assembly Time

Different assembly times (time between adhesive application and hotpressing) were examined using a phenol formaldehyde resin in an attempt to identify any trends associated with the absorption of water and other adhesive ingredients from the phenolic glueline. As the assembly time became excessive the capacitance of the wood

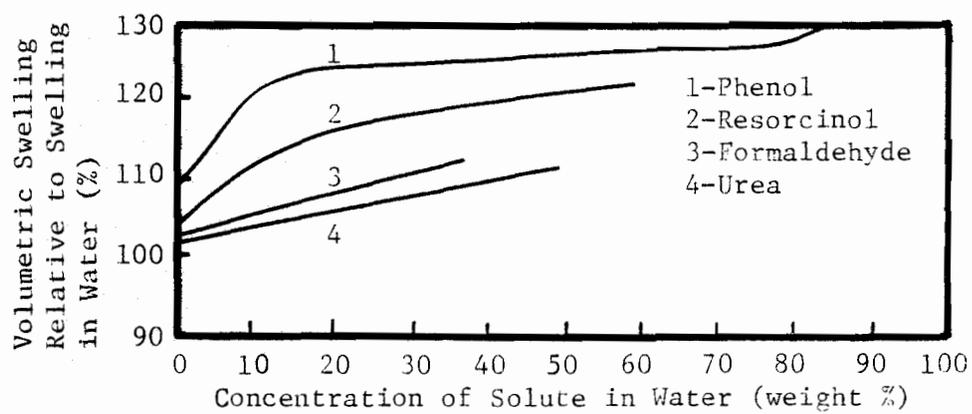


Figure 24. Volumetric swelling of wood in aqueous solutions of adhesive components (Stamm, 1964).

composite should have simulated 1) wood conditioned to a higher moisture content, and 2) adhesives with a higher percent solids.

An interesting phenomenon was noted as the assembly time increased (see Figure 25). After 30 minutes of assembly time the graphs of capacitance versus cure time no longer crossed. Perhaps this suggests that water in the glueline in conjunction with the other adhesive components was responsible in some way for the wide variation in capacitance at the different frequencies. It should also be noted that the capacitance was much less erratic with longer assembly times. Although these graphs were not reproducible, the general trends were. No specific conclusions could be reached about assembly time and capacitance except that longer assembly times resulted in a more consistent capacitance.

Due to time restrictions further studies were prohibited. Future research may wish to consider this variable more closely.

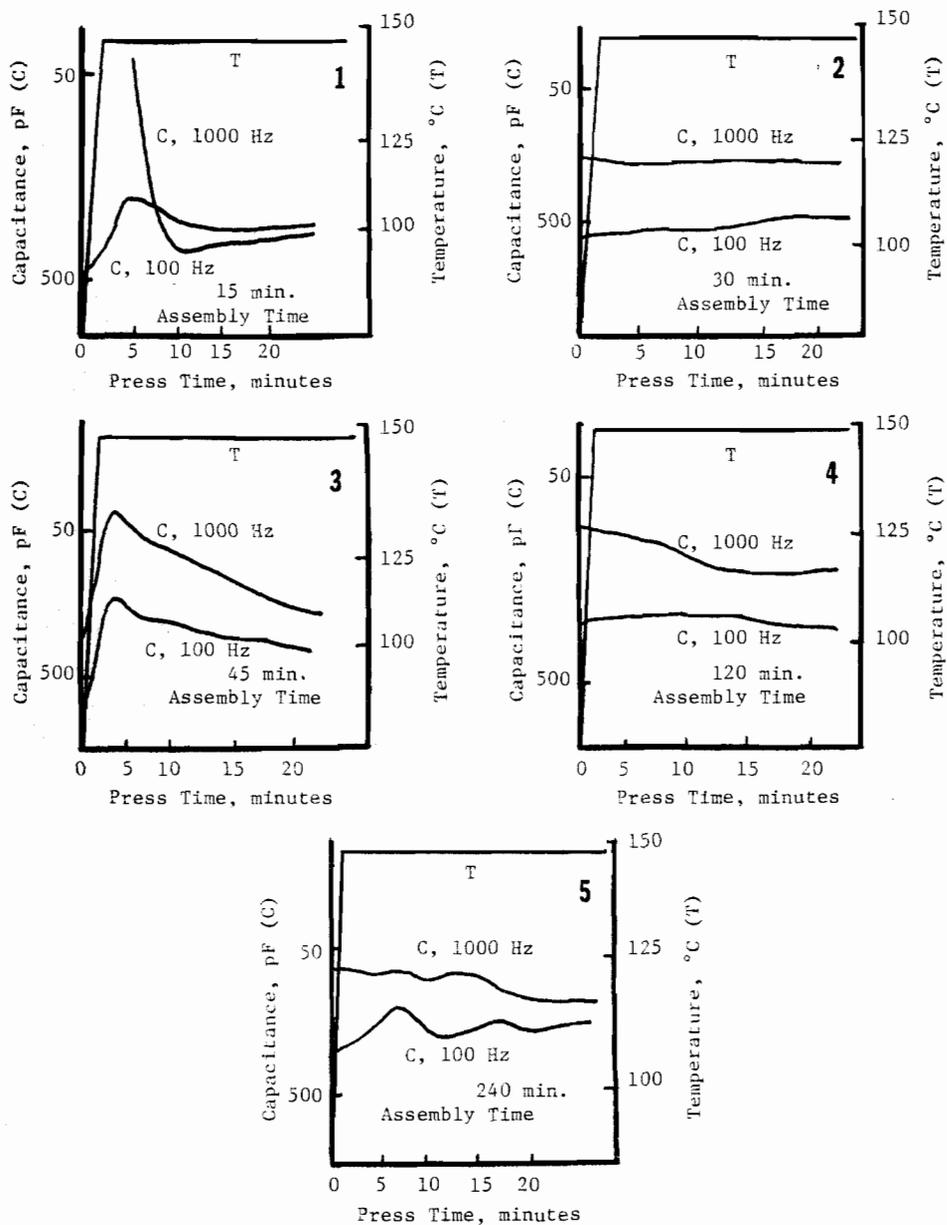


Figure 25. Effects of varying assembly time on capacitance of a phenol-formaldehyde resin.

## V. CONCLUSIONS AND RECOMMENDATIONS

Monitoring the dielectric properties of a wood composite via dielectric analysis has resulted in several conclusions beyond the original scope of this research. Because the capacitance of wood-adhesive composites varied in no reproducible pattern, statistical analysis was not necessary to reach the following conclusions.

### Conclusions

1. The Audrey II dielectric spectrometer did not measure the cure of highly caustic phenolic adhesives, urea-formaldehyde resins, resorcinol-formaldehyde adhesives or dry phenolic adhesive films in Douglas-fir gluelines during the cure process.
2. Of the dielectric properties studied, capacitance was the most responsive to changes that occur in Douglas-fir plywood panels under simulated mill conditions and within the time frame of adhesive cure.
3. The monoprobe electrode system was not a feasible monitoring system for the adhesives studied.
4. Dielectric measurements were very sensitive to the presence of water (confirming studies described by Brown, Panshin and Forsaith, 1952), and the increase in capacitance with an increase in moisture content was reproducible.
5. The addition of phenol or formalin to the wood composite resulted in uncontrollable variation in the capacitance.

6. Increasing amounts of sodium hydroxide in water (2-6%) caused reproducible increases in capacitance.
7. Longer assembly times appeared to result in more reproducible capacitance values than shorter assembly times.

#### Recommendations for Future Research

The need continues for a system that will measure nondestructively adhesive cure of a wood composite panel in-process. More research is needed along the lines of the present study. Because adhesive manufacturers are bringing new and better adhesives "on-line", further research should include examining the ability of these adhesives to respond reproducibly to an electrical impulse. In future studies, wood adhesives should be isolated and tested in a specially designed test cell apart from wood. After successfully monitoring adhesive cure, studies on wood-adhesive composites should follow. Other electrical properties (e.g. resistance, conductivity, etc.) may provide greater insights in electrical monitoring of adhesive cure.

The aerospace industry, where dielectric monitoring was developed, will undoubtedly be a leader in new advances in this technology. Periodic reviews of their findings may reveal new areas of development that could reinitiate research on wood adhesive systems.

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