

RESIN CHARACTERIZATION

(Cooperative Research Between University of Wisconsin--Madison and the USDA Forest Service, Forest Products Laboratory)

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ABSTRACT

Currently, thermosetting adhesives are characterized by physical and chemical features such as viscosity, solids content, pH, and molecular distribution, and their reaction in simple gel tests. Synthesis of a new resin for a particular application is usually accompanied by a series of empirical laboratory and plant

trials. The purpose of the research outlined in this paper was to develop techniques that could be used to characterize thermosetting resins by their time-dependent reaction to the environmental conditions--temperature and moisture--that influence both cure and bonding.

Resin-impregnated glass cloth samples were exposed to controlled temperatures and

humidities for increasing periods. Development of mechanical stiffness in the resin was monitored using a dynamic mechanical analyzer. Chemical advancement was determined using a differential scanning calorimeter. Chemical-mechanical relations expressed as percentage of cure were useful in comparing the reactivity of different resins. Lap-shear tests of flake pairs bonded under the same conditions used to determine resin cure will provide data on the rate of strength development.

By recovering and testing resin cure and flake-bonding specimens from hot-pressed boards, the selective response under controlled conditions can be compared with that under actual operating conditions. Changes in molecular descriptions of newly synthesized resins, obtained using nuclear magnetic resonance and infrared analysis, can then be used to predict the performance of the resins in specific applications.

INTRODUCTION

Optimum conditions for curing thermosetting resins used in particleboard and other composite wood products differ between manufacturing sites and quite often change in any one operation due to planned or unplanned alterations of the process variables. Composite manufacturers rely almost entirely on adhesive suppliers to provide them with the most appropriate resin to bind their product. Necessary changes in resin formulation or synthesis modification are based on empirical experience and often require extensive laboratory and plant trials. Although the performance of a resin depends on its reaction with numerous variable environmental conditions, adhesive characterization is presently restricted to physical and chemical features such as viscosity, solids content, pH, and molecular distribution and reaction in simple gel tests. The purpose of the research outlined

in this paper was to develop a means to characterize a resin based on its response to the common process variables of time, temperature, and moisture.

A number of methods have been used to quantify the extent of reaction in an adhesive as it ages or cures. Gel permeation chromatography (GPC) is useful in the early stages of cure while the resin is still in a liquid stage (Wellons and Gollob 1980). Fourier transform infrared (FTIR) spectroscopy can characterize the reactions into the solid stages, but represents major difficulties in making quantitative determinations (Myers et al., in press). Nuclear magnetic resonance (NMR) spectroscopy has been used to characterize chemical structural changes in phenolics as curing progresses (Young 1985). Differential scanning calorimetry (DSC) measures the cure of resins by sensing the exothermic heat output of a small sample during controlled heating. However, FTIR, NMR, and DSC do not differentiate chain extension from crosslinking reactions, and the data are not directly informative about mechanical property buildup.

Because the objective of the research was to characterize resin response to changes in environmental conditions during the pressing process, a method was required for measuring degree-of-cure in samples that had been exposed previously to different combinations of those variables. Prior research showed that the dynamic mechanical analyzer (DMA) had good potential for meeting these needs (Gillham 1985, Johns and Plagemann 1985, Steiner and Warren 1981, Young et al 1985). The DMA is sensitive to changes in the mechanical stiffness of a curing resin sample and, therefore, gives a direct indication of the molecular crosslinking that occurs in the late stages of resin cure.

The research was motivated in part by problems associated with steam injection

pressing of phenolic resin-bonded composites. During this newly developed process, saturated steam was injected into the mat to enhance transfer of heat to the core (Geimer 1983). Use of phenolics with this pressing system has produced sporadic results. Good bonding was obtained with Douglas fir ring flakes (Geimer 1982). However, bonding was poor when oak disk flakes were used in conjunction with phenolic resins to make structural flakeboard (Geimer and Price 1986).

Poor bond formation has been attributed to various factors, including precure, moisture retardation, starved glue lines caused by excess penetration, and type of wood particle. The steam injection system was appropriate to this study in that environmental conditions are severe and can be finely controlled in the pressing operation. However, techniques developed to characterize the curing and bonding of phenolic resins used in steam injection pressing are also applicable to conventional pressing operations and can be expanded to include other resin types.

SCOPE

The research included eight complementary studies:

1. Development of techniques and procedures to determine the change in mechanical stiffness of a resin, as measured by the DMA, after time-extended exposures to various dry temperatures.
2. Measurement of the chemical advancement of a resin caused by cure in a dry environment
3. Development of correlations between the mechanical stiffness data and the

chemical advancement data to reveal the chemical-mechanical “pathway”, of curing.

4. Extension of the data obtained in studies 1, 2, and 3 to moist exposure conditions.
5. Development of techniques to determine the development of resin-flake bond strength under various conditions of temperature and moisture, and the relationship between these data and resin cure.
6. Measurement of environmental changes occurring in a composite board during the steam injection pressing operation and direct extension of these data to determine the effects of the press environment on the curing and bonding of test samples retrieved from within the board.
7. Exploration of methods to quantify the penetration of resin into a wood substrate and determination of the change in penetration with changing environmental conditions and the consequence for bond strength development.
8. Use of other spectroscopic techniques (FTIR, NMR) to provide a chemical rationale for the observed curing and bonding behavior and development of a data bank useful to resin manufacturers in the synthesis of resins for specific applications or in defining pressing limitations for specific resins.

The following discussion summarizes the results of work completed in the attempt to characterize a resin by its reaction to environmental conditions. Work has been funded, in part, by a grant from the USDA Competitive

Grants fund. This report is intended to promote industrial interest and support for the project.

MECHANICAL PROPERTIES OF A PARTIALLY CURED RESIN-- DMA CHARACTERIZATION

A Du Pont 983 DMA¹ (Figure 1) was used for all mechanical measurements of resin samples. In tests using this DMA, the sample, a special 0.2 by 12.5 by 35 mm nonwoven glass filter cloth impregnated with resin, is clamped

horizontally between the ends of two parallel arms (Figure 2). One arm and the sample are driven to oscillation at a prescribed amplitude by an electromagnetic drive. Energy dissipation by the sample causes the actual sample strain to be out of phase with the driver signal. The instrument detects this time shift as a phase angle and calculations are made to determine the storage modulus E' , a measure of the material's stiffness, and the loss modulus E'' , a measure of the material's viscosity. The ratio of these two properties, E''/E' , is designated tan delta, which is useful in determining the extent of resin mechanical cure (discussed later).



Figure 1.- *Du Pont 983 dynamic mechanical analyzer*

¹The use of trade or firm names in this paper is for reader information and does not imply endorsement by the U. S. Department of Agriculture of any product or service.

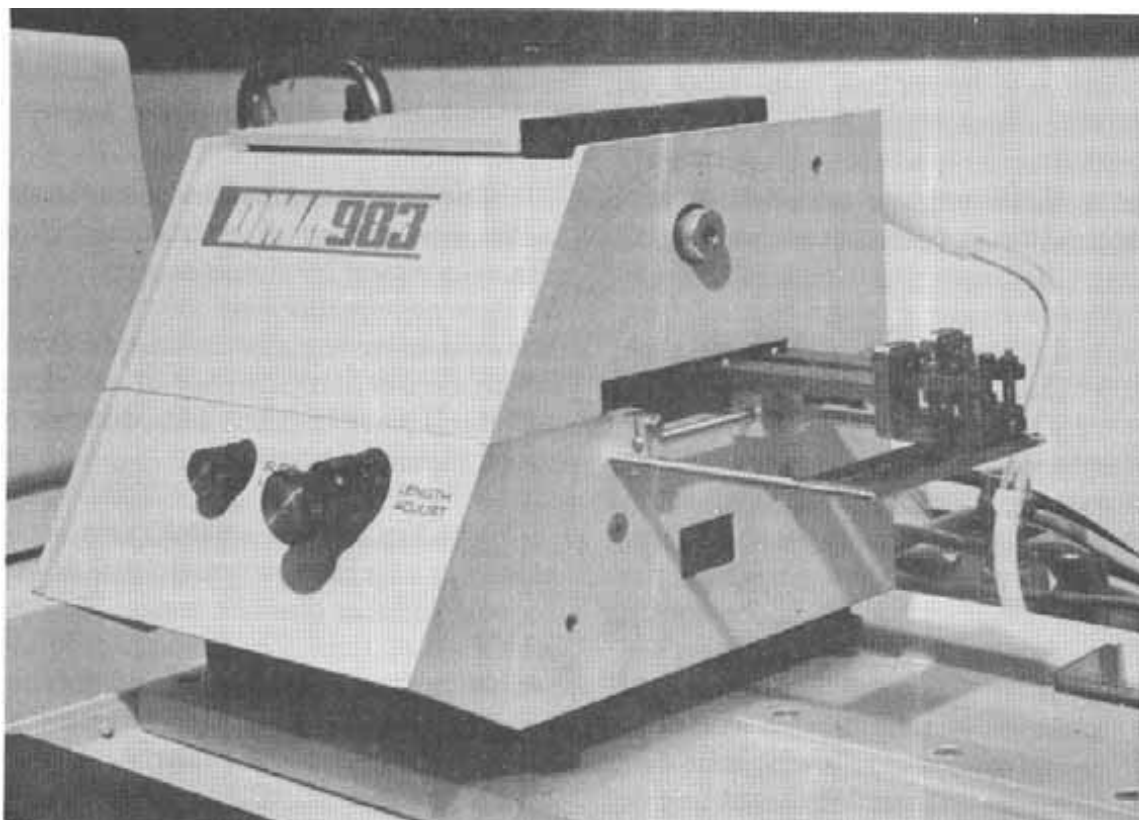


Figure 2.-- Close-up of the dynamic mechanical analyzer showing the sample-holding arms

In practice, the resin-impregnated glass cloth sample is first conditioned at room temperature in a humidity chamber (for the rest of the paper called precure conditioning). The sample is then "precured" for a period of time in an atmosphere of controlled temperature and humidity. After reconditioning (for the rest of the paper called pre-DMA conditioning) at 91 % relative humidity (RH) over a barium chloride solution, the sample is tested in the DMA to determine initial stiffness and its reaction to further heating while in the DMA.

There were numerous difficulties encountered in developing the testing technique. Substrate selection, resin application, resin shrinkage, clamping mode, clamping torque, oscillation amplitude, fixed and resonant frequency modes of DMA operation, and

isothermal and isochronal heating have been discussed in detail elsewhere (Follensbee et al in press). The precure conditioning is necessary to prevent sudden resin expansion or shrinkage with resultant crazing when the sample is exposed for a short time to the desired environmental conditions. For a dry exposure, the sample is conditioned over phosphorous pentoxide to bring it close to 0% moisture content. The pre-DMA conditioning at 91% RH plasticizes the sample, enhancing the response of the partially cured resin to DMA conditions and accentuating differences caused by precure treatments.

Storage modulus E' curves for two samples precured for two minutes at two temperatures are shown in Figure 3. Typically, a sample precured at the higher temperature (160°C)

showed a higher initial modulus. However, after exposure to further heat in the DMA, in this case at a constant temperature of 150°C, the sample cured at the lower temperature (130°C) attained a higher ultimate modulus. It was hypothesized that this phenomenon was caused partially by the lengthening of molecular chains in the precure exposure which reduced their mobility and their subsequent ability to crosslink during the final curing process in the DMA.

Heat-induced softening, counteracted by a loss of moisture, determined the shape of the storage modulus curve prior to the point where the glass transition and storage modulus rise due to rapid molecular crosslinking. Previously, the counteracting effects of heat softening and moisture loss have hindered the expansion of DMA techniques to solvent-based adhesives. Considerable progress in following resin cure has been made due to the refinement of specific procedural techniques, such as substrate selection and pre-DMA conditioning at high RH. Additionally, in the pursuit of data

interpretation, the focus has been shifted from the storage modulus E' to $\tan \delta$ (Christiansen in progress).

The area under the $\tan \delta$ curve is related to the development of mechanical stiffness. $\tan \delta$ curves for a resin exposed to 115°C for three precure times are shown in Figure 4. The areas under the curves indicate the extent to which further development of mechanical stiffness is possible and, as such, decrease with longer precure exposures.

In Figure 5, the $\tan \delta$ area has been plotted as a function of precure time at 115°C for two different phenolic resins. Compared with resin A, resin B had a higher molecular weight and contained less free formaldehyde and more NaOH. At 115°C resin B reacted at a much lower rate than did resin A. However, after precuring at 140°C, both resins developed mechanical properties at relatively fast rates, indicated by a rapid decrease in the $\tan \delta$ areas (Figure 6).

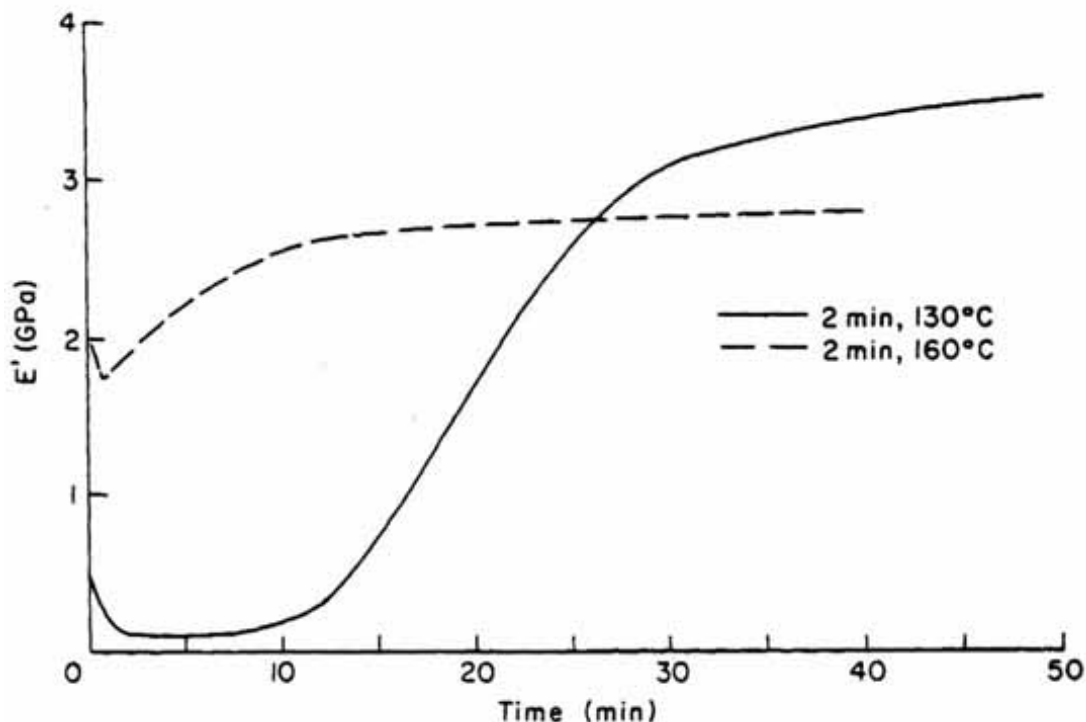


Figure 3.--Storage modulus, Resin A precured at two temperatures; temperature in the dynamic mechanical analyzer was 150°C

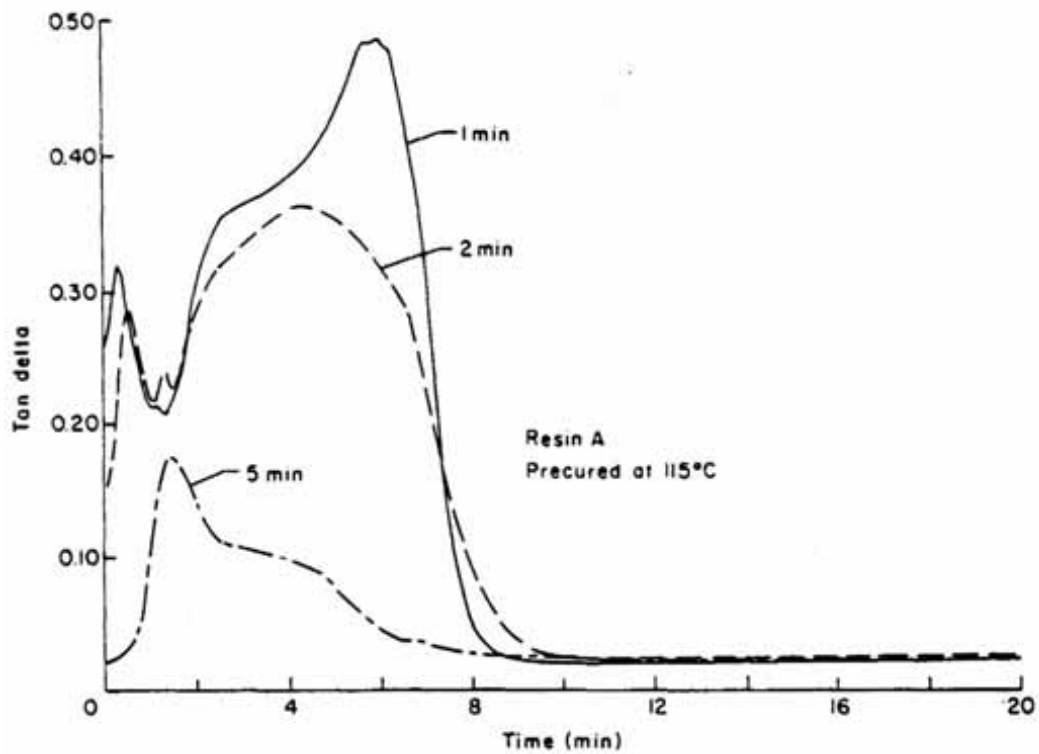


Figure 4.--Tan delta, resin A precured at 115°C for three time periods

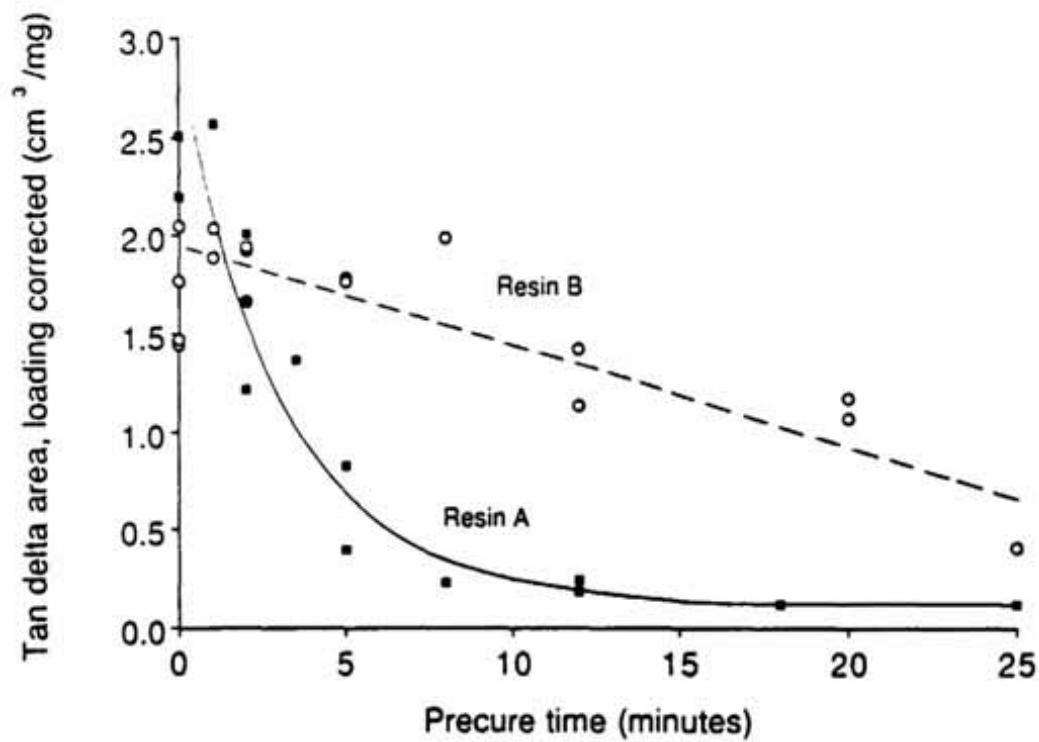


Figure 5.--Mechanical cure from tan delta area, precure at 115°C

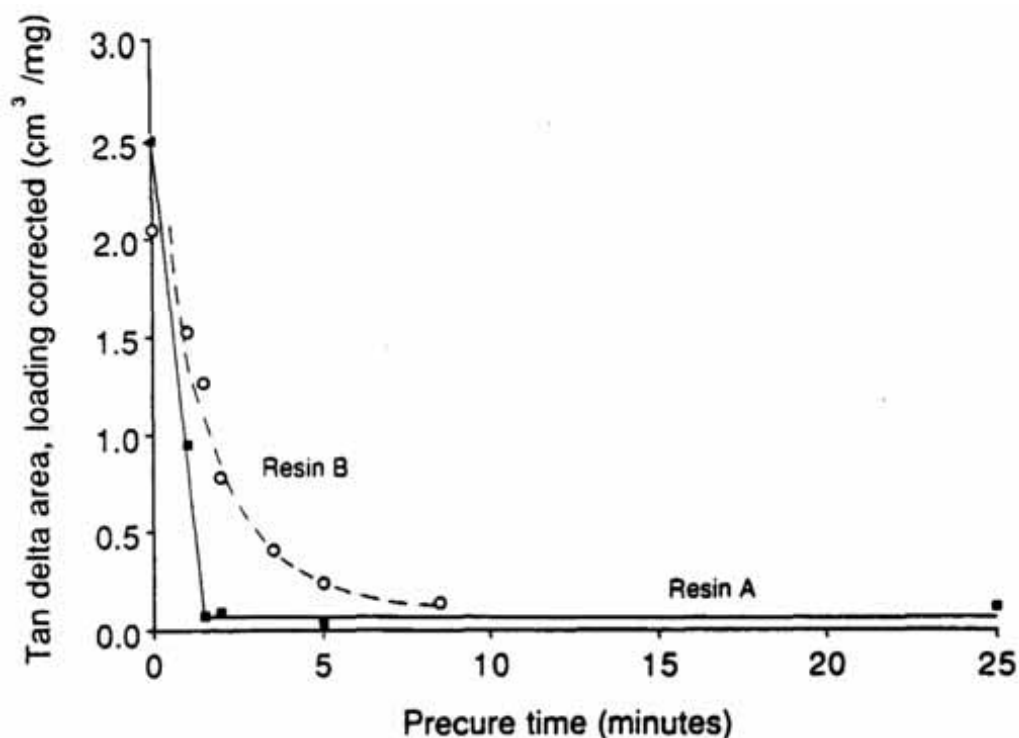


Figure 6.-- Mechanical cure from tan delta area, precure at 140°C

Chemical Advancement of a Partially Cured Resin--Differential Scanning Calorimeter Characterization

To determine how resins differ in their chemical response to environmental conditions, a Perkin-Elmer DSC-2 differential scanning calorimeter (DSC) was used to test a partially cured sample trimmed from the DMA specimen immediately following precure exposure and reconditioned to 91% RH. A 10mg sample was placed in a stainless steel capsule that had been fitted with an O-ring to prevent release of volatile components. The partially cured sample was compared to an empty control capsule. The difference in energy supplied to maintain a constant temperature rise of 10°C/min in each capsule was a measure of the exothermic reaction remaining in the partially cured resin and thus, the chemical response of the resin to heat. The weight-corrected area under the exothermic heat curve decreased with precure exposure time.

The chemical responses of two resins precured at 115°C and 140°C are shown, respectively, in Figures 7 and 8. Unlike their mechanical responses (Figures 5 and 6), the chemical responses of these two resins maintained the same general relationship at both temperatures.

Chemical-Mechanical Relations

The extent of mechanical cure of a partially cured resin sample can be expressed as the difference between the area under the DMA tan delta curve for the partially cured sample (A_c) and that for an uncured sample (A_u), expressed as a percentage of the area under the tan delta curve of the uncured sample:

$$\text{Percentage cure} = [(A_u - A_c) / (A_u)] 100$$

Likewise, percentage chemical cure can be obtained from the areas under the exothermic

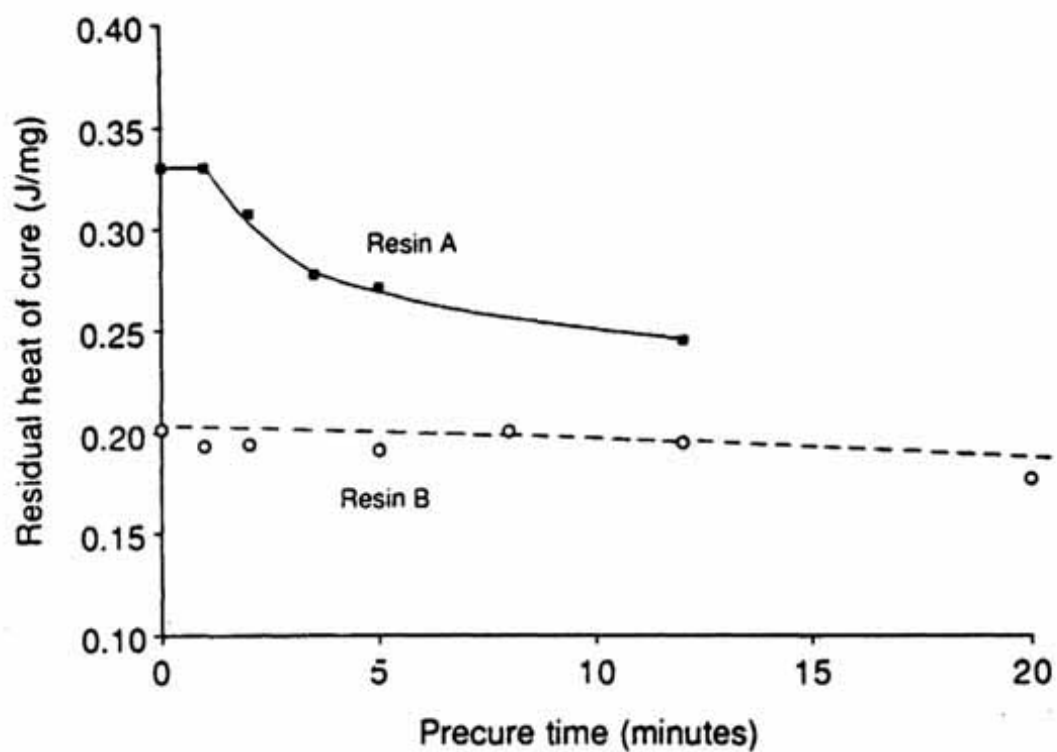


Figure 7.--Chemical cure from residual DSC heat retention, precure at 115°C

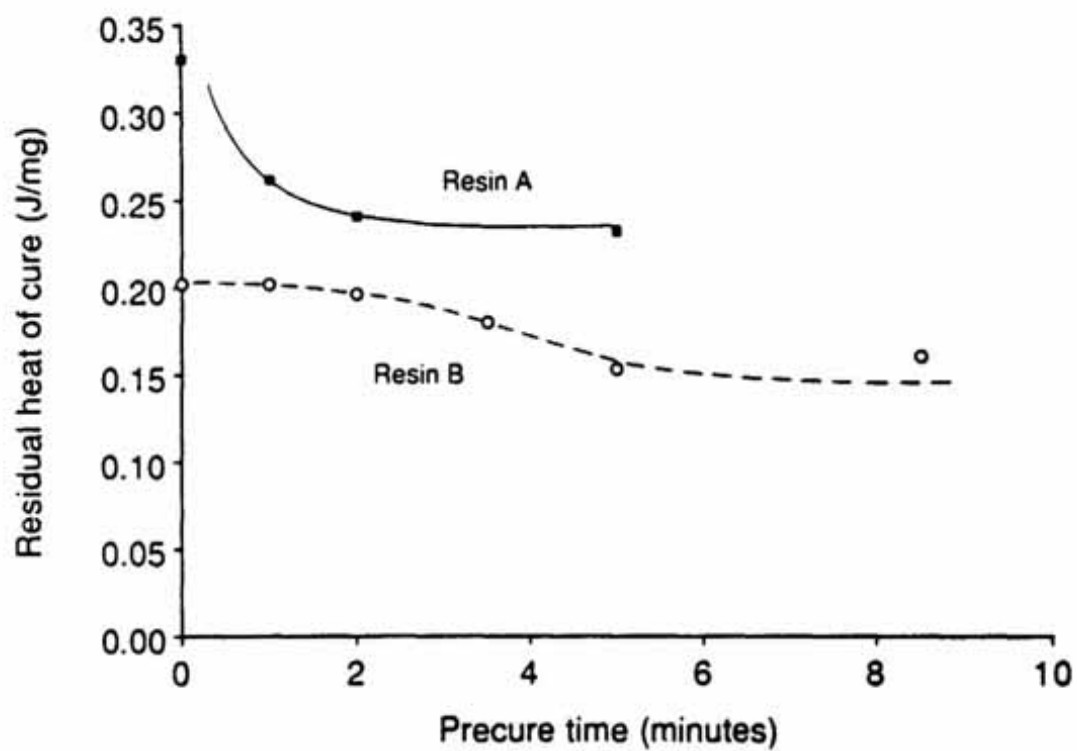


Figure 8.--Chemical cure from residual DSC heat retention, precure at 140°C

curves for partially cured and uncured DSC test samples. The relationship between percentage mechanical cure and percentage chemical cure at 115°C for resins A and B is shown in Figure 9. This type of plot is referred to as a chemical-mechanical response curve and it has been found to be very useful in comparing the responses of different resins to various environmental exposures.

The chemical-mechanical relations of the two resins at 115°C showed relatively slight differences. That is, a small increase in chemical cure (12%) caused a large increase in mechanical cure (60%) for both resins. However, the rate of increase, as denoted by the time (in minutes) associated with each data point of both chemical and mechanical characteristics, was much less in resin B. Where resin A attained 85% of its full mechanical cure in five minutes, the mechanical cure of resin B did not exceed 50% even after 20 minutes of exposure.

The relative position of the chemical-mechanical curves for these two resins changed quite drastically at 140°C (Figure 10). At this higher temperature, the mechanical cure of resin B preceded its chemical cure to an even greater extent than was observed at 115°C. Maximum mechanical cure was developed within five minutes in both resins. Chemical reactions for resin B, although generally faster and more extensive at 140°C than at 115°, lagged considerably behind those observed for resin A. A precure period of 5 - 8.5 minutes was necessary to attain 20% chemical cure for resin B, where the same degree of chemical cure was attained after only one minute of precure for resin A.

Moisture Content as a Variable

During initial developmental work, precure exposures were limited to oven dry conditions. Later experiments incorporated moisture content

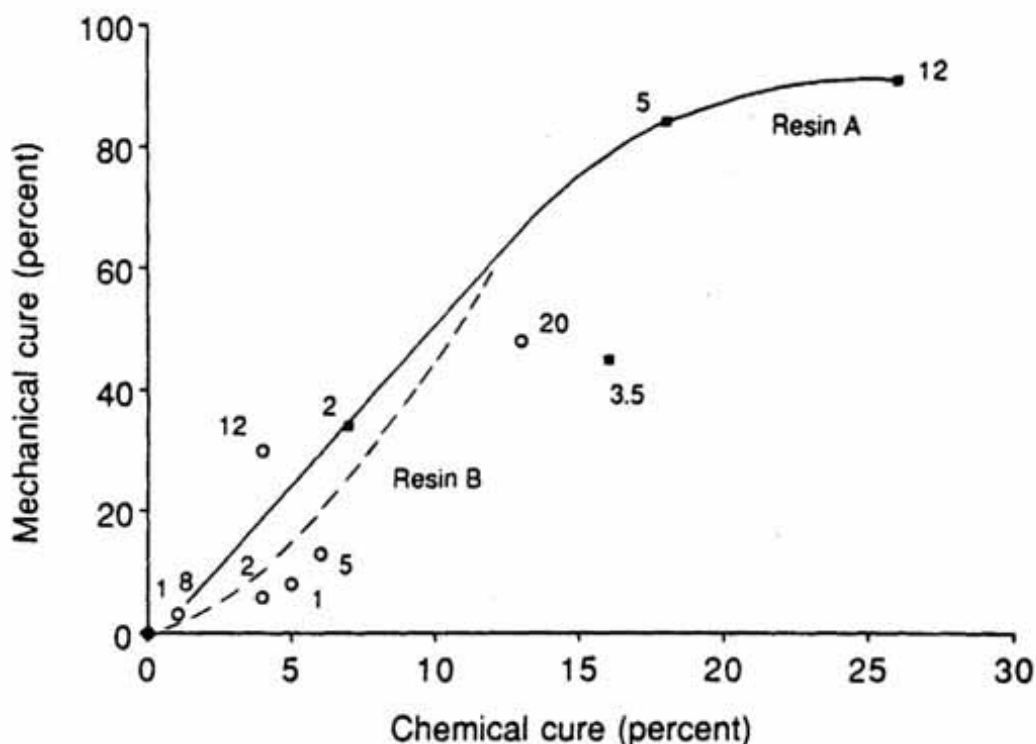


Figure 9.-- Chemical-mechanical curves, precured at 115°C, dry

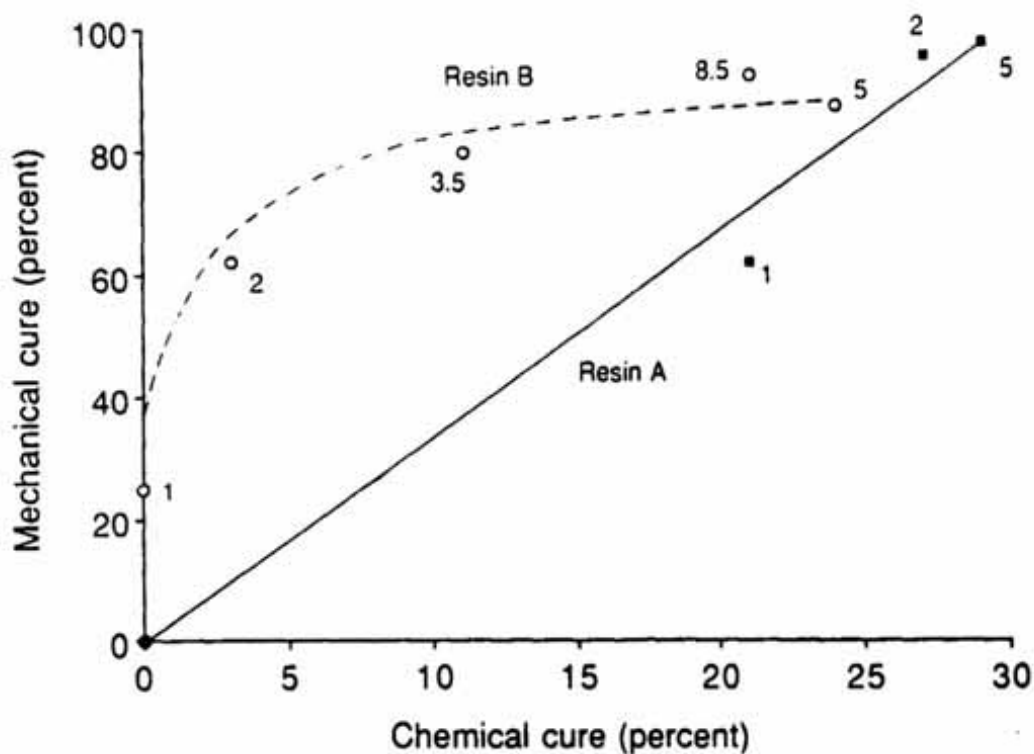


Figure 10.--Chemical-mechanical curves, precured at 140°C, dry

as a variable in the precure environment. The samples were exposed to controlled conditions of temperature and humidity in a specially designed treatment chamber (Figure 11). The desired conditions below 100°C were obtained by the additional heating of an airstream that had been saturated with moisture at a controlled temperature (Figure 12A). At these operating conditions, the chamber was assumed to be at atmospheric pressure. Conditions above 100°C were obtained by superheating saturated steam and controlling the final temperature and pressure in the treatment chamber (Figure 12B). The superheater was capable of reaching temperatures of 200°C, and the chamber was designed to withstand a maximum pressure of 150 lb/in² (1.034 MPa). This allowed the research to obtain a RH of 62% at 200°C and a RH of 100% at any temperature below 180°C. Because the chamber had to be decompressed and opened to insert the specimen, a lag of up to one minute occurred before the equipment

stabilized at exposure temperatures above 100°C. An air lock will be constructed to minimize this time lag in future studies.

Test procedures were modified to condition the test sample to the same RH at which it was exposed during precure. All precured test samples were then reconditioned to 91% RH prior to testing in the DMA. Figure 13 compares chemical-mechanical curves for resin A, precured at 115°C and at RH levels of 41 and 91%, and those obtained at dry conditions. Increasing the humidity to 41% promoted faster chemical response, but had little effect on the development of mechanical properties. Further increasing RH to 91% did not alter appreciably the rates of either chemical or mechanical cure beyond those at 41% RH.

In contrast to observations for resin A, increasing the humidity to 41% dramatically increased the mechanical cure of resin B

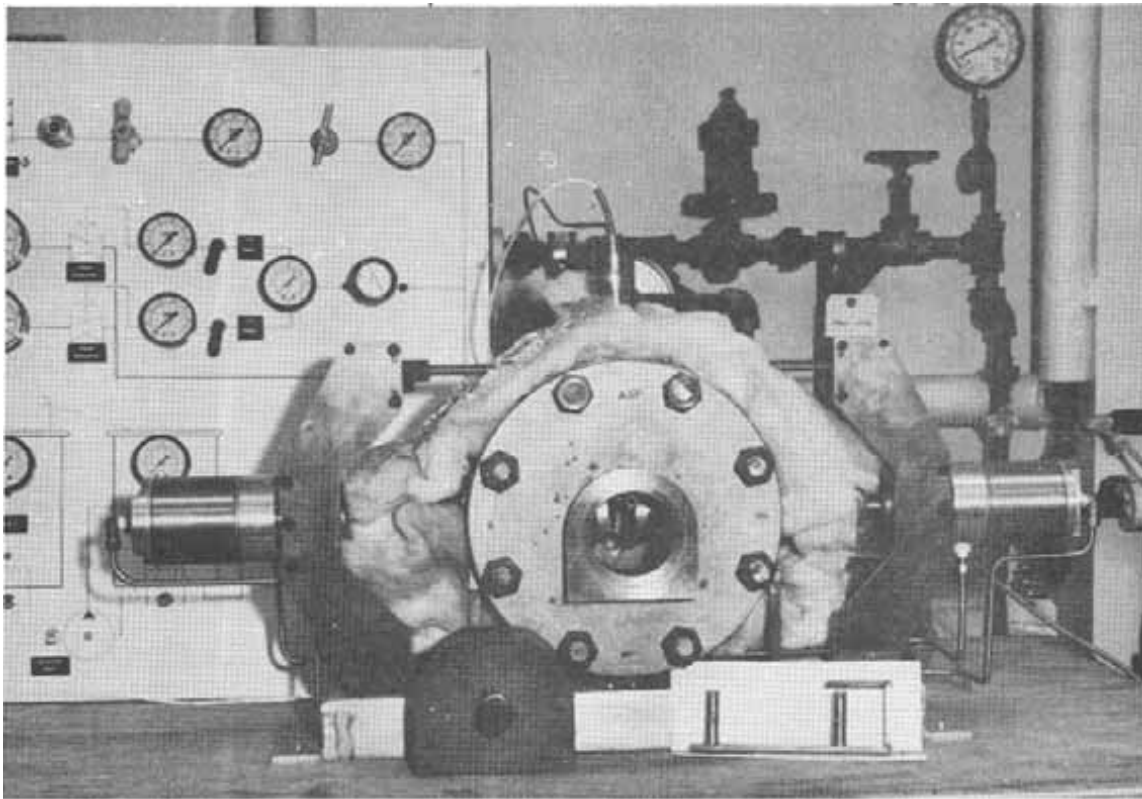


Figure 11.--Environmental chamber

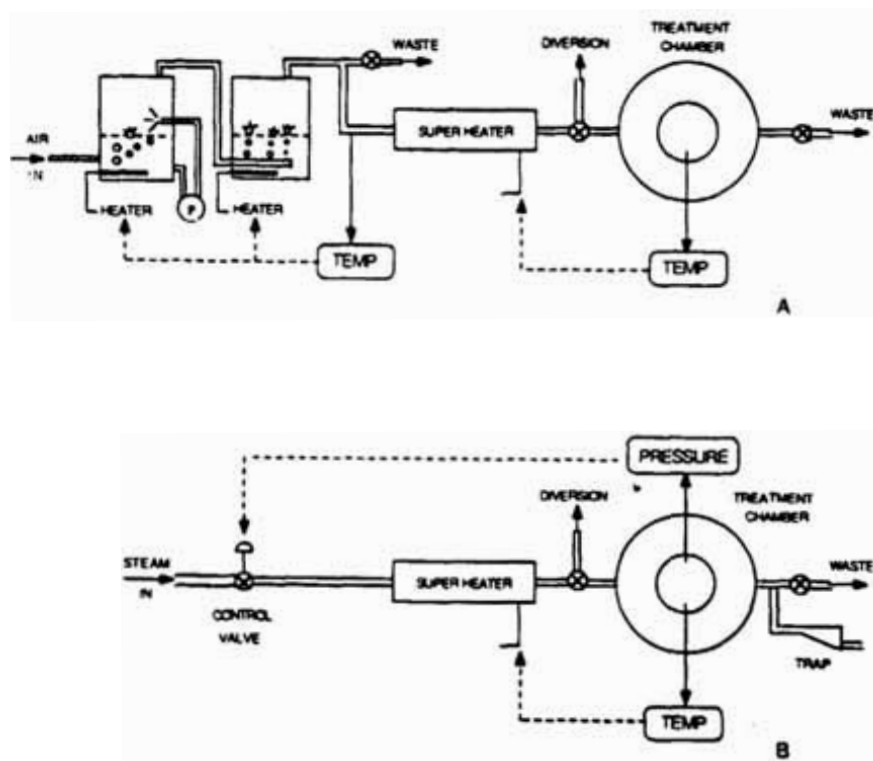


Figure 12.--Schematic of environmental control: A--below 100°C;
B--above 100°C

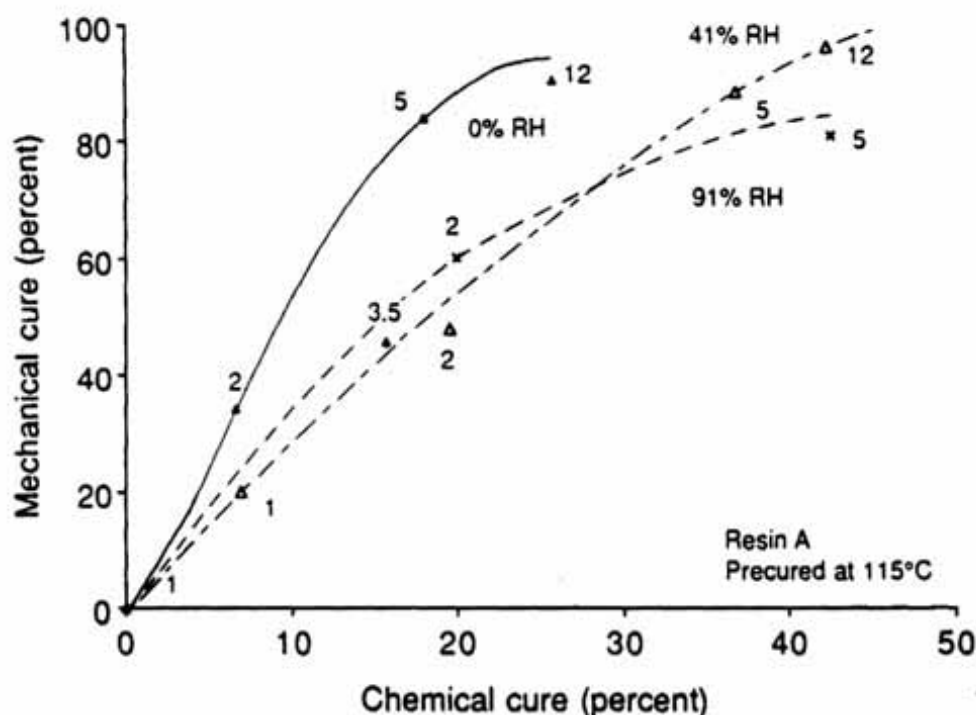


Figure 13.-- Chemical-mechanical curves showing resin A and precure at 115°C, moist conditions

(Figure 14) with only slight increases in chemical cure. Increasing the RH to 91% hastened the early development of mechanical properties and furthermore, increased the extent of chemical cure at all periods of time that were investigated.

Chemical-mechanical curves clearly provide a good measurement of a resin's sensitivity to moisture in the curing environment. Chemical changes measured by the DSC are a summation of all the reactions that occur during testing; the data do not indicate the type of chemical reactions that are taking place. Addition of moisture may affect the type of chemical reactions and, therefore, the change in rate of mechanical strength development. The study information indicates that molecular immobility, whether caused by precure exposure or by pre-DMA conditioning at low RH, reduces the formation of crosslinking reactions that occur in a curing resin. The molecular properties of

the uncured resins were analyzed using GPC, NMR, and FTIR techniques (Myers et al. submitted for publication). It is planned that the FTIR data will be expanded to include information on the structure of partially cured resins.

Flake Bonding

The curing mechanisms characteristic of a resin are important only in that they determine the formation of an adequate bond. Those environments that enhance resin curing do not necessarily provide the best situation for bonding. A technique to follow the strength development of an adhesive-bonded joint is needed. Humphrey and Ren (1989) measured the effects of time, temperature, and moisture variations on the bonding of two wood wafers that were still hot. This research was extended

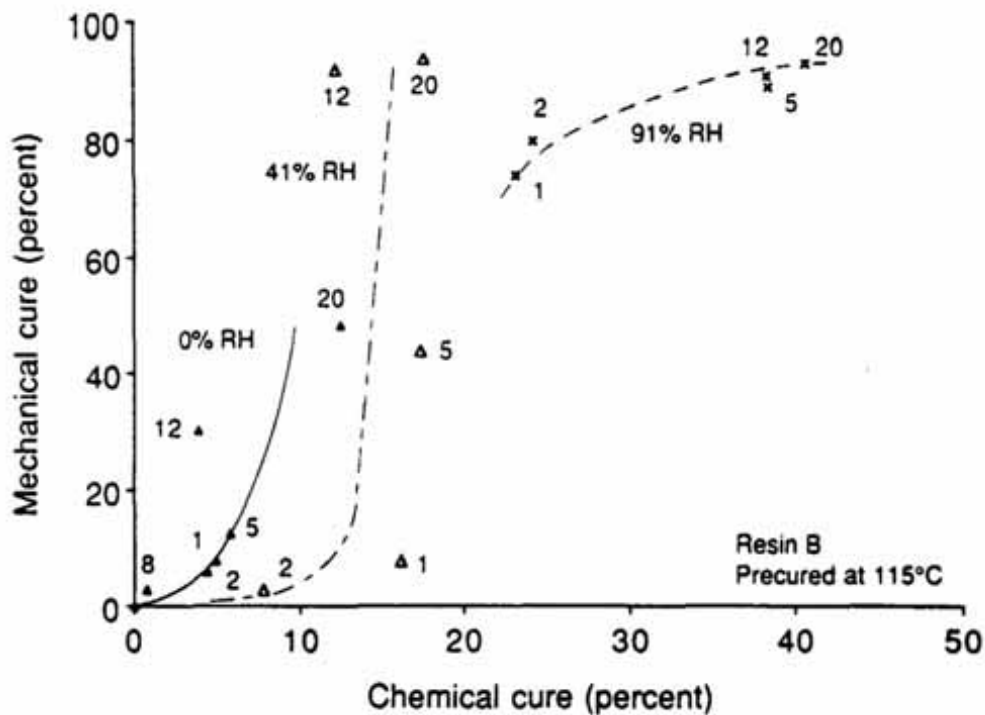


Figure 14.-- Chemical-mechanical curves showing resin B and precure at 115°C, moist conditions

using two flakes bonded in a lap-shear configuration (Humphrey and Zavala 1989). These tests are useful in determining the ability of a composite board to resist delamination forces when the press is opened. A procedure will be investigated that will indicate the shear strength of a bond that is in a cooled condition.

The technique utilizes two flakes bonded and tested in a lap-shear configuration. The bond can be formed easily in the environmental chamber (Figure 11), which is equipped with two opposing air-operated cylinders capable of applying 792 kg. This is sufficient to obtain a pressure of 40.2 MPa on the 200 mm² area being bonded. Steam or conditioned air is introduced to the chamber through the faces of the pressure platens. The 0.8 by 15 by 75 mm flakes are cut with a microtome from water-

saturated quarter-sawn blocks of aspen lumber. The knife is positioned at a slight angle to the grain and moves in a direction perpendicular to the grain. The flakes are dried under glass plates to prevent any warp or curling. Resin is applied in a 15 mm-wide strip across the end of one flake. Following a short open assembly time the second flake is positioned on the adhesive-coated flake and the assembly is bonded in the environmental chamber under exposure to the chosen conditions. Also, the flake pair can be bonded easily within, and retrieved from, a pressed board. Testing is accomplished in a standard tension tester (Figure 15). The flake-bonding work to date has been exploratory in nature with emphasis on the development of a technique. Initial results indicate that the method will be useful in determining the influence of environment on the development of bonds by specific resins (Figure 16).

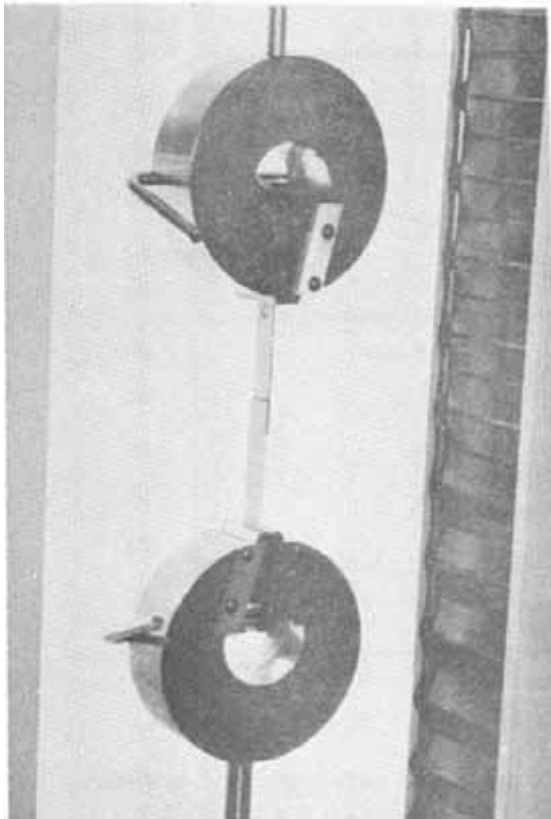


Figure 15.--Lap-shear test specimen

Measurement of Pressing Environments

The authors have been cooperating closely with Dr. Fred Kamke and Steve Johnson at Virginia Polytechnical Institute and State University (VPI) on the development of board-pressing data. Early work by Kamke and Casey (1988), Brady and Kamke (1989), and Wolcott (1989) defined many of the basic relationships between board pressure, temperature, and vapor pressure in conventionally pressed boards. Temperature and vapor pressure in these boards changed relatively slowly. Vapor pressure differentials throughout the thickness and width of the conventionally pressed board were slight and lasted for relatively short periods of time (Figure 17). Temperatures and vapor pressures in steam-injected boards, on the other hand, changed rapidly and drastically. Vapor pressure differentials throughout the board may be large and remain for a relatively long time (Figure 18).

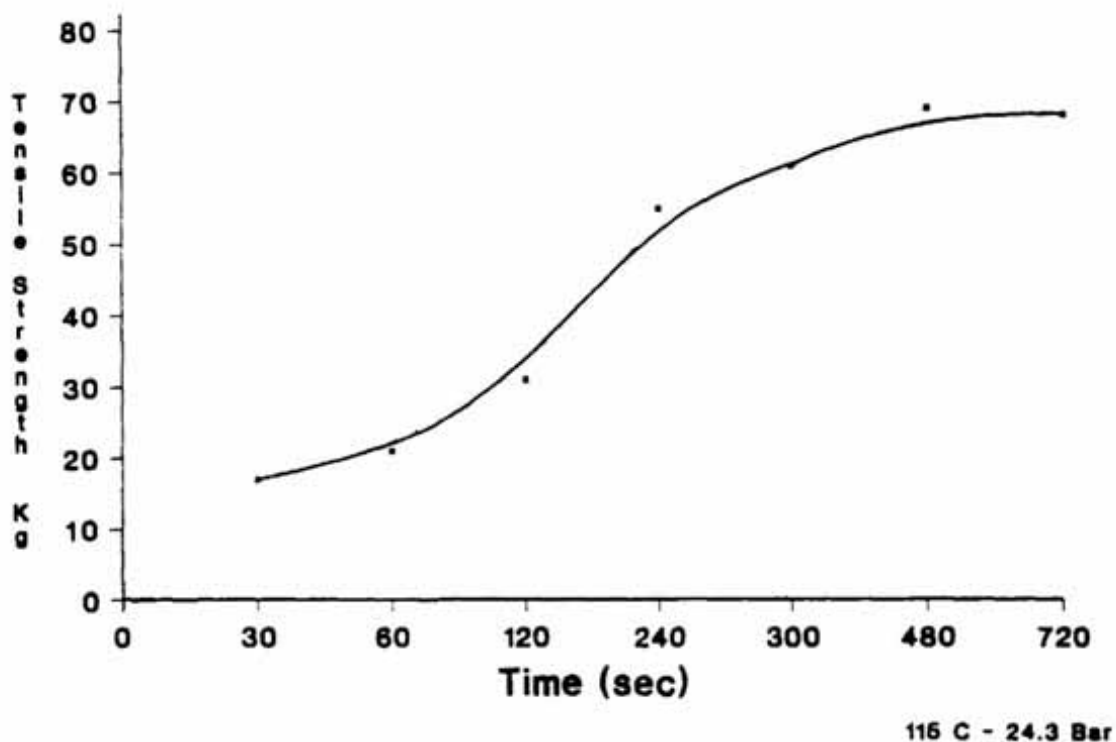


Figure 16.--Bond strength development showing resin A at 115°C, dry

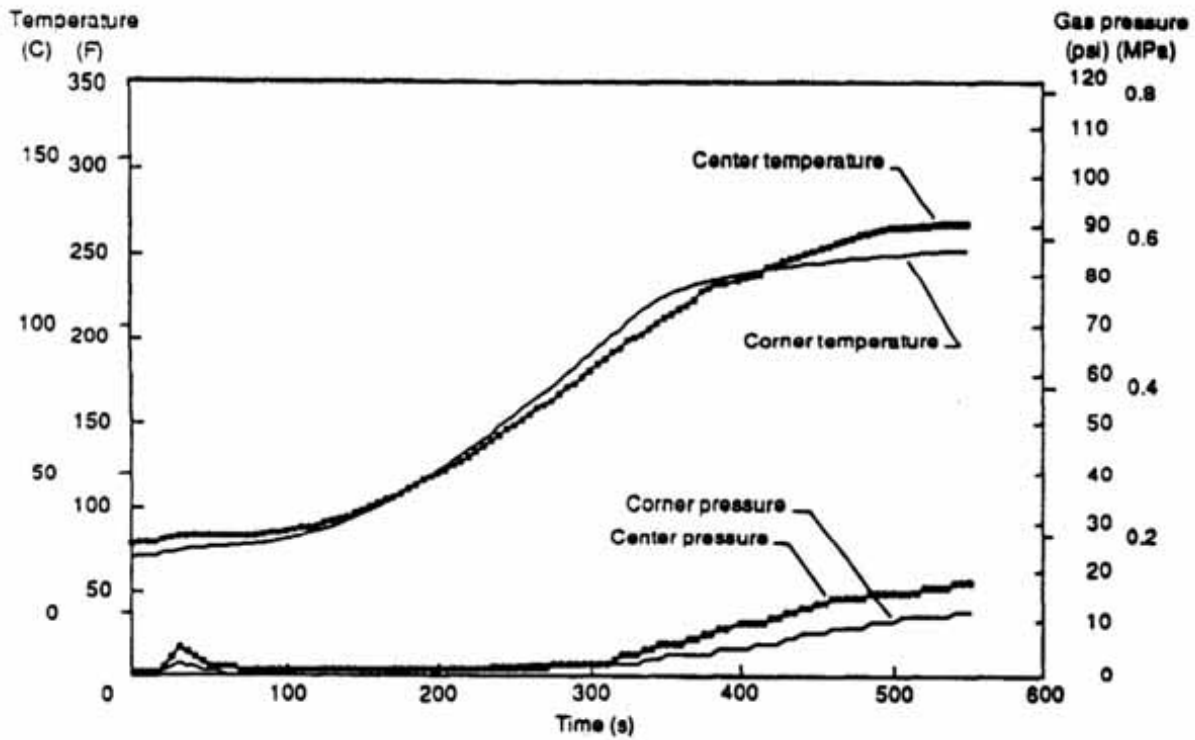


Figure 17.--Temperature and vapor pressure within conventionally pressed boards

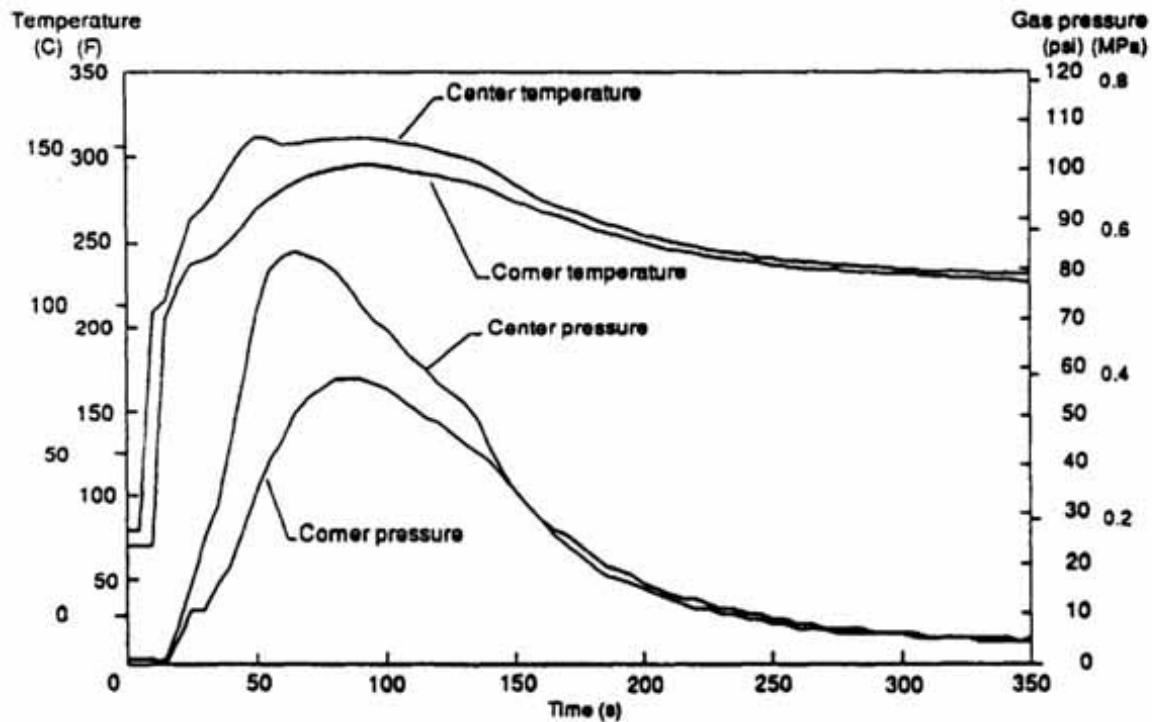


Figure 18.--Temperature and vapor pressure within steam-injected boards

Resin Penetration

The lapped flakes used in testing adhesive bonds can be sectioned readily to determine resin penetration. Work begun at the Forest Products Laboratory in this area is now being continued at VPI. A technique has been developed to dye a microtomed section of the flake pair and observe the bond line under a fluorescent light microscope (Figure 19). The technique works well with phenolics and isocyanate resins. Photographs of the bond line are digitized in an image analyzer, and the data are used to quantitatively determine the degree of penetration.

Practical Application

Each of the seven discussion subjects described so far in this paper are, by themselves, interesting from an academic standpoint. Combined, their value increases. In conjunction

with other modern chemical and molecular analyses such as FTIR and NMR techniques, the information provides extremely useful guidelines in synthesizing a resin for a specific application or in modifying or controlling a process within the parameters defined by the resin's characteristics. As with any characterization process, the information describing a specific resin is more meaningful as the data base expands. It is hoped that the knowledge of resin behavior will be broadened by investigating other phenolics, ureas, and isocyanates. Incorporation of new techniques such as dielectric or sonic cure measurements may also prove beneficial. Finally it may be useful, especially from the standpoint of improved dimensional stabilization, to combine a resin's curing and bonding characteristics with information describing the viscoelastic reaction of the wood substrate under the same environmental conditions.



Figure 19.-- Resin penetration: photomicrograph under fluorescent light for isocyanate-bonded aspen flakes

CONCLUSION

This paper has outlined research designed to characterize a thermosetting resin with respect to its time-related response to the environmental conditions of temperature and moisture content. Techniques have been developed to determine the progressive increase of mechanical properties during cure using a dynamic mechanical analyzer. The chemical advancement of a partially cured resin can be related to its residual exothermic activity as determined in a differential scanning calorimeter. Chemical-mechanical relations have been developed for several laboratory-synthesized phenolic resins under dry conditions. The data show that development of mechanical properties is not linear with the progression of chemical activity and that this relation changes within and between resins at different temperature exposures. Initial materials incorporating moisture as a variable have indicated that chemical and mechanical cure can be either enhanced or inhibited, depending on the original chemical state or molecular structure of the resin.

Techniques to monitor bond strength development in individual flakes are being established. In addition, progress has been made in quantifying resin penetration. Data describing the environmental conditions of temperature and vapor pressure that occur during both conventional and steam injection pressing operations are now available. Information from all these tests is necessary to determine the relations among resin cure, resin bonding, and final board strength under actual pressing conditions.

Advances in understanding the curing and wood-bonding mechanisms of thermosetting resins depend on developing data in several related areas. The characterization of many resins using the techniques described and alluded to in the paper affirm the authors' belief that a

more meaningful approach to resin synthesis can be developed.

ACKNOWLEDGEMENT

This material is based on work supported by the U.S. Department of Agriculture Competitive Research Grants program under agreement 65-FSTY-9-0079.

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