

THE EFFECT OF HOT-PRESSING PARAMETERS ON  
RESIN PENETRATION AND FLAKEBOARD LAYER PROPERTIES

by

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(ABSTRACT)

The area of penetration of phenol-formaldehyde resin into aspen (Populus tremuloides) and Douglas-fir (Pseudotsuga menziesii) flakes, and the layer properties of yellow poplar (Liriodendron tulipifera) flakeboard were investigated to determine how they were influenced by various pressing parameters. The evenness of penetration was found to be a function of the natural variability of the wood and was not influenced by the pressing parameters of temperature, moisture content, pressure, or time. These four parameters were found to influence the area of penetration by controlling the viscosity and flow of the resin.

The temperature, gas pressure, and platen pressure history at any particular plane through a flakeboard panel thickness were found to be directly and interactively determined by the pressing parameters of platen temperature, initial mat moisture content, and press closing time. The specific gravity profile was observed to be a function of the press closing time while platen temperature and the mat moisture content influenced the amount of springback which the panel exhibited. Layer-shear and the corresponding specific gravity at a particular plane

were found to be similarly dependent on environmental history. The layer-shear strength increased consistently toward the surface of the panel and exhibited a lower coefficient of variation nearer the surface.

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## INTRODUCTION

The utilization of timber resources of decreasing size and quality has proven a significant challenge to the forest products industries. The development of non-veneer structural panels (NVSP) has been fostered, in part, by various competitive advantages, such as the utilization of a low value timber resource. An additional advantage for the NVSP industry is available from reduced shipping costs as a result of locating manufacturing facilities nearer residential markets. Locating facilities in proximity to these markets requires that present technology be expanded to utilize the species which are readily available in the particular region.

Further development of NVSP has led to reducing costs of production and improving product performance through the design of products and more efficient utilization of the wood and resin. The adhesive component generally constitutes about 25 to 30 percent of the total material cost of NVSP. The efficient utilization of the materials, therefore, requires a greater understanding of the adhesion process in these panels.

The process of hot-pressing NVSP can be described using the principles of heat, mass, and momentum transfer. The process is mathematically complex and difficult to model, though some progress has been made in this respect (Harless et al., 1987). The measurement of the real-time pressing environment at various layers through a mat provides essential empirical data to relate such conditions to the process of adhesion and the subsequent bond strength.

NVSP is used extensively in residential construction as sheathing and roofing. In these applications, the most critical performance characteristics of the panel are the bending strength and stiffness. The method of transformed cross sections based on the vertical density gradient can be used to model NVSP in bending. The adequacy of using this technique is dependent on the ability to measure the vertical property distribution through the panel. An expanded understanding of the formation of both the density gradient and the mechanical property distribution as influenced by the local environment created by various combinations of pressing parameters will provide the information necessary to manufacture panels to meet specific mechanical requirements with the efficient utilization of materials.

## OBJECTIVES

The overall goal of this investigation was to provide experimental data and techniques which will facilitate the efficient engineering design of structural flakeboard. Four objectives contributed to this overall goal. The first objective was to determine whether hot-pressing parameters affect the penetration of phenol-formaldehyde resin into thin wood flakes. The pressing parameters examined were platen temperature, platen pressure, initial flake moisture content, and pressing time.

The second objective was to determine if the pressing parameters and the local environmental conditions created during hot-pressing affect the layer-shear strength and specific gravity at various layers through the panel thickness. The third objective was to establish an empirical relationship between layer-shear strength and the local specific gravity in flakeboard panels. The fourth objective was to determine if in situ resin penetration was related to layer-shear strength in structural flakeboard. The pressing parameters of platen temperature, initial mat moisture content, and press closing time; and the localized temperature, gas pressure, and platen pressure history were examined.

## LITERATURE REVIEW

### Wood Adhesion

Bateup (1981) defined adhesion science as the study of molecular forces between bodies and the prediction of adhesive-joint strength from the magnitude of these forces. He further defined adhesion on a molecular level as the state in which an interface is formed between two bodies such that the molecular forces across the interface resist interfacial separation. Our present understanding of adhesion is well represented by the absence of a universal theory of adhesion. The actual mechanism of adhesion probably is very different in many systems and most of these mechanisms are not fully understood.

Three main theories have been proposed to account for the phenomenon of adhesion (Bateup, 1981). The first is the adsorption theory of adhesion. The adsorption mechanism requires good interfacial contact and van der Waals interactions are believed to be sufficient for adhesion. A second prevalent theory is the mechanical interlocking theory. This theory provides for adhesion on porous or rough substrate surfaces. Increased difficulty is encountered in wetting such surfaces due to the uneven topography. The third adhesion theory is the diffusion theory. This results from interdiffusion across the substrate surface, and is dependent on molecular mobility and mutual solubility.

Two mechanisms are argued to provide for the largest proportion of the adhesion of wood surfaces. Mechanical adhesion or mechanical interlocking is facilitated by the interdiffusion of the adhesive into

the porous structure of the wood substrate. The second mechanism which Bateup (1981) identifies as contributing significantly to wood adhesion is chemisorption. Chemisorption attributes adhesion to chemical interactions such as, covalent or hydrogen bonding, dipolar interactions and dispersion forces. Chemisorption is similar to the adsorption theory but provides for the interaction of additional chemical bonding forces. The actual mechanism of wood adhesion is probably a balance of these two component mechanisms. The relative balance of one mechanism versus the other being dependent upon many characteristics of both the wood substrate and the adhesive.

Blomquist (1981) described an adhesive as a substance capable of holding materials together by surface attachment. The adhesion system can be represented using a 5-link analogy. Links 1 and 5 are the adherend, links 2 and 4 are the adherend-adhesive interphase, and link 3 is the adhesive. The chain analogy is also directly applicable to the mechanics of the bond. The entire bond is only as strong as the weakest link. The interphase regions of the chain analogy are further divided into various links by the relative concentration of the adhesive present. The five link analogy is only a simplified representation of the major regions of the adhesive interface.

The formation of an adhesive bond involves several steps. The adhesive must first wet and spread on the substrate to provide for adequate surface coverage. The internal strength of the adhesive must then be established to ensure permanency of the bond. Processes which provide for the development of the adhesive strength include: solvent drying, hot melt or fusibility (thermoplasticity), pressure application,

and chemical reaction.

Bryant (1977) indicated the necessity for an adhesive to penetrate to the level of solidly attached fibers to facilitate adequate wood adhesion. At this depth, the adhesive must then adhere to the wood substrate by specific adhesion or secondary valence forces. In most wood/adhesive systems, the adhesive is considered to be stronger than the wood component. The wood or some area within the wood/adhesive interphase should therefore prove to be the weak link in the chain analogy mentioned previously.

Bryant (1977) also indicated that wood fracture, whether through failure or machining, could be characterized using two failure mechanisms. If wood fractures between fibers, the fracture is described as intrawall or intracellular. If wood fractures within the fiber, the fracture is described as transwall fracture. Transwall fracture may occur parallel, perpendicular, or at some angle to the axis of the fiber. These two mechanisms of failure expose different chemical and topographical surfaces which interact differently in both chemisorption and mechanical interlocking.

When wood experiences intracellular fracture, the middle lamella is exposed. This provides a surface rich in lignin. When transwall fracture occurs, two surfaces are exposed. Depending on the location of the fracture, more or less of both the cell wall and the lumen are exposed. Exposure of the cell wall provides a surface rich in oriented cellulose but poor in lignin. Conversely, the lumen surface is rich in lignin-type compounds but poor in cellulose content. The exposure of the hygroscopic cellulose should provide for greater mechanical

interlocking and greater resin penetration with aqueous resin systems. The exposure of hydrophobic lignin surfaces should reduce the extent of mechanical interlocking due to reduced penetrability.

### Substrate Surface

The surface of a wood substrate has a significant influence on the adhesion process. Burrows (1961) used various light microscopy techniques to observe resin efficiency in particleboard manufacture. The findings from his investigation indicated that the depth of damage on flakes was only one to two cells regardless of the moisture content of the wood at the time of flaking. He hypothesized that the mechanism of adhesion in particleboard was a 'spot weld' phenomenon rather than a continuous thin film. He associated a smoother surface with better resin efficiency. Reduced efficiency was observed to result largely from failures and microcracks in the surface of the flakes.

Koran and Vasishth (1972) completed a similar study of plywood gluelines using scanning electron microscopy (SEM) techniques and shear ply tests. They found that a rough wood surface and high moisture content resulted in low values in shear ply tests. Jokerst and Steward (1976) obtained similar results in observing that the wood substrate surface significantly effects the quality and durability of the adhesive joint. Low shear ply values were attributed to the uptake of resin into capillaries exhibited on rough surfaces. Low shear ply values obtained from high moisture content specimens were believed to result from overpenetration of the resin due to decreased viscosity. Dried-out

gluelines were found to result from the lack of glue transfer, glue flow, wetting, and penetration. All of these phenomena were indicative of a high resin viscosity. Intrawall and parallel transwall fracture constituted most of the failure surfaces. Perpendicular transwall fracture was found to be rare.

Similarly, Wilson and Krahmer (1976) applied SEM techniques to describe the failure surface and failure mechanism characteristic of internal bond test specimens from particleboard. Observations of the failure surface revealed that the micro-characteristic fracture in particleboard is similar to that observed in plywood. Again a relationship was found to exist between the initial surface of the particles and the amount of transwall and intrawall failure. Their observations indicated that the mechanism of bonding, whether spot weld or continuous thin layer, is dependent on the surface of the flakes as adherend. Additionally, they observed a relationship between decreased resin droplet size and increased resin efficiency measured as internal bond.

Wellons, et al. (1982) used SEM as well as light and fluorescence microscopy in observing that the major zone of compressive failure in hot pressed plywood occurred at or near the glueline. They also observed that many earlywood fibers located in proximity to the glueline were saturated with adhesive. Latewood fibers exhibited substantially less compressive failures and consequently, reduced saturation with adhesive. Apparently, the filling of the lumens with adhesive was related to the extent of cell wall failures which would seem to provide an avenue for adhesive penetration.

## Resin Penetration

Several approaches have been used to examine resin penetration into wood surfaces. Tarkow, et al. (1964, 1966) conducted two investigations on the interaction of wood with polymeric materials. Using poly-vinyl-acetate (PVA) to determine the adsorption characteristics of sitka spruce, they concluded that wood exhibited a greater adsorption for polymers which had a greater capacity for hydrogen bonding. In the second study, they investigated the relationship between the size of a molecule and the capacity of said molecule to penetrate the surface region of wood. The penetration of a series of poly-ethylene-glycol (PEG) compounds of increasing size was measured to determine the limiting size for penetration into the cell wall of wood. They found that the limiting size for penetration of a water soluble material into green wood is a PEG molecule with a molecular weight of 3000. This is significant in that the size of most phenolic resins is less than the size of the limiting PEG-3000 compound, though molecular configurations also must be considered.

Tarkow, et al. (1966) hypothesized that the void size in partially swollen wood may control penetration of resin into the surface region. Additionally, elevated temperatures were believed to influence penetration in two ways. Since resin penetration may be dependent on the void size, high temperatures increase the void size and consequently, increase the amount or depth of penetration. Penetration may also be dependent to some degree on micromolecular motion. Again,

elevated temperatures increase such motion and consequently would increase penetration.

Siau, et al. (1978) completed an investigation related to Tarkow's study on the interaction of polymers with wood. Using an impregnation technique, they found that 0.88 fraction of the voids which existed before treatment were filled during the impregnation process. From these results, they concluded that excellent penetration into wood was obtainable. The depth and amount of penetration was greatest in sapwood and juvenile wood.

In one of several investigations which Stamm completed on the penetration of liquids into wood (1973), he concluded that longitudinal penetration of hardwoods by water and toluene was almost instantaneous. However, transverse penetration was found to be directly proportional to the square root of time. The empirical relationship that Stamm recorded was that the rate of longitudinal penetration was about 32.5 times that of the transverse penetration.

Wilson and Kraemer (1978) investigated the correlation of resin wetting and molecular weight (MW) distribution to internal bond and MOR. Light microscopy photographs of phenolic and ligno-sulfonate resin droplets yielded poor correlations to board strength. These results led to the conclusion that wetting is not a sufficient condition for good bonding, and that adsorption and interdiffusion were essential in the adhesion process. They also used refractometry and gel permeation chromatography (GPC) to characterize the molecular weight distribution of the resin at various pH conditions and resin viscosities. Wilson and Kraemer (1978) found that as the viscosity was increased at constant pH,

the average MW also increased. They also found that as the pH was increased at constant viscosity, the average MW increased. When MW was correlated to Internal bond (IB), a significant relationship was observed. A higher average molecular weight yielded boards with higher IB values ( $r=0.905$ ). The lower values of IB recorded for the lower average molecular weight resins was attributed to overpenetration of the smaller molecules into the wood substrate. This relationship was found to hold for the MW distribution as well. Resins with molecular weight distributions skewed towards higher values corresponded to boards with higher IB values. Resins with more low and medium weight molecules corresponded to boards with lower IB values.

Several investigators have made observations of the penetration of resin into wood using various techniques. Smith and Cote completed a study (1971) of resin penetration into the wood cell wall using scanning electron microscopy (SEM) and energy-dispersive X-ray analysis (EDAX). They found that the EDAX technique was useful for determining the depth of resin penetration only when the resin was tagged in some manner. Using this technique, they were able to identify a resin concentration gradient across the cell wall at the wood-adhesive interphase. The concentration of resin was observed to be greatest in secondary walls near lumens which were filled with resin. The absence of the bromine tracer in the middle lamella indicated that resin probably did not penetrate in this region. Penetration was also found to be deeper in the earlywood cell walls than in the latewood cell walls.

Hare and Kutscha (1973) observed shallow penetration of phenolic resin used in bonding thin wood plies. Overpenetration was found to

reduce shear-ply values. The penetration which did occur was primarily into wood failures. The researchers believed that the strongest bonds developed when penetration was limited to the depth necessary to reinforce damaged fibers. Maximizing the contact surface was believed to be of paramount importance. They found that the adhesive should penetrate and make contact with the walls of the lumens to maximize the contact surface. They also hypothesized that the evenness of penetration may well be as important as the depth of penetration in providing high quality gluelines with the most efficient use of the adhesive.

White, et al. (1977) found that the penetration of resin into southern pine earlywood was more extensive than the penetration into latewood of the same species. Consequently, a low bond strength was found to exist between latewood surfaces (White, 1977). Significant penetration was also observed into the rays. Observations were facilitated by tagging the resin with bromine and subsequently taking serial sections parallel to the glueline, the depth of penetration was determined using neutron activation analysis (NAA). White, et al. (1977) suggested that the bromine used to tag the resin may have been liberated to some extent during the curing process. The bromine has a high vapor pressure at room temperature and may therefore diffuse into the wood more readily than the resin.

Gallob, et al. (1985) investigated the relationship between the characteristics of PF resins and the performance of the adhesive. Plywood specimens were made using different ratios of neat resin, extender, and filler. They found that the percent wood failure in

shear-ply specimens was related to the weight average molecular weight of the neat resins and the relative branching of the adhesive. High average molecular weight adhesives were found to reduce the values obtained for the shear ply specimens. The reduced shear ply values were believed to be associated with the poor flow properties characteristic of high molecular weight molecules. Adhesives with a high relative branching index were also found to reduce the values obtained in the shear ply tests. This phenomenon was apparently due to the escape of moisture into the wood substrate.

The observation was made that when the resin is mixed with extenders and fillers, water is trapped and the water loss properties associated with the neat resin are masked. Resins with a low average molecular weight and a low branching index were found to be least sensitive to long assembly times. This was probably due to the ability to hold water and avoid dry-out. Additionally, fluorescent microscopy was used to examine the penetration characteristics of the different resin blends. Resins which permitted the rapid escape of water were found to be deficient in penetration. Penetration was restricted to a depth less than that necessary to anchor torn and damaged fibers. No relationship was observed between the resin gel time and the resin performance.

Bolton, et al. (1985) used EDAX techniques in their study on the microdistribution of urea-formaldehyde resin. Their results showed a continuously varying pattern of resin concentration. They hypothesized that these results were indicative of a thin continuous film in contrast to the generally supported theory that the mechanism of adhesion in non-veneer wood panels was a spot-weld phenomenon. Their results also

indicated that resin penetrated 2 to 3 cells into wood in good quality chipboard. Included in these findings was evidence of the penetration of the resin into cell walls. The investigators were unable to determine any systematic variation in the degree of penetration in the different cell wall layers. They were also unable to determine a microdistribution difference which would account for the large performance difference between phenol and urea based formaldehyde resins.

Furuno, et al. (1983), and Youngquist, et al. (1987) used fluorescence microscopy to evaluate various properties of resin distribution in particleboard. Both studies indicated that fluorescence microscopy was an excellent technique for the identification of phenolic resin. Furuno, et al. compared several other techniques, including SEM, and determined that fluorescence yielded better results in analyzing the characteristics of resin distribution than the other techniques.

### Resin Cure

Phenol and formaldehyde in aqueous solution with NaOH dominates the plywood and much of the exterior particleboard/flakeboard industries. Some of the reasons for this are the ease of processing, low cost relative to alternatives, minimal emissions from formaldehyde, common solvent (water), waterproof glue line, infusibility, and durability once cured. PF resins are a mixture of polymer molecules of different MW, shapes, and degree of substitution of methylol groups and methylene linkages. Due to the variety of formulations which are synthesized, the

exact molecular structure of PF resins is relatively unknown. Several investigations have, however, provided a substantial understanding of the influence of various parameters on the curing of these resins.

Chow and Mukai (1972) studied the degree of cure of phenolic resin at high vapor pressure using the ultraviolet spectrum of the resin with water as a solvent. The degree of cure was found to be linearly related to the moisture content of the resin. A higher moisture content was found indicative of a lower degree of cure. With the moisture content of the resin held constant, the time-temperature relationship to 80% cure was found to be curvilinear. As one might expect, a longer cure time was found to provide for a higher degree of cure. At low temperature, 100°C, the time required to achieve 80% cure is dependent on amount of moisture present. At high temperature, 140°C, the time required for 80% cure was found to be less dependent on moisture content.

Pillar (1966) conducted a study in efforts to describe the curing process of resin. Using methods of free vibration and rate of strength development, the curing of resin was described in terms of two transition points. The gel transition was characterized by the existence of an average of a single crosslink per molecule and the cessation of steady-state flow. The glass transition was characterized by the discontinuity of the thermal expansion coefficient. Glass transition involves the hardening of amorphous polymers and the onset of brittleness. Pillar also indicated that the three types of motion related to resin cure are: gross movement (Newtonian mechanics), segmental motion (viscoelasticity), and bond movement (Hooke's law

elasticity).

Chow et al. (1969) conducted an investigation into the curing of resin, using a spectrophotometric technique to measure the degree of cure of a phenol formaldehyde resin. Chow found that below 212°F, polymerization of the particular PF resin was through a condensation reaction. As one might expect, the presence of preexisting moisture inhibited the cure of the resin. A linear relationship was found to exist between percent wood failure in shear and percent resin cure. This relationship was found to be curvilinear for wet strength samples tested for internal bond. Additionally, the influence of additives and extractives was found to be negligible.

Chow (1977) used techniques of infrared spectrometry, differential thermal analysis, and softening temperature in describing the curing of resin using thermodynamic principles. The curing of phenol-resorcinol-formaldehyde resin was found to occur in two steps. The first step was an exothermic reaction which occurred below 85°C and resulted in resin solidification and initial strength development. The second step was found to be an endothermic dehydration reaction which occurred above 110°C.

Chow and Caster (1978) observed that the softening temperature of an adhesive provided a good indicator of bond durability. A higher softening temperature was found to be indicative of a durable bond. Of the resins included in the study on durability, phenol-formaldehyde and phenol-resorcinol-formaldehyde were found to possess the highest softening temperatures. Correspondingly, these were expected to exhibit the best bond durability properties. These findings have been supported

through the development of phenol-formaldehyde as the most desirable resin in the manufacture of exterior plywood and non-veneer structural panels.

In yet another study describing the resin curing process, Chow and Steiner (1979) used differential scanning calorimetry (DSC) thermograms to characterize the thermodynamics of resin cure of the two types of phenol-formaldehyde resin commonly used in particleboard production. A novolac resin (formaldehyde to phenol ratio less than 1.0) was characterized by an exothermic peak between 150 and 170°C. A resol resin (formaldehyde to phenol ratio greater than 1.0) was characterized by an endothermic peak between 135 and 160°C. Up to approximately 135°C, both types of resin exhibited similar thermograms with the resol consistently more endothermic. At about 175°C, the resol becomes exothermic and the novolac becomes endothermic. These two peaks are hypothesized to be the points at which the resins developed their respective thermoset properties. Although the curing properties of a particular resin are most certainly important in the overall bond quality, the authors were unable to determine any significant difference manifested in internal bond or dimensional stability tests.

Ebewele, et al. (1982) also used differential scanning calorimetry, in addition to infrared adsorption and gel permeation chromatography, in proposing a two stage curing mechanism for phenolic resin. The initial step in cure was believed to be formation of ether-linked polymers by condensation between two adjacent methylol groups. The second step in the curing process was believed to follow one of two different mechanisms. The mechanism may continue by the splitting of ether

bridges to yield methylene bridges, which partially decompose to give phenolic aldehydes and methylated phenols. The alternative second step was believed to be the formation of quinone methides.

### Resin Efficiency

Burrows (1961) defined resin efficiency as the application of the minimum amount of adhesive to wood particles with resulting optimum physical properties in a pressed particleboard. Clad (1967) found that efficient utilization of resin reduced the thickness swelling of high density (45 pcf and higher) particleboard. He also indicated that greater efficiency was promoted by greater compaction and increased interparticle surface contact.

Lehmann (1965, 1968, 1970) conducted several investigations into factors affecting resin efficiency. Increased atomization and uniformity of distribution were found to increase board modulus of rupture (MOR) 8-19% and internal bond 25-42%. Better distribution of resin was obtained with finer atomization, and resulted in stronger boards. Using photoelectric reflection photometry to estimate resin coverage, resin efficiency was found to be affected by degree of atomization, resin content, and board density. Internal bond was found to increase with increased resin content at the same application conditions. In addition, fine atomization yielded boards with the same strength properties using 19 to 28% less resin.

Duncan (1974) refuted the existing theory that 'fines rob resin'. Duncan's theory was that it is the coarser particles which actually

reduce resin efficiency. Wilson and Hill (1978) quantified this observation when they found that coarser particles receive a disproportionate amount of resin, equal to eight times that distributed on the smallest fraction of particles.

In a related investigation, Hill and Wilson (1978) made observations concerning the influence of uniformity of resin distribution on panel strength. It was stated that blending is one of the most important steps in the formation of particleboard because of the influence which it exhibits in cost and quality of the final product. The ideal situation is one in which the smallest amount of resin can be used in covering the largest area while establishing an adequate bond. The coarse fraction of particles were found to absorb 5.9 times more resin than the fine fraction of particles. The authors suggested that separating particles through screening may facilitate the application of an even amount of resin to each fraction.

### Hot-Pressing

The interactions of resin and wood with heat, moisture, and time have been described by several investigators. These interactions exhibit relatively simple relationships considering the initial complexity of the resin and wood components. The hot-pressing process is used to consolidate these two components with the application of heat and pressure over time. The interactions between the environmental factors and the two components are complicated by the hot-pressing process.

In a study on Douglas-fir flakeboard, Stickler (1959) found that the rate of heat penetration into a flakeboard mat increases with a higher initial mat moisture content, with increased surface MC, and with increased initial platen pressure. Increasing the surface MC was also found to increase the MOR, MOE, and thickness swelling characteristics of the panel and decrease internal bond. Low initial pressures were found to require longer close times and facilitate a more uniform density profile throughout the board. Low initial pressure was also found to increase the internal bond and shear values.

An investigation of the factors which influence the density distribution in flakeboard was conducted by Suchsland (1962). He concluded that the effects of temperature and moisture content on mat compressibility were responsible for the formation of a density gradient through the board thickness. Heat transfer is facilitated mainly through the movement of steam inward to the cooler regions of the board. Temperature and moisture content reduce the wood's resistance to compression and hence, densification occurs in the surface sooner and under less pressure than it occurs in the core.

Smith (1982) observed the compression characteristics of aspen waferboard. The results of this investigation indicate that the density of the aspen flakes in a 42.5 pcf board are 1.75 times the density of the unpressed flakes. Bodig (1963) found that a pressure of 1100 to 1400 psi was required to densify solid wood to a factor of 1.2 its original density in a cold press. When one compares these two independent findings, it is obvious that the application of heat has a significant effect on the plasticization of wood. The phenomenon of

wood flow is believed to be facilitated not only by heat but also by moisture. Heat and moisture provide for the plasticization of wood and therefore, greater possible densification.

Smith (1982) further described the process of pressure application during hot pressing as a failure-type mechanism. The most dense portions of the wood support the pressure until the combination of heat and pressure cause the wood to fail and flow, thereby redistributing the stress concentration. The failure is believed to be within the cell wall in the form of transwall or intrawall failures. In his investigation, Smith found that short closing times produced a characteristic U-shaped density distribution through the thickness of the board. In this case, the center or some plane very near the center of the board was found to be the least dense plane. As the closing time was serially increased, the density profile became more uniform. At very long press closing times, it was possible to produce boards which possessed an M-shaped density profile. In a board with such a profile, the interior of the board may be more dense than the surface of the board. The internal bond, bending strength, and steam dissipation are all related to the density profile. The rate of core temperature rise was found to increase with an increase in the board density and a corresponding decrease in press closing time. Contrary to expectations, the IB values increased with a decrease in press closing time. A limit was found to exist in the rate of press closing. At some point, the rate became counterproductive in that stresses built up from the inability of steam to escape.

Gelmer, et al. (1972) concluded that moisture movement during hot

pressing was inhibited by the larger particles in flakeboard. It was hypothesized that the majority of moisture movement in a flakeboard mat was around and between particles and not through them. In a similar study Denisov, et al. (1975), presented findings from an investigation on the permeability of particle mats. Their findings were in agreement with those observed by Geimer. They concluded that particle geometry significantly influenced the rate of moisture migration to the core of a mat and therefore influenced the range and shape of the density gradient.

In a study on steam injection pressing, Geimer (1982) found that a faster heatup time for the core of particleboard facilitated quicker cure of the resin. This increased rate was supported by steam injection pressing due to the excellent heat transfer properties of steam. It was also found that steam injection pressing facilitated the plasticization of the wood component, thereby, reducing the pressure necessary for board formation. This in turn produced a more uniform density profile through the board.

### Bond Quality

The ASTM internal bond test (ASTM D 1037-80) has long served as a measure of the quality of the glue bond developed in the hot-pressing process (Youngquist, et al., 1987). Since the standardization of the IB test, several alternative methods have been developed in an attempt to provide a more adequate measure of bond quality.

Lehmann (1965) proposed a push-out test as a simplified method of

determining the strength through the thickness of particleboard. The method required two circular plug cutters and a load applicator. The test was introduced to provide a test for full size panels. Accurate strength determination was based on the assumption that the center plane is the weakest plane through the thickness of the panel. This is generally considered to be a reasonable assumption since the center plane generally constitutes the lowest density plane.

Shen (1970) proposed a method of determining the internal bond or shear strength of particleboard which may well be the most rapid method developed to date. The method involved the twisting of thin plates of the panels of interest. Shen believed that since longitudinal torsion shear tests have been used to determine the shearing modulus of wood, it should also be applicable to particleboard. Plates 1/4 inch thick of several different widths and lengths were tested using this method. Each of the different sizes were correlated to values obtained in shear testing. The lowest correlation coefficient was that obtained for specimens of dimension 1 inch wide by 4 inches in length, with an r-value of 0.92. The highest correlation to shear values was obtained using specimens with a 2 inch by 6 inch dimension, with an r-value of 0.98.

Another method which has received a great deal of interest for providing a measure of the quality of the glue bond in non-veneer panels is torsional shear. Heebrink and Gatchell (1965) proposed a 1/2 inch plug torsion test for particleboard. A simple plug cutter is used to extract test specimens. It was, however, recognized that a problem arises as a result of the small size of the test specimens. The values

obtained were consistently lower than those obtained for internal bond using the same boards.

Shen and Carroll (1969, 1970) also investigated the adequacy of using torsional shear methods as a replacement for the IB test in evaluating the bond quality in particleboard. In one study, Shen and Carroll (1969) used a hand-held torsion wrench to determine torsional-shear in the center of the panel. As stated previously, one may generally assume that the center plane is the weakest plane of a panel. The specimens used for the investigation ranged in size from 1/2 inch square to 2 inches square. The smaller specimens were necessary for testing 1/4 inch thick panels due to the presence of crushing of the edges of the specimens when larger samples were tested. A correlation coefficient of 0.98 was obtained for the 2 inch square specimens and 0.94 for the 1/2 inch square specimens.

In a related study, Shen and Carroll (1970) extended the torsional-shear technique to the determination of layer strength. This study used only 1 inch square specimens for torsion testing. Again a hand-held dynamometer was used. The torsional shear strength was tested at several locations through the thickness of each panel. The depth of testing was controlled by placing spacers in the stationary end of the testing apparatus. The layer strength was found to be highly dependent on the density profile. A reasonably good correlation was found to exist between this method and IB values. The r-value for correlation to IB values was determined to be 0.89.

Gertjeansen and Haygreen (1971) proposed a similar torsion shear test for particleboard which was adaptable to a universal testing

machine. Specimens were one inch square and of variable thickness. This apparatus permitted testing of torsional shear in a predetermined location through the thickness. The correlation coefficient between the values obtained with this instrument and standard IB specimens was 0.96. This test was one of the first torsional shear tests which provided the consistency provided through use of a testing machine instead of a hand-held torsion wrench.

Passialis and Tsoumis (1982) proposed the use of circular geometry specimens in torsional shear testing. The correlation coefficient between the values obtained from this method and the values obtained in internal bond testing was found to be 0.86. An advantage over other torsional shear tests is the simplification of stress due to the elimination of corners which are areas of uncertain torsional stress distribution.

Suchsland (1977) used the principle that a maximum shear stress develops at an angle of 45 degrees to the plane of compressive force application to propose a method of bond quality through the thickness of non-veneer panels. Suchsland used disposable specimen holders fabricated from the test panel. The correlation to internal bond values was found to be 0.917. Less variability was found to exist in the values from this method of testing than in the values obtained from internal bond testing.

Hall and Haygreen (1983) introduced an apparatus known as the Minnesota Shear Tester (MST) which incorporated the principles used by Suchsland (1977). In addition, the MST provided a standardized apparatus for testing a specimen in compressive shear and the increased

accuracy which accompanies such standardization. The MST required between 15 and 30 seconds to fail a specimen. No glueup was associated with the test procedure and the coefficient of variation obtained from specimen testing was similar to that obtained from internal bond specimens. The operation of the MST resulted in shear failure at the weakest plane through the thickness of the board.

# THE EFFECT OF HOT-PRESSING PARAMETERS ON RESIN PENETRATION

## Introduction

The non-veneer structural panel (NVSP) industry in the United States has grown rapidly over the past decade (Vajda, 1983). A high rate of growth is expected to continue, with NVSP accounting for 50 percent of the structural panel industry by the year 2000 (Fuller and Berg, 1984). The adhesive component constitutes about 25 to 30 percent of the total material cost of NVSP. As a petroleum-based product, the constant fluctuation in the price of petroleum creates instability in both the cost and availability of these resins (White, 1979). The desire for more efficient utilization of materials provides incentive for investigating the influence of the various hot-pressing parameters on the resin/wood interphase.

The objective of this study was to provide quantitative and qualitative evaluations of the penetration of resin into a wood substrate as influenced by hot-pressing parameters.

## Literature Review

Our present understanding of adhesion is represented by the absence of a universal theory of adhesion (Bateup, 1981). The adhesion mechanism is probably very different in many systems and most of these mechanisms are not fully understood. The two prominent theories argued to provide for the adhesion of wood surfaces are mechanical interlocking

and adsorption. The actual mechanism is probably a combination of these theories, with the relative balance of one versus the other dependent upon many characteristics of both the wood and the adhesive (Bateup, 1981).

The formation of an adhesive bond involves several steps (Blomquist, 1981). Initially, the adhesive must wet and spread on the substrate surface to provide adequate coverage. The internal strength of the adhesive must then be established to ensure permanency of the bond. Bryant (1977) indicated that to facilitate adequate wood adhesion, the adhesive must penetrate to the depth of solidly attached fibers before cure. The penetration of aqueous resin solutions into wood is due to hydrodynamic flow and capillary action (Tarkow, 1964). The surface damage of flakes and plies has generally been observed to be restricted to a depth of one to three cells (Burrows, 1961 and Bolton, et al., 1985).

Fractured wood surfaces exhibit either intrawall or transwall failure (Wilson and Kraemer, 1976). Intrawall failure is the separation between cells in the region of the middle lamella. Transwall failure results in the lumen being exposed through fracture of the cell wall. This may occur either parallel to the long axis of the fiber, or perpendicular to this axis. The flaking and peeling of wood produce mainly intrawall and parallel transwall fractures due to the orientation of the cutting edge relative to the long axis of the fibers (Wilson and Kraemer, 1976; and Koran and Vasishth, 1972). A shallower depth of surface damage improves both the efficiency and the quality of the bond which is formed by controlling the penetration of the adhesive (Burrows,

1961; and Hare and Kutscha, 1973).

Several other processing parameters have been shown to affect the depth of resin penetration. A higher temperature enhances resin penetration by increasing the void size in wood and increasing the micromolecular motion (decreasing the viscosity) of the adhesive (Tarkow, 1966). Higher temperature is also counterproductive to penetration by facilitating cure, increasing the molecular weight, and increasing viscosity. A higher level of available moisture in the resin or the wood would also decrease the viscosity of the adhesive and thereby increase penetration. Penetration is inhibited by the higher density latewood regions in wood (Smith and Cote, 1971), and also by inactivation of the wood surface (Hare and Kutscha, 1973).

A limited number of techniques have been employed to examine the resin/wood interphase. The most widely used method appears to be scanning electron microscopy (SEM) with energy-dispersive X-ray analysis (EDAX) (Koran and Vasishth, 1972). Neutron activation analysis has also been employed (White, et al., 1977) to provide a quantitative analysis of penetration. This technique is dependent on tagging the adhesive to facilitate analysis. Several recent investigations have made use of fluorescence microscopy to provide a better visual analysis of the resin/wood interphase (Furuno, et al., 1983; Gallob, et al., 1985; Youngquist, et al., 1987). Furuno, et al. (1983) provided a comparison of the various techniques used in examining resin distribution and penetration. They concluded that fluorescence microscopy yielded better results than SEM or any of the other methods which they considered.

## Experimental

Aspen (Populus tremuloides) and Douglas-fir (Pseudotsuga menziesii) logs were obtained to produce flakes for use in evaluating the penetration characteristics of a phenol-formaldehyde resin into thin wood flakes at various pressing conditions. The logs were maintained in the green condition until use in flake preparation. Several blocks of both species were subsequently cut from these logs and saturated under a vacuum. Two hundred 0.076 by 1.27 by 7.62 cm (0.030 by 0.5 by 3 in.) tangentially oriented test flakes of each species were sequentially sliced from the saturated blocks. The order of removal of the flakes was maintained to facilitate identification of earlywood and latewood in Douglas-fir. Flakes were conditioned to moisture contents of 15 and 4 percent and stored in plastic bags to maintain the established moisture contents.

A supply of phenol-formaldehyde resin was synthesized at the U.S. Forest Products Laboratory and obtained for use in this investigation. The resin specifications are listed in Table 1. The resin was shipped and maintained frozen (5°C) until use. The viscosity of the resin was determined to be 400 cps at 21°C (70°F) and remained within 5% of this value after an 8 hour exposure to the same temperature. The temperatures used for evaluating the penetration characteristics of phenol-formaldehyde (PF) resin into aspen and Douglas-fir flakes were determined on the basis of differential scanning calorimetry (DSC) and the preparation of preliminary penetration samples.

The rate of scan for the DSC thermograms was set at 10°C per minute

Table 1: Specifications for phenol-formaldehyde resin acquired from the U. S. Forest Products Laboratory.

Property	Measured Value
Total Solids	41.2%
Total Alkalinity	6.4%
Free Formaldehyde	0.2%
pH	12.1
Viscosity (25°C)	400 cps
Shelf Life	
24°C	2 wks
5°C	16 wks

based on previous DSC work by Ebeuele, et al. (1982), and Chow and Steiner (1979). The lower and upper scan limits were set at 35°C and 220°C respectively, based on these same DSC investigations of particleboard-type PF resins. The DSC thermogram obtained for this resin exhibited an exothermic peak in the region of 150 to 160°C, as expected based on previous work (Chow and Steiner, 1979). The higher temperature for the resin penetration investigation was set at 155°C to provide a level which was highly favorable to cure. The lower temperature for the investigation was set at a level which exhibited appreciable cure, as demonstrated by the DSC thermogram, and a sufficient temperature for establishing bonding between flakes, based on the preliminary penetration samples. The lower temperature level for the resin penetration investigation was set at 105°C to provide a sufficient, yet significantly different, curing rate from that at the 155°C level.

A technique was developed to ensure the application of a drop of resin of consistent weight using the head of a pin. The coefficient of variation for this technique was approximately 15 percent. A template was used to apply a drop of resin at three locations on the surface of a single flake. The droplets were placed at positions along the length 1.27 cm (0.5 inches) from each end and at the center of the length of the flake. All droplets were applied at the center of the width of the flake. The flake was immediately covered with a second flake of corresponding moisture content and species, and pressed according to the experimental design given in Figure 1. The assembly time was maintained at a consistent period of two to three minutes for all specimens.

Species	Growth Ring Position	Moisture Content (%)	Platen Temp (°C, °F)	Pressing Time (min.)	Platen Pressure (MPa (psi))	
					2.07 (300)	6.89 (1000)
Aspen	assumed homogeneous	4	105 (221)	2	10 assemblies	-----
				5	10 assemblies	-----
			155 (311)	2	10 assemblies	-----
				5	10 assemblies	10 assemblies
		15	155 (311)	5	10 assemblies	10 assemblies
Douglas fir	Spring wood	4	155 (311)	5	10 assemblies	-----
	Summer wood	4	155 (311)	5	10 assemblies	-----

Figure 1: Experimental design for the evaluation of resin penetration into aspen and Douglas-fir as influenced by pressing parameters.

Two resin/flake assemblies were formed simultaneously to provide mechanical balance across the platens during pressing. The electrically heated elements from a 6 inch laboratory press (Carver) were attached to a load and displacement testing machine (Tinius Olsen) using a pivoting support for uniform pressure application. This apparatus provided accurate heat and pressure control. All of the resin/flake assemblies were promptly removed from the heating elements at press opening, permitted to cool, and placed in polypropylene bags until microscopic examination.

The resin/flake assemblies were saturated under a vacuum for a period of 24 hours to facilitate subsequent sectioning. A sliding microtome was used to prepare 0.01 cm (0.004 in) slide sections from each of the resin/flake assemblies at the center of each cured resin droplet. The locations of the cured droplets were easily identified by exposure to transmitted light. All of the Douglas-fir resin/flake assemblies separated during the saturation process and proved difficult to microtome. It was necessary to examine the smooth cross sections of the Douglas-fir flakes rather than thin microscope slide sections.

All resin/flake assembly sections were dried to laboratory conditions after microtoming and then stained using a 0.2% acridine red fluorochrome dye solution. The aspen sections were stained using a three stage slide preparation procedure. The Douglas-fir sections were stained by soaking in dye solution for one hour. Specimens were subsequently rinsed in distilled water to eliminate viewing interference which results from the surface deposition of excess dye.

The slide sections were examined using incident fluorescence

microscopy. The ultra-violet light source was a mercury bulb which was filtered to produce a characteristic wavelength of 410 nanometers. The Douglas-fir specimens required a 510 nanometer wavelength for optimum resolution. Fluorescent micrographs were produced using a camera which was mounted on top of the microscope. The camera shutter was controlled by a preset electronic timer. Kodak tungsten-enhanced, ASA 160 film was used to produce color slides of the resin/flake assembly cross sections.

All of the fluorescent micrographs were analyzed using a manual digitization technique in which the color slides were projected onto graph paper with a square grid size of 0.13 cm. A grid count was made to measure of the area occupied by the immediate interphase at 24 consecutive segments along an 800 micron length of interphase. This procedure was completed for 10 replicates for each combination of pressing parameters to provide a population of 240 observations; the average of these observations were used for comparison. Voids and any bulk resin present at the interface between flakes or on the surface of flakes was not included in the measure of penetration area. The resin penetration values recorded for the aspen and Douglas-fir assemblies represented a double and a single resin/wood interphase, respectively.

## Results and Discussion

### Penetration Variability

Examination of the frequency distributions for the aspen and Douglas-fir populations verified the assumption of normality necessary

to justify the use of normal population statistics. Table 2 includes the coefficients of variation (C.V.) for the various combinations of pressing parameters used in consolidation of the aspen resin/flake assemblies. These values indicate a similarity for the percent variability about the mean for all of the aspen parameter combination populations. This observation is significant in that it has been hypothesized by Hare and Kutscha (1973) that the uniformity of resin penetration may well be as important as the depth of penetration in the efficient utilization of resins and adhesives. This seems a logical hypothesis since analysis of a bondline of constant thickness would intuitively provide for the most efficient mechanical performance of the bonding material.

Bartlett's test for homogeneity of variances was executed to determine that no significant differences between the variances of the seven aspen populations were evident at an alpha level of 0.01. This test statistically verified the homogeneity of variances assumption necessary to provide a statistical comparison of the population means. Though this investigation was limited to the examination of two levels of each parameter, these levels were set at extremes such that any differences that were present should have been detected.

The lack of influence exhibited by pressing parameters over the variability of resin penetration, as indicated by the homogeneity of variances, was not entirely unexpected. Much of the variability apparent in the area of resin penetration into the wood substrate was directly attributable to the natural variability of the wood. This

Table 2: Summary of the average area of penetration and coefficient of variation of resin into aspen flakes for 240 double interphase readings per set of pressing parameters.

Moisture Content (%)	Platen Temp (°C, °F)	Platen Pressure (MPa)	Pressing Time (min.)	Resin Penetration (u <sup>2</sup> )	Coefficient of Variation (%)
4	105 (221)	2.07	2	4973	41.2
			5	3680	46.3
	2		5027	47.3	
	155 (311)		5	4773	39.8
			5	4418	40.2
	15		155 (311)	2.07	5
6.89		5		5240	44.2

hypothesis is supported by examination of the results obtained for the variability of penetration of the resin into Douglas-fir earlywood and latewood.

Examination of the coefficients of variation for the two Douglas-fir sample populations (Table 3) indicates a disparity of nearly 12 percent. Bartlett's test was executed to statistically compare the two population variances and indicated that the sample variances were significantly different at an alpha level of 0.01. Significant differences in density, cell wall area, and other anatomical attributes exist between the earlywood and latewood of Douglas-fir (Panshin and de Zeeuw, 1980). Since the only parameter not common to these two populations was the growth ring origin, the difference in the variances exhibited by these populations must be a function of the anatomical and property differences associated with earlywood and latewood. It is therefore prudent to hypothesize that the natural variability of wood exhibits greater influence over the evenness of resin penetration than any of the pressing parameters examined in this investigation.

#### Penetration Area

Tables 2 and 3 also include the values obtained for the area of penetration of resin into the Douglas-fir and aspen flakes. Duncan's multiple range test was utilized at an alpha level of 0.05 to compare the means of the various sample populations. The homogeneity of variances is a requirement for computing the least significant range for use in this test. The variances of the aspen sample populations met

Table 3: Summary of the average area of penetration and coefficient of variation of PF resin into Douglas-fir earlywood and latewood for 240 single Interphase readings per set of pressing parameters.

Ring Orientation	Resin Penetration ( $\mu^2$ )	Coefficient of Variation (%)
Earlywood	6529	32.9
Latewood	2333	44.5

\* All specimens were pressed under the following conditions:

155°C (311°F)

4% Moisture Content

2.07 MPa (300 psi) Platen Pressure

5 Minute Total Pressing Time

this stipulation, however, the sample variances for the Douglas-fir earlywood and latewood exhibited non-homogeneity. Kramer's (1956) adjustment to Duncan's multiple range test was used to permit calculation of the appropriate least significant range for the Douglas-fir populations.

Comparisons between the aspen and Douglas-fir sample populations were necessarily limited in application. The limitation was imposed by the examination of a single resin/wood interphase for the Douglas-fir specimens and a double resin/wood interphase for the aspen specimens. Doubling the Douglas-fir values to obtain a proportional value for comparison to the aspen values was believed to be inappropriate in providing a common basis for evaluation of the two species.

#### Growth Ring Origin

The mean area of resin penetration for the Douglas-fir earlywood and latewood were statistically different, as calculated using Duncan's least significant difference method at a 0.05 level. The average area of penetration for the earlywood was calculated to be nearly three times that obtained for the latewood based on 240 observation segments. The decreased penetration into the latewood is probably attributable to the greater density and lower permeability of the region. These findings are in agreement with those of Smith and Cote (1971). Using SEM techniques, they found that phenol-formaldehyde resin penetrated more extensively into earlywood than into latewood of Douglas-fir and southern yellow pine. White et al. (1977) likewise observed greater

penetration of resorcinol-formaldehyde into earlywood than into latewood of southern pine.

In many of the Douglas-fir latewood specimens, large quantities of resin cured between the two flakes in a particular assembly. The pressure which was used during the consolidation process was apparently insufficient to overcome the low permeability of the latewood regions which would require additional time for the penetration of a given quantity of resin. Since the cure of PF resin is time and temperature dependent (Chow and Steiner, 1979), an increased amount of resin will experience conditions which are adequate for cure before penetrating into the flake surface. This condition results in reduced strength and bonding as observed by White (1977) and in this investigation.

Figure 2 diagrammatically defines the spread and area of penetration as used in this investigation. Post-pressing examination indicated that the resin spread on the surface of the earlywood flakes to cover an area three to four times that observed on the latewood flakes. The penetration into earlywood flakes was also more extensive than into latewood flakes, yet neither established a sufficient bond to withstand the vacuum saturation process.

Though the aspen and Douglas-fir populations were not statistically compared, a brief qualitative comparison is in order. The values for area of resin penetration are larger for the single interphase observed in the Douglas-fir earlywood than for any of the double interphase observed for the aspen specimens. It is hypothesized that in the earlywood resin/wood assemblies, the combination of extensive resin spread and penetration were sufficient to create a condition of starved

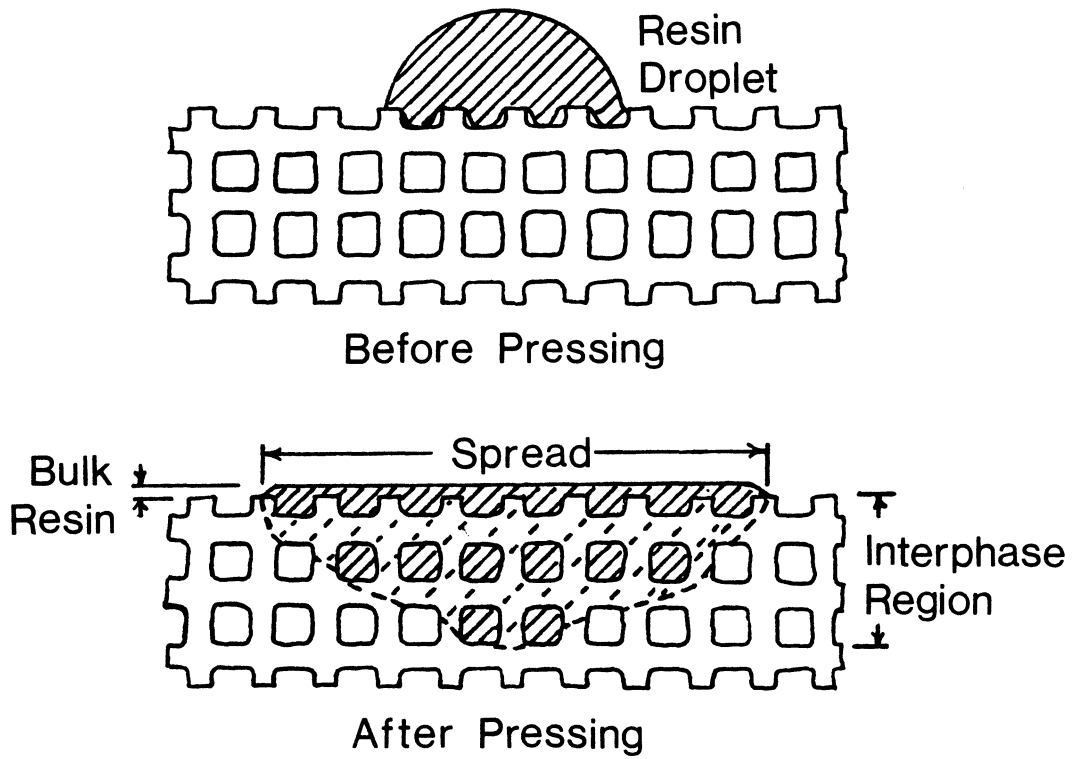


Figure 2: Illustrated definition of resin spread, and interphase or area of penetration region.

gluelines. Conversely, the reduced permeability of the latewood flakes prevented adequate penetration of the resin and permitted excessive cure on the flake surface. Both of these phenomena result in reduced bond strength and explain the separation of the specimens during the saturation process. Gallob, et al. (1985) made similar observations concerning starved gluelines and lack of penetration in Douglas-fir plies.

#### Moisture Content

The incomplete block design used to evaluate the penetration of phenol-formaldehyde resin into aspen flakes provides an analysis of the interaction of each pressing parameter with a single additional parameter. The two moisture content levels, 4 and 15 percent, were compared at pressure levels of 2.07 and 6.89 MPa (300 and 1000 psi). Both of these treatment levels were pressed at 155°F (311°F) for 5 minutes. The mean area of penetration for the specimens formed at 4 and 15 percent moisture content with 2.07 MPa (300 psi) of pressure were not significantly different as indicated by Duncan's comparison of means at the 0.05 level. The average area of penetration recorded for these moisture contents at 6.89 MPa (1000 psi) of pressure were significantly different at the same alpha level. At this pressure level, a higher initial mat moisture content resulted in greater penetration.

The influence of moisture on penetration is believed to be a result of better mobility of the resin solids during hydrodynamic flow. At both levels of moisture content (4 and 15 percent), the moisture in the

wood is present as bound water. At the higher moisture content, however, a larger number of hydroxyl sites are involved in hydrogen bonding with water molecules. The water present in the aqueous resin solution tends to remain in solution, maintaining a low viscosity, and increasing the penetration of the resin into the wood. Conversely, a smaller fraction of the available hydroxyl sites are occupied at the lower moisture content and the affinity for water molecules in the resin solution is greater. More water is removed from the solution, increasing the viscosity, and decreasing the penetration. A higher level of pressure appears to enhance hydrodynamic flow and provide a disproportionate increase in penetration at the higher moisture level as compared to the lower moisture level.

#### Applied Pressure

The values obtained at the two levels of applied pressure, 2.07 and 6.89 MPa (300 and 1000 psi) (Table 2), indicate a lack of consistency. At the 4 percent moisture content level, the sample means for the two levels of pressure application were significantly different as indicated by Duncan's least significant range test at a 0.05 alpha level. The higher average area of penetration was associated with the lower (2.07 MPa (300 psi)) applied pressure. At the 15 percent moisture content level, the mean area of penetration associated with the two pressure levels were not significantly different at the 0.05 level.

The application of pressure influences both the spread and penetration of the resin. The higher moisture level apparently

establishes a more favorable path for the transport of the adhesive into the wood based on better hydrodynamic flow characteristics. The increased pressure at high moisture content also enhances the spread rate, but to a lesser extent than the penetration rate and a net increase in penetration is observed. The low moisture level is more conducive to spreading than to penetration due to poorer hydrodynamic flow characteristics. A higher pressure level at lower moisture content enhances the rate of penetration and the rate of spread, but the net result is increased spread of the resin at the expense of penetration. One would intuitively anticipate that the level of pressure would influence the thickness of the bondline, however, there was no discernable bulk resin at the interface between flakes due to the extensive porous and capillary structure of the surface, and the small quantity of available resin.

#### Pressing Time

The influence of pressing time on the average area of penetration provided a consistent relationship at the two levels of comparison. All of the specimens were pressed at 2.07 MPa (300 psi) of applied pressure with an initial flake moisture content of 4 percent. At the 105°C (221°F) temperature level and 2 minute pressing time, the average area of penetration was higher than the average area of penetration obtained at the same pressing conditions and a 1 minute pressing time. These two means significantly different as indicated by Duncan's multiple range test at the 0.05 alpha level. At the 155°C (311°F) temperature level,

the means were not significantly different. These results indicate a relationship of decreased resin penetration with increased pressing time.

The explanation of this phenomenon is associated with the degree of cure. The DSC thermogram discussed earlier indicated that both temperatures, 105 and 155°C (221 and 311°F), were sufficient to promote accelerated cure. The longer pressing time provided longer exposure of the resin to an elevated temperature. The longer exposure interval at a given temperature, resulted in a higher degree of resin cure at press opening. The higher degree of cure provided less uncured resin for post-pressing penetration, by diffusion, into the wood and resulted in an overall reduction in the area of resin penetration. This theory was further supported by a greater difference in the penetration values for specimens pressed at the lower temperature where the cure rate is slower and a larger quantity of uncured resin was present after pressing.

#### Platen Temperature

The values obtained for the average area of penetration at the two temperature levels were indicative of the same time/temperature relationship alluded to by the results of the pressing time comparisons. When a 2 minute pressing time was used in consolidating the resin/flake assemblies, the area of penetration means for pressing at 105 and 155°C (221 and 311°F) were not significantly different at a 0.05 alpha level, using Duncan's multiple range test. At the longer pressing time (5 minutes), the mean penetration area associated with the 105 and 155°C

(221 and 311°F) temperature levels were significantly different. At the 5 minute time level, the specimens pressed at the higher temperature exhibited a higher average penetration. This is explained on the basis of the degree of cure theory and on the affect of temperature on the viscosity of resins.

The rate of decrease in the viscosity of liquids which react chemically at elevated temperatures, such as phenol-formaldehyde resin, is interrelated with the cure rate of the resin. The rate of the reaction is influenced by the additional energy available at a higher temperature. As the cure proceeds, the average molecular weight of the resin increases as it is polymerized through a condensation reaction. This in turn increases the viscosity of the resin and reduces mobility. The viscosity of the resin solution also decreases as a function of increased temperature. The relative magnitudes of the viscosity and the rate of the condensation reaction determine the amount of penetration which occurs before cure inhibits further mobility. The amount of uncured resin, available for further diffusion into the wood after pressing, is likewise influenced by this mechanism. These results indicate that the influence of the pressing parameters on the penetration of resin into the wood substrate is manifested in the effect they have on the resin viscosity.

#### Penetration Observations

The fluorescence micrographs which were analyzed using the manual digitization technique were further evaluated to obtain general

observations of the resin penetration characteristics. When the resin was initially applied, little tendency for resin spread was observed. Related to the spreading of the resin on the wood substrate is the surface condition of the flakes prior to resin application. Ideally, the depth of damage of any wood surface which is to be bonded should be minimized. A smaller quantity of resin is necessary to efficiently bond flakes which have less surface damage. The surface damage of the aspen flakes appeared to have occurred to an average depth of about one to three fibers, or a single vessel.

The earlywood Douglas-fir flakes also exhibited surface damage to a depth of about one to three cells. Again, the surface damage appeared to have occurred during the flaking process, and appeared as shear failures in a parallel transwall mode (Wilson and Kraemer, 1976). Some cell wall buckling, attributable to the pressing process, was observed in three of the earlywood specimens.

The Douglas-fir latewood flakes appeared to have been damaged to a depth of only one cell or less. The failures observed at the latewood flake surfaces appeared to be equally distributed between the parallel transwall and intrawall modes. The cell walls of the latewood flakes exhibited almost no buckling. The only other failures present in the latewood specimens was intrawall fracture, which was believed to be attributable to microtoming the specimens since no resin was present in any of these failures.

Buckling failures in the common walls of adjacent vessels were observed to be abundant throughout most of the aspen flakes. These failures along with buckling of the fiber cell walls in the vicinity of

the bondline were the result of the pressing process. The high moisture content at the bondline contributes to compressive deformation (Wellons, et al., 1982). Penetration into the Douglas-fir flake surfaces appeared to follow the fracture surfaces which were present. The latewood exhibited few failures and penetration was limited to a relatively shallow depth. Fractures were abundant along the surface of the earlywood flakes and the resin consistently followed these avenues of penetration.

The resin applied to the aspen and Douglas-fir earlywood appeared to have penetrated into the flake completely and no bulk-resin bondline was apparent at 160X magnification. Bulk-resin was observed on the surfaces of the Douglas-fir latewood flakes. The absence of bulk resin for most of the specimens is a result of the small quantity of resin applied to the flakes. The resin was generally observed to penetrate one half to one full cell beyond the level of damaged fibers in both the aspen and the Douglas-fir flakes. The previously mentioned failures in the aspen vessels were consistently filled with resin and accounted for much of the large variation in the level of penetration along any single bondline. In most instances the vessel lumens, which did exhibit cell wall failures, provided direct avenues for penetration and were filled with resin. Also evident in these observations were a limited number of lumens which were filled with resin, but appeared to have no direct connection to the immediate bondline. These cases were attributed to the angle at which the flakes were cut from the saturated block (Kitazawa, 1946), the area of these vessels were not included in the calculation of the area of resin penetration.

## Summary and Conclusions

Fluorescence microscopy was found to be an excellent technique for evaluating the interphase between resin and wood. The results obtained with this method indicate that temperature, moisture content, pressure, time, and growth ring origin exhibit an influence over the amount of penetration of aqueous PF resin into a wood substrate. The four pressing parameters yielded significantly different levels of penetration at only one of two levels of a second parameter, indicating limited interdependence of the parameters.

Higher levels of moisture increased penetration by maintaining a low viscosity and facilitating resin flow. Pressure interacted with the level of available moisture to provide a mechanism to control hydrodynamic flow. Time and temperature also interacted to influence resin penetration. The relative balance between the degree of cure, which is time-temperature dependent, and resin viscosity, which is temperature dependent, contributed to determining the extent of penetration. These findings provided the basis for two significant conclusions regarding the effect of pressing parameters on resin penetration;

1. The uniformity of resin penetration is influenced more by the natural variability of the wood than by temperature, moisture content, time, or pressure.
2. The pressing parameters of temperature, moisture content, and time influence the extent of resin penetration by controlling

the viscosity of the resin. Pressure influences penetration by providing a mechanism to control hydrodynamic flow. The interactions of moisture content with pressure, and temperature with time also influence resin penetration by affecting the viscosity, with the time-temperature interaction providing the larger influence.

The depth of flake surface damage was observed to be two to three cells, with the mode of fracture being either intrawall or parallel transwall. The resin generally followed the avenues provided by fractures in the wood surface. Extensive buckling was observed in the vicinity of the interface between the aspen flakes, but was rare in either the earlywood or latewood of Douglas-fir specimens. This deformation was attributed to the high moisture concentration provided by the resin. Though resin was observed to have cured on the surface of Douglas-fir latewood flakes, no bulk-resin bondline was evident at the interface between aspen flakes or on the surface of Douglas-fir earlywood flakes.

# THE EFFECT OF HOT-PRESSING PARAMETERS ON FLAKEBOARD LAYER PROPERTIES

## Introduction

Over the past decade, the non-veneer structural panel (NVSP) industry in the United States has experienced strong growth, which is expected to continue through the year 2000 (Vajda, 1983). Initially, aspen served as the major species utilized in NVSP. Much of the recent expansion in production has occurred at facilities located near markets far removed from the aspen growth range. Locating production facilities in proximity to these markets requires that present technology be expanded to utilize the species which are readily available in the particular region.

Further development of NVSP has led to reducing costs of production and improving product performance through the design of products and more efficient utilization of raw materials. Since these panels are stressed largely in bending in construction applications, it is desirable to model the bending properties through the use of such methods as transformed cross sections. The vertical distribution of density, strength, and stiffness is determined by complex hot-pressing interactions, and is critical in determining the overall properties of the panel. The effective design of NVSP is dependent on developing an extensive understanding of the relationship between the hot-pressing process and the resultant distribution of properties through the panel.

The objective of this study is to provide a quantitative evaluation of the affects of hot-pressing parameters on the layer properties at

various planes through the thickness of flakeboard panels.

### Literature Review

The hot-pressing process is used to consolidate resin and wood with the application of heat and pressure over time. The environment particular to a location through the panel has been shown to be influenced by a number of factors (Casey, 1987). The rate of heat transfer into the mat, for instance, is influenced by mat moisture content (Casey, 1987), initial platen pressure or press closing time (Stickler, 1959), flake geometry (Geimer, 1982), and panel density (Smith, 1982). The moisture and pressure environment at a particular location are similarly a result of complex interactions which are not easily modeled (Casey, 1987; Harless, et al., 1987).

The environment which is created by the mutual interaction of pressing parameters affects the densification of wood (Smith, 1982) and the cure of resin (Geimer, 1982), and serves to create a density gradient through the panel thickness (Suchsland, 1962). High initial pressure (fast press closing) has been shown to facilitate formation of a characteristic U-shaped density gradient (Smith, 1982), while low initial pressure produces a more uniform density gradient through the thickness (Stickler, 1959). Additionally, rapid heat transfer to the core has been found to provide more uniform densification of the flakes, and hence a more uniform density profile (Geimer, 1982).

The mechanical properties of panels as well as the quality of the glue bond have been related to the shape of the density profile (Smith,

1982). Traditionally, the density gradient has been determined using gravimetric techniques, which are prone to inaccuracies. The development of low energy radiation methods (Laufenberg, 1986; Winistorfer, et al., 1987) has facilitated the accurate determination of density gradients and provided data which is essential in analyzing the factors which influence the mechanical properties of the board and the quality of the adhesive bond.

The ASTM internal bond test (ASTM D 1037-80) has long served as the standard method to measure the quality of glue bond in non-veneer composite wood panels. Torsional shear techniques were developed (Heebrink and Gatchell, 1965; and Shen and Carroll, 1969) to analyze bond quality while providing simplified specimen preparation relative to, and high correlation with, internal bond testing. Additionally, compressive shear methods were developed (Suchsland, 1977; and Hall and Haygreen, 1983) to provide a similar evaluation, but a simplified stress analysis relative to torsional shear. A more comprehensive evaluation of bond quality was facilitated by improving techniques to include the determination of adhesive performance at predetermined locations through the thickness of a panel (Shen and Carroll, 1970; Gertjejansen and Haygreen, 1971).

## Experimental

Yellow poplar (Liriodendron tulipifera) logs were obtained to produce flakes for the manufacture of twenty-four flakeboard panels. The logs were processed on a laboratory disk flaker (CAE) to a target

flake thickness of 0.08 cm (0.03 in.) and a target length of 7.6 cm (3 in.). The flakes were dried in two groups to 2 and 11 percent moisture content, screened to a width of 0.6 to 2.5 cm (0.25 to 1 in.), and stored in polypropylene bags to maintain the established moisture content conditions until use in board manufacture.

Twenty-four 51 by 56 by 1.9 cm (20 by 22 by 0.75 in.) panels were manufactured using eight different press schedules, with three replications (Figure 3). A five percent resin solids content (based on oven-dry weight of the wood) was used in all panels. Resin was applied in a drum blender rotating at 16.5 rpm. The blender had a single spray head which applied resin at a rate of 14.4 ml/min. under 207 kPa (30 psi) of air pressure.

Two levels each of press temperature (154 and 190°C (310 and 375°F)), press closing time (1 and 2 minutes), and initial mat moisture content (6 and 15 percent) were used. A 30 second prepress, 12 minute total press time, and six minute venting period were used throughout. A target density of 721 kg/m<sup>3</sup> (45 lb/ft<sup>3</sup>) was used in the production of all panels. The mats were hand-formed with random flake orientation. The press was computer-controlled on position in an effort to obtain repeatable press cycles.

During the pressing process, data was acquired for platen pressure, platen position, press time, and gas pressure and temperature at both the face and core layers of the mat according to the methods described by Casey (1987). A linear variable displacement transducer (LVDT) mounted directly onto the hot-press provided position readings. Platen pressure was determined by a pressure transducer incorporated into the

Initial Mat Moisture Content (%)	Press Closing Time (min.)	Platen Temperature	
		154°C (310°F)	190°C (375°F)
6	1	Panels 1,2,3	Panels 13,14,15
	2	Panels 4,5,6	Panels 16,17,18
15	1	Panels 10,11,12	Panels 22,23,24
	2	Panels 7,8,9	Panels 19,20,21

Figure 3: Experimental design for the manufacture and testing of yellow poplar flakeboard panels.

press' hydraulic system. Mat temperature was monitored by Type-K thermocouples. A gas pressure probe was designed by Casey (1987) using a pressure transducer mounted on the outside of the mat and attached to a stainless steel tube (0.16 cm OD, 0.025 cm ID) filled with silicon oil. The gas pressure probe and thermocouple were located as near the center of the mat as possible, in both the face and core layers. The face and core layers were defined as  $1/5$  and  $1/2$  the thickness of the mat from the surface, respectively. An automatic data acquisition system (Hewlett Packard model 3054A) was used to collect data every 5 seconds during the 12 minute press cycle.

Eighteen layer-shear specimens, 2.5 by 10 cm (1 by 4 in.) and six internal bond specimens, 5.1 by 5.1 cm (2 by 2 in.) were cut from each panel according to the diagram in Figure 4. The six internal bond specimens were tested according to ASTM standard D 1037-80. The specific gravity profiles of six of the layer-shear specimens from each panel were non-destructively determined by the method of gamma radiation attenuation (Laufenberg, 1986). A device was designed and manufactured to test specimens in compressive shear at any predetermined plane through the thickness of the panel (Figure 5). Six specimens from each panel were sheared at the center of the specimen, to obtain average stress at shear failure. This procedure was repeated with the same number of specimens at locations approximately  $1/6$  and  $1/3$  the thickness of the panel from the surface. The speed of testing was maintained at 0.06 inches per minute for all layer-shear specimens.

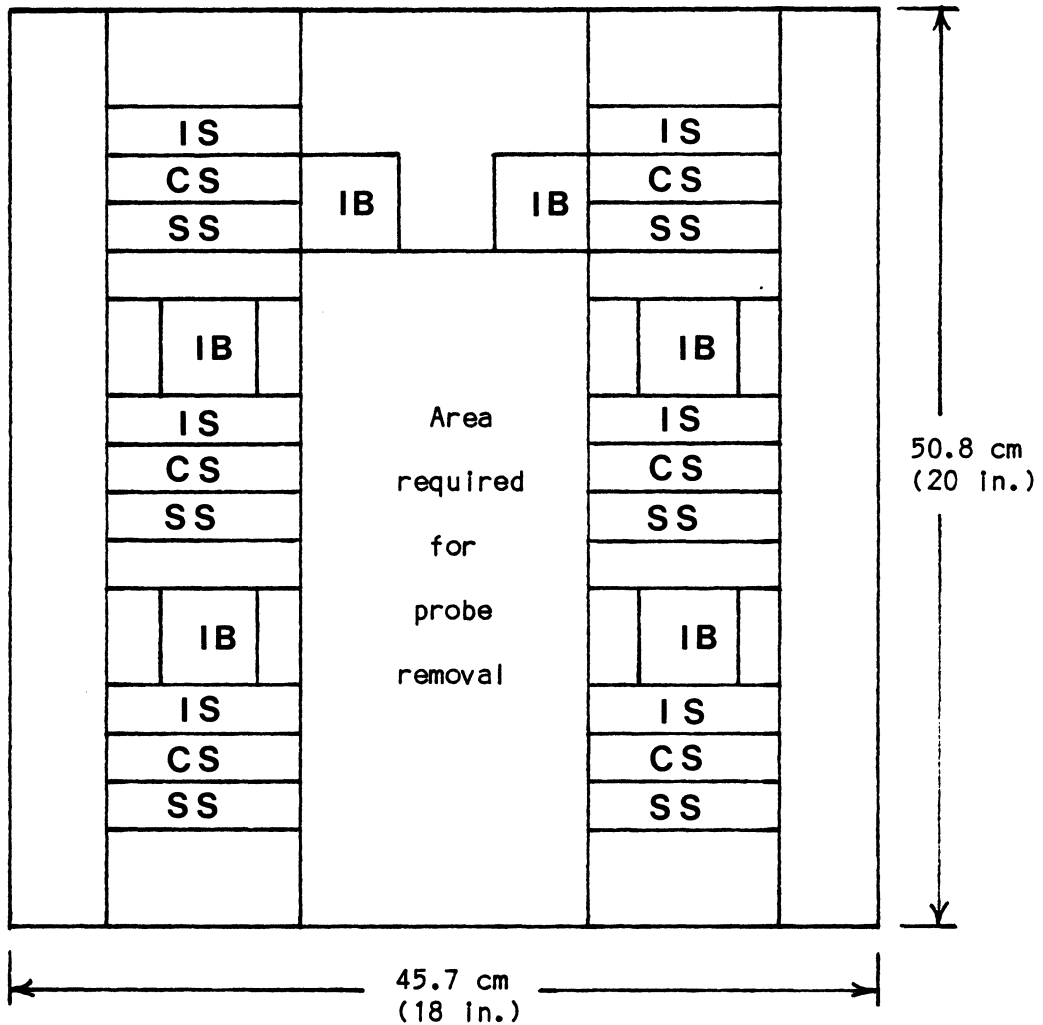


Figure 4. Cut-up pattern for obtaining layer-shear and internal bond specimens from yellow poplar flakeboard panels.

- IB - 5.1 x 5.1 cm (2 x 2 in.) internal bond specimen
- CS - 2.5 x 10 cm (1 x 4 in.) core layer-shear specimen  
first analyzed for specific gravity profile.
- IS - 2.5 x 10 cm (1 x 4 in.) intermediate layer-shear specimen
- SS - 2.5 x 10 cm (1 x 4 in.) face layer-shear specimen

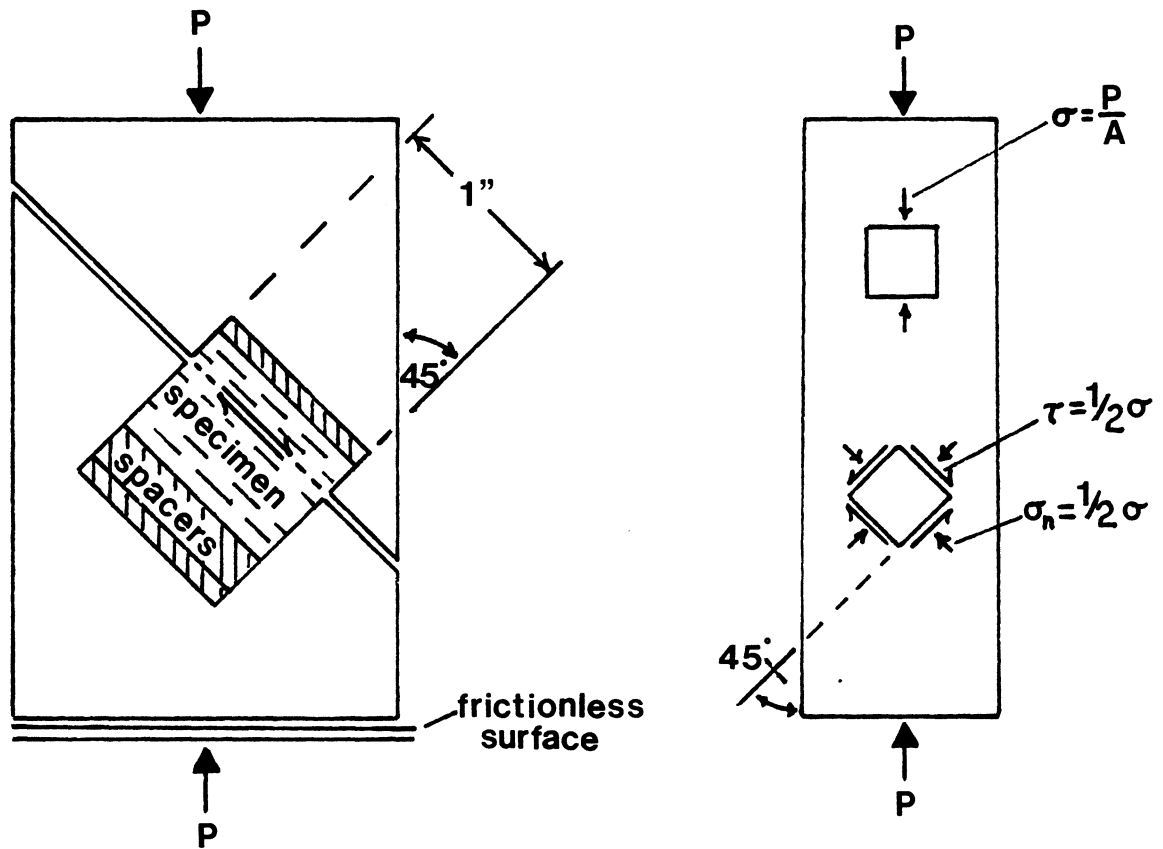


Figure 5: Device for the testing of layer-shear at predetermined locations through the thickness of flakeboard panels.

## Results and Discussion

### Data from Panel Manufacture

To provide a quantitative analysis of the environmental history of a particular location through the panel thickness, the time-integral of temperature and gas pressure were calculated from the data collected for each panel. Figure 6 indicates a typical curve used in calculating the time-integral of temperature and gas pressure. The time-integral of platen pressure was also calculated from the available data. This brief analysis of the time-integrals of platen pressure, temperature, and gas pressure is intended only to provide a synopsis of the data collected during the pressing of the 24 yellow poplar flakeboard panels. An excellent treatise of the data is presented by Casey (1987).

Temperature and gas pressure probes were placed at two different layers through the mat thickness during forming. This was achieved on the basis of weight of material. The resulting location of the probes, relative to the core of the panel, were obtained by dissecting the panels after pressing. A table-top thickness gauge was employed to provide accurate determination of the distance of the probes from the surface of the panel. These values were then used to calculate the location relative to the core, based on the half thickness of the panel. Because of the variability inherent to the mat forming process, the face and core probe locations varied from panel to panel. Since the layer-shear test could be tightly controlled, the layer-shear failure locations were selected for comparison between shear values and the

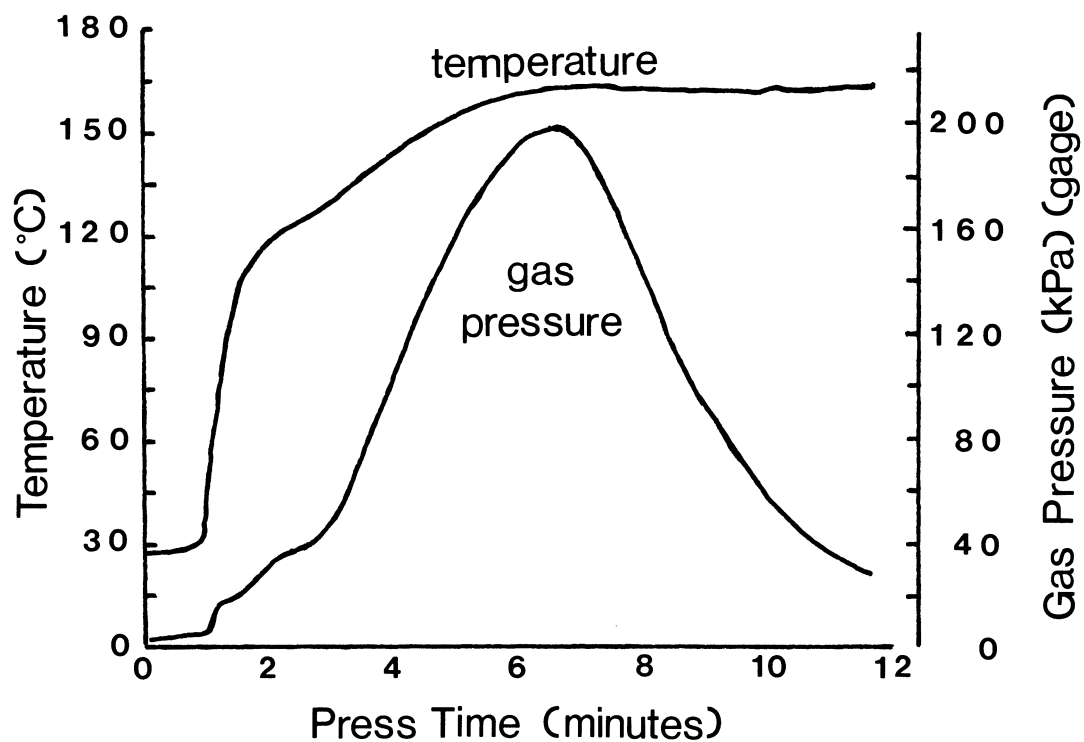


Figure 6: Typical curves used in the calculation of the time-integrals of temperature and gas pressure.

time-integral data. This procedure required interpolations of the time-integral data for the desired locations.

Since the difference between the face and core time-integrals of gas pressure were small, a linear relationship was assumed between this value and the distance through the panel. Several other relationships were explored, but none yielded results significantly different from linearity. Since the difference between face and core time-integrals of temperature were quite large for any particular panel, an assumption of linearity did not suffice. An additional point was provided by assuming that the surface of the mat experienced the platen temperature for the entire pressing cycle. The influence of the stainless steel caul plates, and surface effects between the caul plates and the mat were ignored in this assumption. The representation of the time-integral of temperature as a function of the square of the position from the core provided an adequate relationship for interpolating the time-integral of temperature for the desired locations.

A significant fluctuation in the total pressing time was exhibited among the 24 panels. This inconsistency was due to the algorithm used to control the press cycle. In order to provide a comparison of the time-integral values for various treatments, a common total pressing time was necessary. The shortest total pressing time experienced by any of the panels was 11.4 minutes, therefore, all of the values used for comparison of the panels were standardized to include only the events occurring in the first 11.4 minutes of the pressing cycle. The evaluation of shear data required the use of the time-integral values obtained over the entire pressing cycle. This provided a relationship

between the layer-shear and the true environmental history of a particular layer through the thickness of the panel.

Although the press was controlled based on position, all of the 6 percent mat moisture content panels reached the maximum available platen pressure before they reached final thickness. As a result, the press closing times for these panels were longer than the target values. However, the platen pressure time-integral values are probably not significantly effected because, with the exception of a slight rate of loading effect, the same amount of work would be required to close the press.

Examination of Table 4 indicates that the time-integral of pressure is higher for panels pressed at the slower press closing rate. Initially, the faster rate of loading requires a higher applied pressure, since the rate of loading exhibits a significant influence on the measured strength of wood. The slower rate of loading requires a lower maximum applied pressure, but the stress relaxation following press closure occurs at a slower rate. This results in a higher net time-integral of applied pressure for the longer press closing time. This observation was verified using Duncan's test for the least significant difference between means. At the 0.05 alpha level, the mean time-integral of pressure obtained for the two minute press closing time was significantly higher than the mean time-integral of pressure obtained for the one minute press closing time.

The presence of moisture decreases the resistance of wood to transverse compression, and hence, higher moisture content mats require less pressure to obtain the same level of compaction as a lower moisture

Table 4: Summary of average time-integrals of platen pressure for the initial 11.4 minutes of the press cycle, for the eight treatment levels of panel manufacture.

Platen Temperature (°C (°F))	Initial Mat Moisture Content (%)	Press Closing Time (min.)	Platen Pressure Time-Integral (kPa x min.)
154 (310)	6	1	19659
		2	19235
	15	1	11969
		2	14270
190 (375)	6	1	13522
		2	14668
	15	1	13003
		2	14823

content mat. Duncan's test confirmed this observation since the means for the two moisture content levels were found to be significantly different at the 0.05 level. The higher initial mat moisture content resulted in lower time-integral of platen pressure values.

The relation of temperature to the time-integral of platen pressure is not evident from visual examination of the average time-integrals of pressure (Table 4). One would expect that a higher temperature would provide for greater plasticization of the lignin and hemicelluloses. This condition would require less pressure to obtain the same level of compaction. The average values obtained for the time-integral of pressure appear to follow this hypothesis at low moisture content, but contradict it at the higher moisture content.

At the combination of higher moisture content and higher temperature, a sufficiently high gas pressure is created to offset the increased pliability of the wood component. Indeed, reference to Table 5 indicates a significant increase in the gas pressure encountered at the combination of high temperature and high moisture content. Duncan's test was executed to check for significant differences between the sample population means of the two temperature levels. The two means were found to be significantly different at an alpha level of 0.05. The higher temperature resulted in a reduced time-integral of platen pressure. The interaction between temperature and moisture content was also significant at the 0.05 level.

Further reference to Table 5 indicates the trends exhibited in the time-integral of gas pressure as influenced by the various pressing parameters. As expected, a higher initial mat moisture content produced

Table 5: Summary of average time-integrals of gas pressure for the initial 11.4 minutes of the press cycle, for the eight treatment levels of panel manufacture.

Platen Temperature (°C (°F))	Initial Mat Moisture Content (%)	Press Closing Time (min.)	Gas Pressure Time-Integral Face (kPa x min. (gage))	Gas Pressure Time-Integral Core (kPa x min. (gage))
154 (310)	6	1	109	84
		2	104	104
	15	1	433	419
		2	355	286
190 (375)	6	1	239	229
		2	277	240
	15	1	1014	942
		2	905	736

higher time-integral of gas pressure values, apparent at both the surface and the core. A greater platen temperature has the potential for a greater water vapor pressure, and consequently a higher total gas pressure. The affect of moisture content, temperature, and their interaction were all found to be statistically significant at the 0.05 level. Press closing rate did not appear to provide a significant influence over the time-integral of gas pressure.

Table 6 contains the time-integral of temperature for the eight treatment levels used in pressing. Platen temperature, as expected, exhibited a significant influence over the total temperature experienced at both the face and the core. This relationship was statistically significant at the 0.05 alpha level. In each case, the time-integral of temperature increased at a higher platen temperature.

The addition of moisture to the flakeboard furnish was expected to increase the rate of heat transfer from the platens to the core of the panel. In addition, a higher moisture content would require more energy for vaporization at the expense of latent heat. Consequently, the panels which were pressed at 15 percent moisture content exhibited no consistent difference in the time-integral of temperature from those which were pressed at 6 percent moisture content, as indicated by Duncan's test at a 0.05 alpha level.

The press closing time was observed to provide a statistically significant increase in the time-integral of temperature with an increase in the rate of closing. The moisture content interacted with the press closing time to result in a higher time-integral of temperature at the higher level of initial mat moisture content. By

Table 6: Summary of average time-integrals of temperature for the initial 11.4 minutes of the press cycle, for the eight treatment levels of panel manufacture.

Platen Temperature (°C (°F))	Initial Mat Moisture Content (%)	Press Closing Time (min.)	Temperature Face (°C x min.)	Time-Integral Core
154 (310)	6	1	1447	994
		2	1370	939
	15	1	1426	976
		2	1343	860
190 (375)	6	1	1725	1105
		2	1634	1057
	15	1	1724	1125
		2	1624	970

closing the press more quickly, a longer period is provided in which more intimate contact is established between flakes. Because the compaction capacity of the press was reached with the low mat moisture content flakes, press closing time and mat moisture content were not independent variables. The actual press closing time was longer for the low mat moisture content, therefore, a significant interaction was expected.

#### Internal Bond

Six specimens from each of the 24 flakeboard panels were tested for internal bond according to ASTM D 1037-80. These values were used to provide a comparison of weakest plane adhesive bond quality between the eight treatment levels. All internal bond values, along with the panel average and coefficient of variation are listed in Tables B5 and B6, in Appendix B. The average and coefficient of variation of the eighteen specimens for each of the treatment levels are listed in Table 7.

The internal bond values were analyzed as a complete factorial design, with two levels each of platen temperature (154 and 190°C (310 and 375°F)), press closing time (1 and 2 minutes), and initial mat moisture content (6 and 15 percent). Analysis of variance was performed to detect significant treatment differences at an alpha level of 0.05 for each of the pressing parameters. All two-way interactions and the single three-way interaction of these variables were also examined.

At the 0.05 alpha level, the two levels of temperature, initial mat moisture content, and press closing time each provided significantly

Table 7: Summary of average internal bond values and coefficients of variation for the eighteen observations, for each of the eight treatment levels of panel manufacture.

Platen Temperature (°C (°F))	Initial Mat Moisture Content (%)	Press Closing Time (min.)	Internal Bond (kPa) (psi)		Coefficient of Variation (%)
154 (310)	6	1	384	56	31.6
		2	452	67	20.7
	15	1	416	60	25.2
		2	330	48	32.2
190 (375)	6	1	646	94	27.2
		2	628	91	17.7
	15	1	425	62	22.2
		2	278	40	45.3

different internal bond values. The two-way interactions of temperature with initial mat moisture content and press closing time with initial mat moisture content were also found to provide significantly different internal bond values. The two-way interaction of temperature with press closing time and the three-way interaction of all of the parameters were not significant at the 0.05 alpha level.

The higher temperature level (190°C, 375°F) provided an overall increase in the magnitude of IB values, with the exception of the 15 percent moisture content, 2 minute press closing time treatment. Likewise, all of the treatments at the faster press closing time (1 minute) exhibited higher IB values than the treatments at the slower press closing time (2 minutes). Again, a single exception is apparent. The sample population mean for the 154°C (310°F), 6 percent moisture content, 2 minute press closing time treatment is higher than the sample population mean for the corresponding treatment with a 1 minute press closing time. The treatment means obtained for the two moisture content levels (6 and 15 percent) exhibit a similar single disparity from the general trend. The overall affect of the lower initial mat moisture content level was to increase the magnitude of the values obtained in internal bond testing. The single exception is that the sample population mean for the 154°C, 1 minute press closing time, 6 percent moisture content treatment is lower than the corresponding treatment mean with a 15 percent initial mat moisture content.

The significant interaction indicated for the parameters of temperature and moisture content is manifested in the increased difference observed between treatment means at the two initial mat

moisture content levels. The overall increase of internal bond values at the lower moisture content is more dramatic at the higher temperature. A higher moisture content imparts greater mobility to the resin component by retarding the sorption of moisture from the resin and, thereby, maintaining a lower resin viscosity. This condition provides less resin at the interface between two flakes, and creates a condition equivalent to starved gluelines observed in plywood.

Increasing the temperature at a given moisture content level creates additional steam which moves toward the center of the mat, which is at a lower temperature than the face. High humidity conditions or even condensation may lead to an even higher moisture level at the center of the mat than provided by the initial mat moisture content. This serves to further increase the mobility of the resin component. Moisture inhibits the cure of phenol-formaldehyde resin because the cure occurs through a condensation reaction. The higher moisture level shifts the equilibrium to favor the reactants rather than the cured resin product. Although an elevated temperature tends to increase the rate of cure, a higher moisture content inhibits the cure of the resin and provides a longer time for resin flow. At lower moisture levels, the affect on resin cure is reduced and less of the resin migrates away from the interface between flakes before the resin gels and migration is halted.

The significance of the interaction observed between initial mat moisture content and press closing time is less clear. The rate at which the press is closed is directly correlated to the pressure required to do so. The closing rate or time of closing can affect the resin in two aspects. Closing the press more rapidly will require a

greater compaction pressure and consequently increase the penetration or spread of the resin, or both. It is hypothesized that at a higher initial mat moisture content, a faster press closing rate provides a net increase in penetration of the resin which better anchors torn or damaged fibers and increases internal bond. At the lower moisture content, increased pressure results in a net increased spread of the resin and corresponding reduced penetration which is insufficient to adequately reinforce damaged fibers resulting in decreased internal bond.

#### Specific Gravity Profiles

Six shear specimens from each of the 24 panels were analyzed to determine the specific gravity profile through the thickness of the panel. The method of attenuation of gamma radiation developed by Laufenberg (1986) was used in obtaining the profiles. The analysis used in this investigation was based on the assumption that the flakeboard panels were symmetric about the dimensionally central plane through the thickness of the board. Visual examination of the individual profiles provided sufficient verification of this assumption of symmetry. The specific gravity profiles were therefore analyzed from the center of the panel outward to one surface, chosen randomly. Averaging the eighteen profiles for each of the treatment levels served to eliminate much of the localized variability observed in any particular profile. This provided a smooth curve for comparing the affects of the various levels of pressing parameters on the specific gravity profile.

Laufenberg (1986) indicates that a limitation is associated with this particular method. The ability of the technique to provide accurate determination of the specific gravity of the panel near either surface is questioned. The inaccuracy is limited, however, to a region of approximately 0.05 cm (0.02 in.) at each surface which is the width of the radiation emission slot on the testing apparatus. These thin surface regions are not of particular interest for relating to the shear values, since they are not obtainable at such shallow depths from the panel surface.

Smith (1982), and Plath and Schnitzler (1974) have indicated the significance of the press closing time in controlling the density profile in non-veneer composite wood panels. These investigations also indicated strong correlations between the density profile and mechanical properties of such panels. Figure 7 illustrates the specific gravity profiles obtained for the treatment levels of 190°C (375°F), 15 percent initial mat moisture content, and one and two minute press closing times. This graph demonstrates the influence that press closing time typically exhibited on the shape of these curves. The panel pressed with a longer press closing time exhibited a peak in specific gravity at some distance from the surface and the specific gravity decreased from that point. The specific gravity profile obtained for the panel pressed at the one minute closing time exhibits a continuous increase in specific gravity to the surface of the panel, a U-shaped profile.

Smith (1982) indicated similar findings in his investigation of aspen waferboard. The influence of the press closing time is manifested in controlling the location and amount of wood densification. At faster

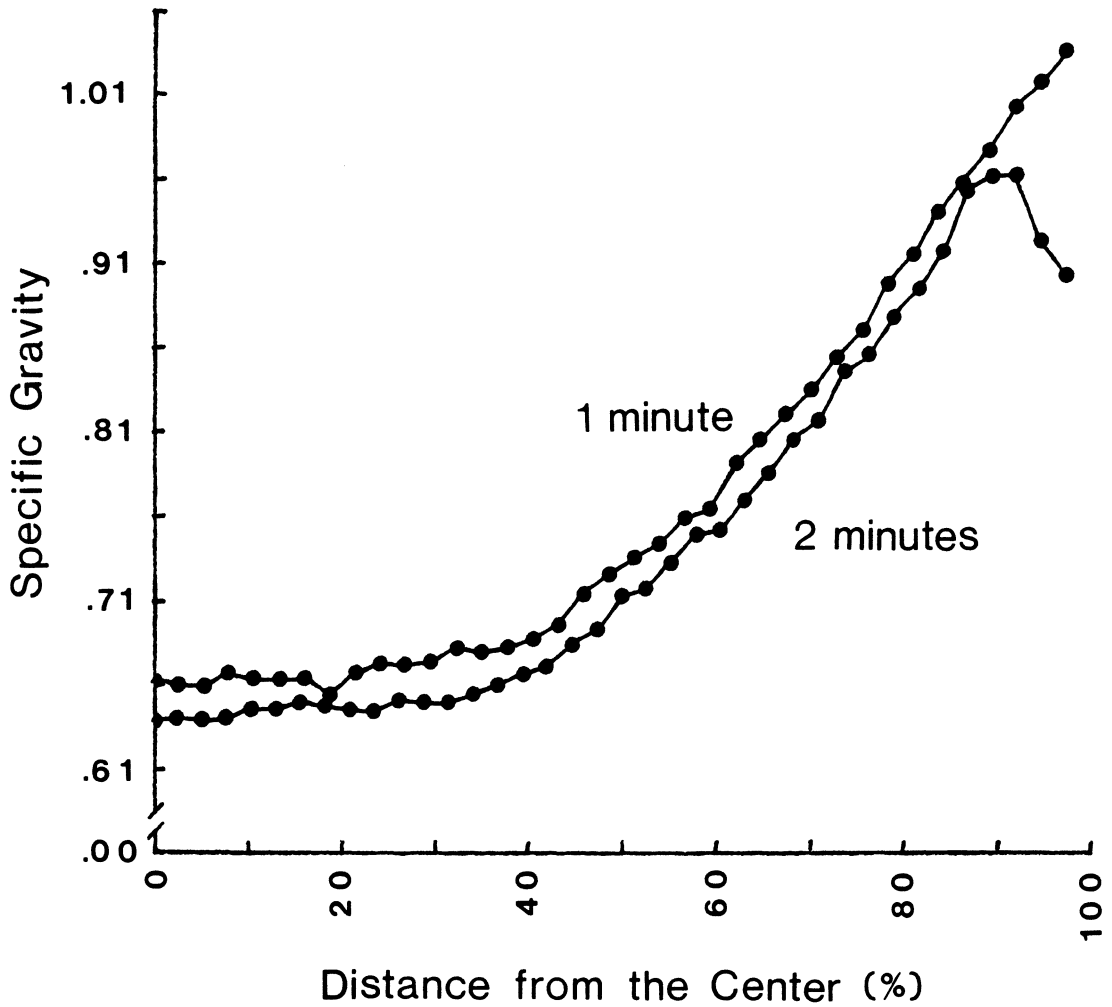


Figure 7: Comparison of average specific gravity profiles for panels pressed at 190°C (375°F) platen temperature, 15% initial mat moisture content, and 1 and 2 minute press closing times.

press closing times, the flakes at the surface are exposed to compressive forces before the moisture in the wood is heated to evaporation and forced toward the center of the mat. The higher moisture content provides for greater plasticization of the lignin and hemicellulose components. This results in a decreased resistance of the wood to compressive deformation and hence, greater densification of the surface regions.

The influence that the moisture and heat components exhibit on the the specific gravity profile of a panel are essential, yet somewhat less clear due to their mutual interaction and interaction with press closing time (Smith, 1982). The effect of moisture appears to be to increase the specific gravity through the entire panel. This is illustrated by the profiles for the treatments of 190°C (375°F), 2 minute press closing, and 6 and 15 percent initial mat moisture contents (Figure 8). An upward shift of the entire profile at higher initial mat moisture content is a result of less springback, and consequently a smaller panel thickness.

Figure 9 indicates the influence of the temperature parameter on the specific gravity profile of flakeboard. This graph includes the profiles for the treatments pressed at 15 percent moisture content, with a 1 minute press closing time, and at temperatures of 154 and 190°C (310 and 375°F). An upward shift of the entire specific gravity profile for the higher press temperature is clearly apparent. The overall increase in the specific gravity profile for panels pressed at the higher temperature are a direct function of the reduced final thickness of the panel. Since the target thickness of 1.9 cm (0.75 in.) was achieved for

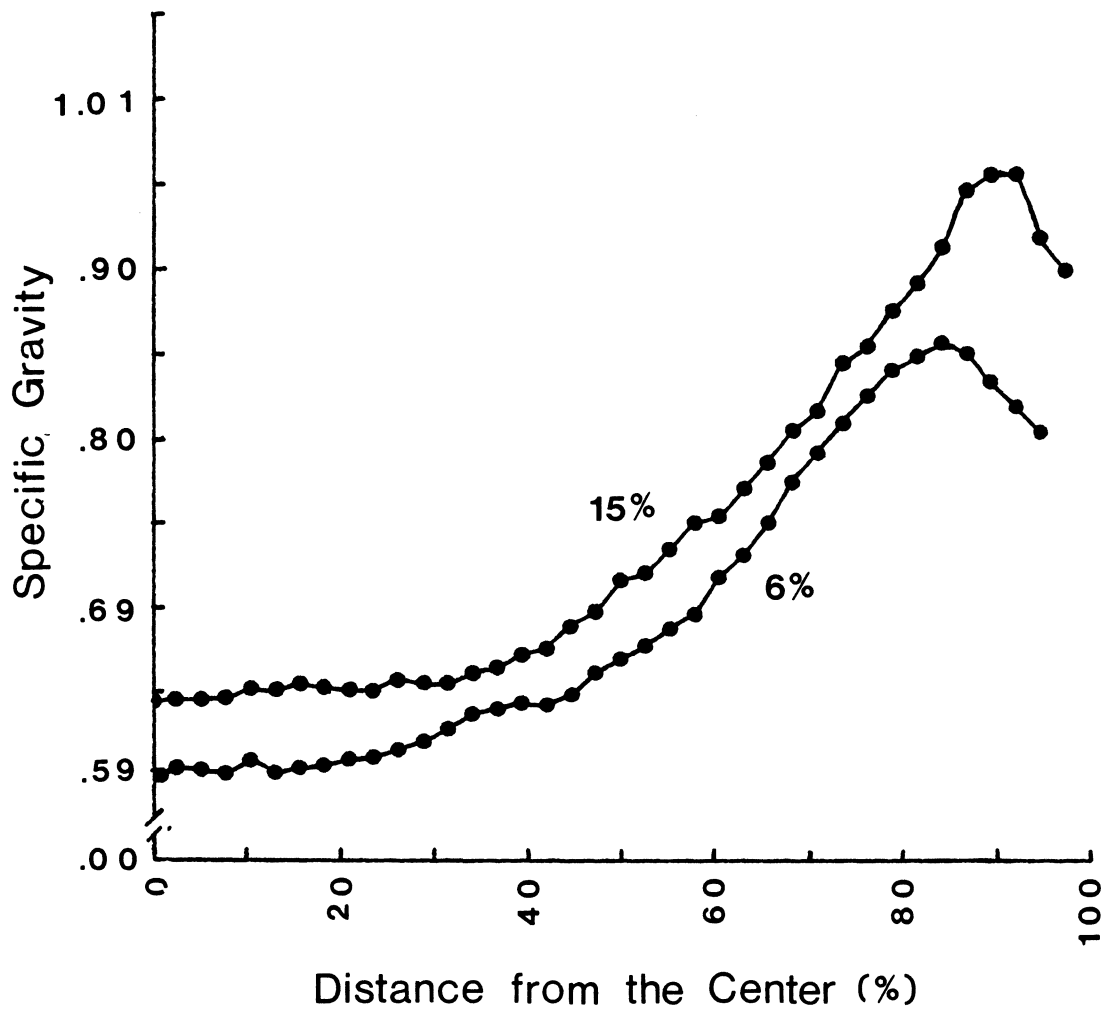


Figure 8: Comparison of average specific gravity profiles for panels pressed at 190°C platen temperature, 2 minutes press closing time, and 6 and 15 percent moisture contents.

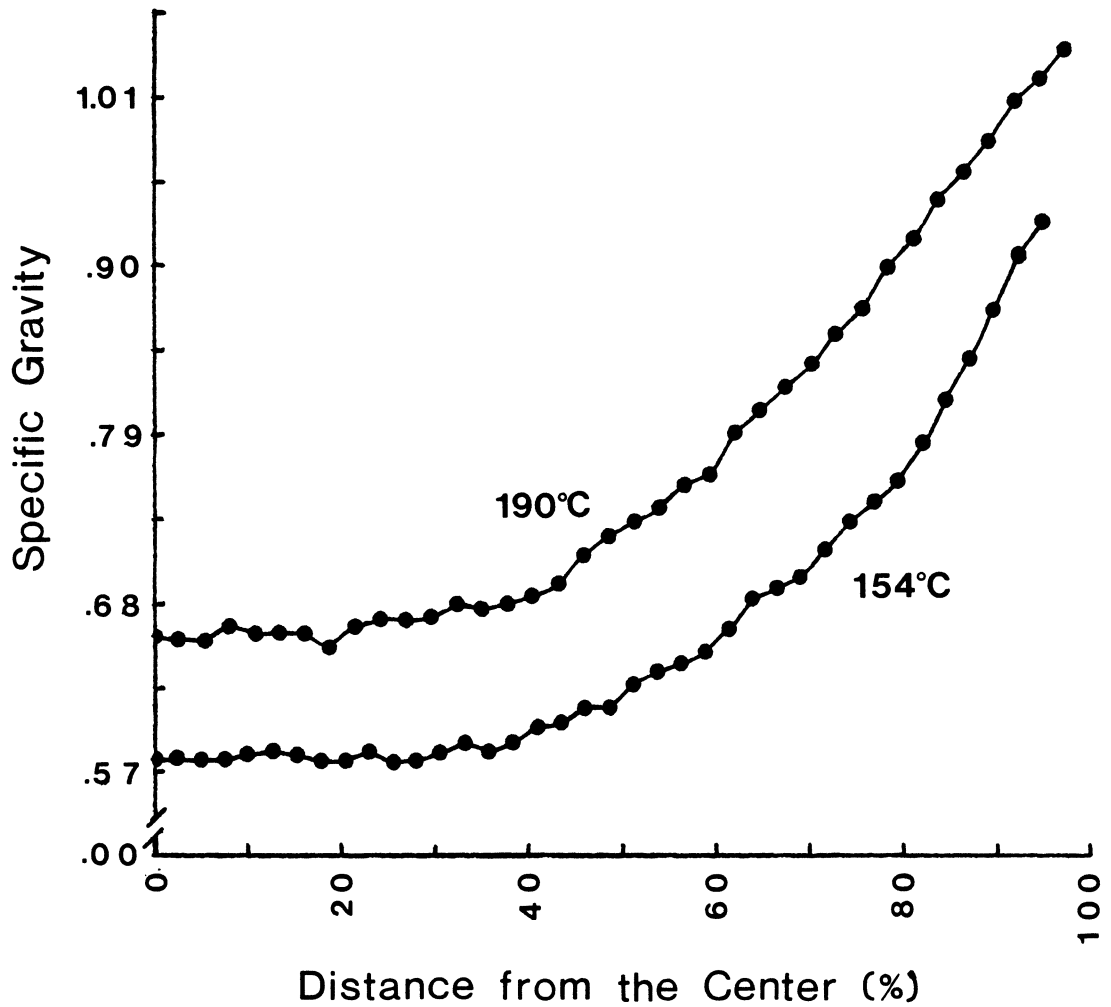


Figure 9: Comparison of average specific gravity profiles for panels pressed at 1 minute press closing time, 15 percent moisture content, and 154 and 190°C (310 and 375°F) platen temperatures.

each of the panels during pressing, final thicknesses greater than this were a result of recoverable deformation or springback. The higher temperature level promoted permanent deformation by providing for flow of the amorphous regions while reducing the amount of recoverable elastic deformation. Since springback is a function of the elastic recovery, the higher temperature resulted in decreased springback, decreased final thickness, and increased specific gravity.

The influences of temperature and moisture content on the specific gravity profile appear to be interrelated. At the higher level of one parameter, the other exhibits a significant influence. At the lower level of one parameter, the influence of the other is reduced. This interaction is explained by the compounding of the previously mentioned plasticization effects of the temperature and moisture parameters. The 6 percent moisture content is probably insufficient to reduce the glass transition temperature of the lignin below the temperature available at most locations through the panel before most of the compaction has occurred. Likewise, the 154°C (310°F) temperature level is inadequate to exceed the glass transition temperature provided by the 15 percent moisture content level. The moisture content and temperature environment present at any particular location through the thickness is further complicated by the time element. Time influences formation of the specific gravity profile in that the temperature and moisture content levels present, at a particular layer and time during the pressing cycle, are constantly changing.

## Layer-Shear Analysis

The results which Suchsland (1977) and, Hall and Haygreen (1983) obtained in relating compression shear values to internal bond indicated a good correlation between these two test methods. These two investigations employed compressive shear as a measure of the weakest plane through the panel thickness. By controlling the location of shear, this test method provides a measure of the bond quality at various layers in the panel. This relatively simple test procedure provides information to facilitate the expanded use of design techniques in the manufacture of flakeboard panels.

The use of 2.5 by 10 cm (1 by 4 inch) specimens was deemed necessary to avoid edge crushing similar to that encountered by Shen and Carroll (1970) in testing torsional layer-shear specimens. The long dimension provides a bearing area, twice that provided by the square specimen of the same cross-sectional area and therefore only half the bearing stress. It was desirable to maintain the same cross-sectional area as used for internal bond testing for comparative purposes with respect to sample size.

Each panel provided 18 shear specimens, 6 to be sheared at each of three different layers. The target half-thickness of the panels was 0.95 cm (0.375 in.). Specimens were tested at layers which were 0.32 and 0.63 cm (0.125 and 0.250 in.) inward from the surface of the panel, and at the core. These layers were chosen to tri-sect the half-thickness of the panel and provide an adequate representation of the layer-shear strength through the thickness, assuming symmetry about the

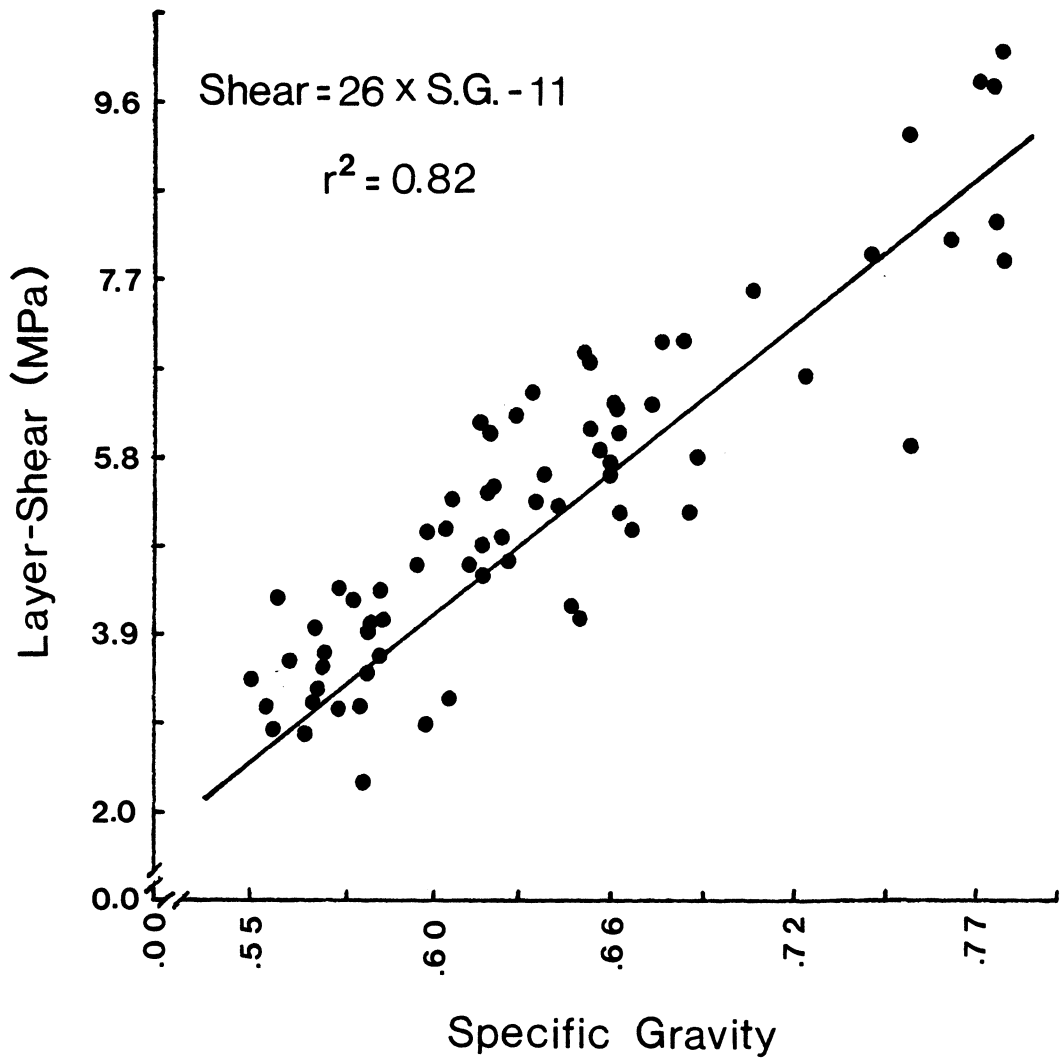
central plane.

The results of the specific gravity profile analysis provided the information necessary to relate the shear strength to the specific gravity at the same location. Although the technique used in determining the specific gravity profiles provides an excellent general analysis of the overall curve, some variability is associated with analysis at a specific point (Laufenburg, 1986). For this reason, the average profiles were obtained for each of the 24 panels and the various layers were analyzed on the basis of these average profiles. The profiles were manually evaluated to obtain the specific gravity values which corresponded to each of the average shear values at the 3 positions in each panel. This provided 72 average shear values with corresponding average specific gravity.

Layer-shear and specific gravity values were statistically analyzed to determine the correlation and regression relationships between the two variables. Figure 10 indicates the scatterplot of the average layer-shear stress versus the average layer specific gravity. The simple linear regression line obtained for the relationship of these two values is described empirically as;

$$\text{Layer-Shear (MPa)} = 26 \times (\text{Specific Gravity}) - 11$$

The regression line was included on the scatterplot to demonstrate the variability of the values about this line. The linearity of this regression relationship is applicable only to the range of specific gravity values obtained in this investigation. The relationship is not intended to provide for extrapolation beyond these limits since the overall relationship is probably curvilinear. The relationship provided



an r-squared value of 0.824, indicating that over 82 percent of the variability observed in the average shear values was associated with the specific gravity.

The average shear stress at failure, along with the coefficient of variation (C.V.), for the eight treatment levels are listed in Tables 8, 9, and 10 for the core, intermediate, and face layers, respectively. From examination of the C.V. values, it is evident that the coefficient of variation for the core shear values are similar to those obtained for the internal bond values (Table 7). Hall and Haygreen (1983) also observed similar C.V.s for internal bond values and weakest plane compressive shear values.

For every treatment level, the coefficients of variation decrease for the intermediate values and decrease further for the face shear values. This trend is probably a result of an increasing variation of heat and moisture transport as the distance from the surface increases. It is also apparent that the C.V. values are consistently higher for the higher (15%) moisture content level, regardless of the location of shear, level of temperature, or length of press closing time considered. The rate of press closing also resulted in a consistent change in the C.V. values. The faster rate of closing was associated with higher coefficients of variation.

An analysis of variance based on the complete factorial design was executed to determine significant influences exhibited by the three main parameters of temperature, moisture content, and press closing time. All possible two-way interactions of the three main parameters were also examined for statistical significance. All tests for significance were

Table 8: Summary of average core layer-shear values and coefficients of variation for the eighteen observations, for each of the eight treatment levels of panel manufacture.

Platen Temperature (°C (°F))	Initial Mat Moisture Content (%)	Press Closing Time (min.)	Average Stress (MPa) (psi)		Coefficient of Variation (%)
154 (310)	5	1	2.95	427	26.2
		2	3.04	442	20.8
	15	1	3.96	575	41.2
		2	3.34	484	21.3
190 (375)	5	1	4.55	661	16.5
		2	4.36	633	19.1
	15	1	5.90	856	26.4
		2	4.96	719	22.9

Table 9: Summary of average intermediate layer-shear values and coefficients of variation for the eighteen observations, for each of the eight treatment levels of panel manufacture.

Platen Temperature (°C (°F))	Initial Mat Moisture Content (%)	Press Closing Time (min.)	Average Stress (MPa) (psi)		Coefficient of Variation (%)
154 (310)	5	1	3.73	541	26.0
		2	3.89	565	18.1
	15	1	4.20	608	30.9
		2	3.40	493	19.7
190 (375)	5	1	5.52	801	16.1
		2	5.16	748	14.6
	15	1	6.11	886	25.0
		2	5.59	810	20.7

Table 10: Summary of average face layer-shear values and coefficients of variation for the eighteen observations, for each of the eight treatment levels of panel manufacture.

Platen Temperature (°C (°F))	Initial Mat Moisture Content (%)	Press Closing Time (min.)	Average Stress (MPa) (psi)		Coefficient of Variation (%)
154 (310)	5	1	5.84	847	18.3
		2	5.93	860	10.6
	15	1	6.53	947	19.1
		2	5.66	821	14.6
190 (375)	5	1	7.00	1016	13.5
		2	7.48	1084	12.9
	15	1	9.41	1365	18.8
		2	9.08	1316	17.4

considered at the 0.05 alpha level. Each of the layers of shear testing were individually analyzed to provide indications of significantly influential parameters at each layer.

Temperature was the only main parameter which proved to be significant at the 0.05 level for all three populations of shear testing. Figure 11 indicates an upward shift in the shear values associated with an increased temperature, similar to the shift observed for the specific gravity profiles. The moisture content level was significant at the 0.05 level for the core and face shear values, but not for the values at the intermediate shear values. Though this insignificance is viewed as being related to the heat and mass transfer during pressing, its explanation alludes the author. Figure 12 illustrates a similar upward shift for higher moisture contents as that observed for higher temperature. This shift appears correlated to specific gravity profiles obtained for the same treatments.

Press closing time was observed to be statistically significant in relation to shear values at two of the three layers of shear testing. Press closing time was not significant at the 0.05 level for the values obtained for shear at the face of the panel. Previous relationships which were established between the press closing time and the specific gravity profile, and between specific gravity and shear would intuitively lead one to anticipate a significant influence of press closing time on the shear stress at failure at the face. The location at which the face shear values were determined was not in the region where the influence of press closing time is exhibited on the specific gravity profile, hence a significant influence is not exhibited on the

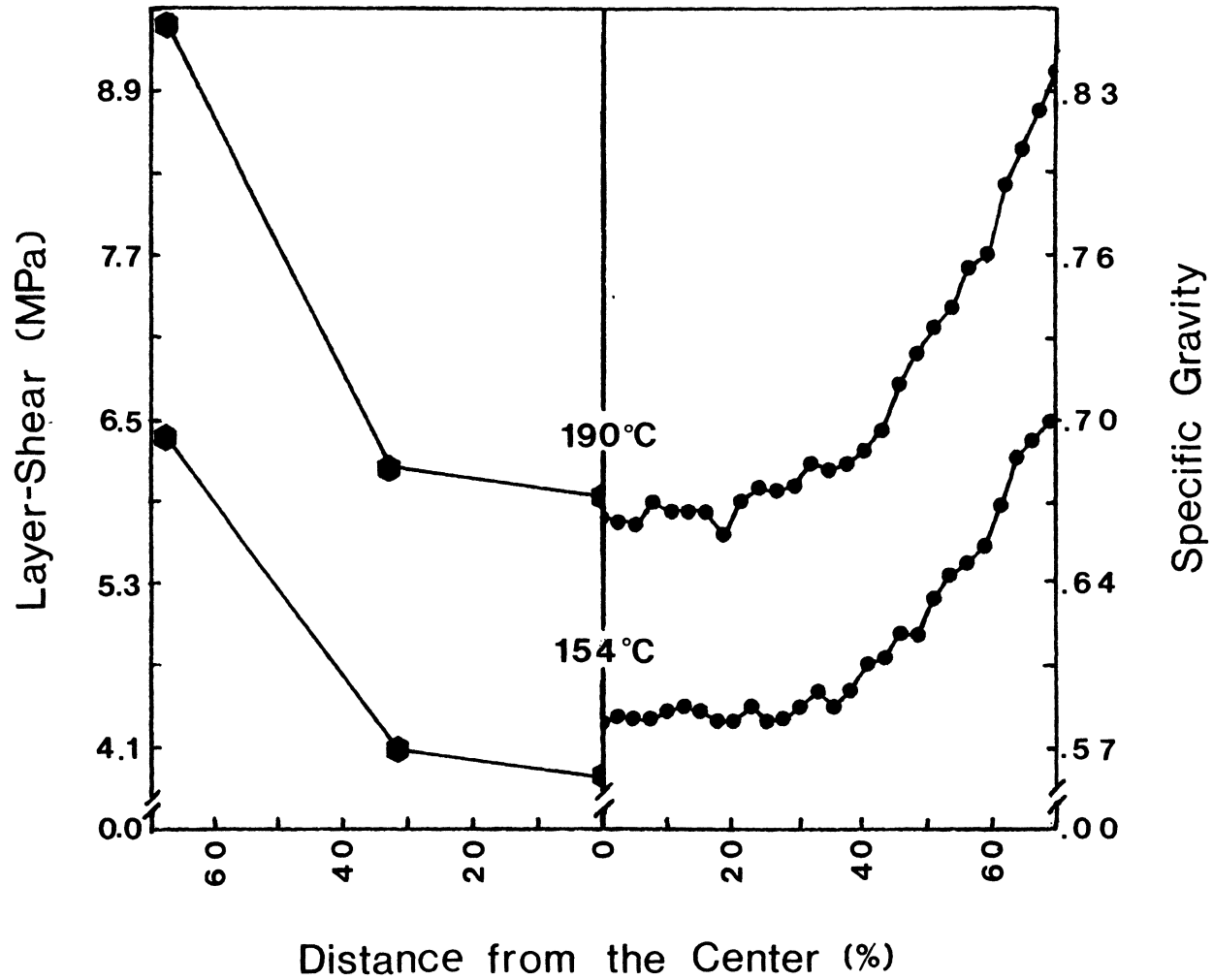


Figure 11: Layer-shear and specific gravity plotted versus distance from the panel center for the treatments of 15% moisture content, 1 minute press closing time, and 154 and 190°C (310 and 375°F).

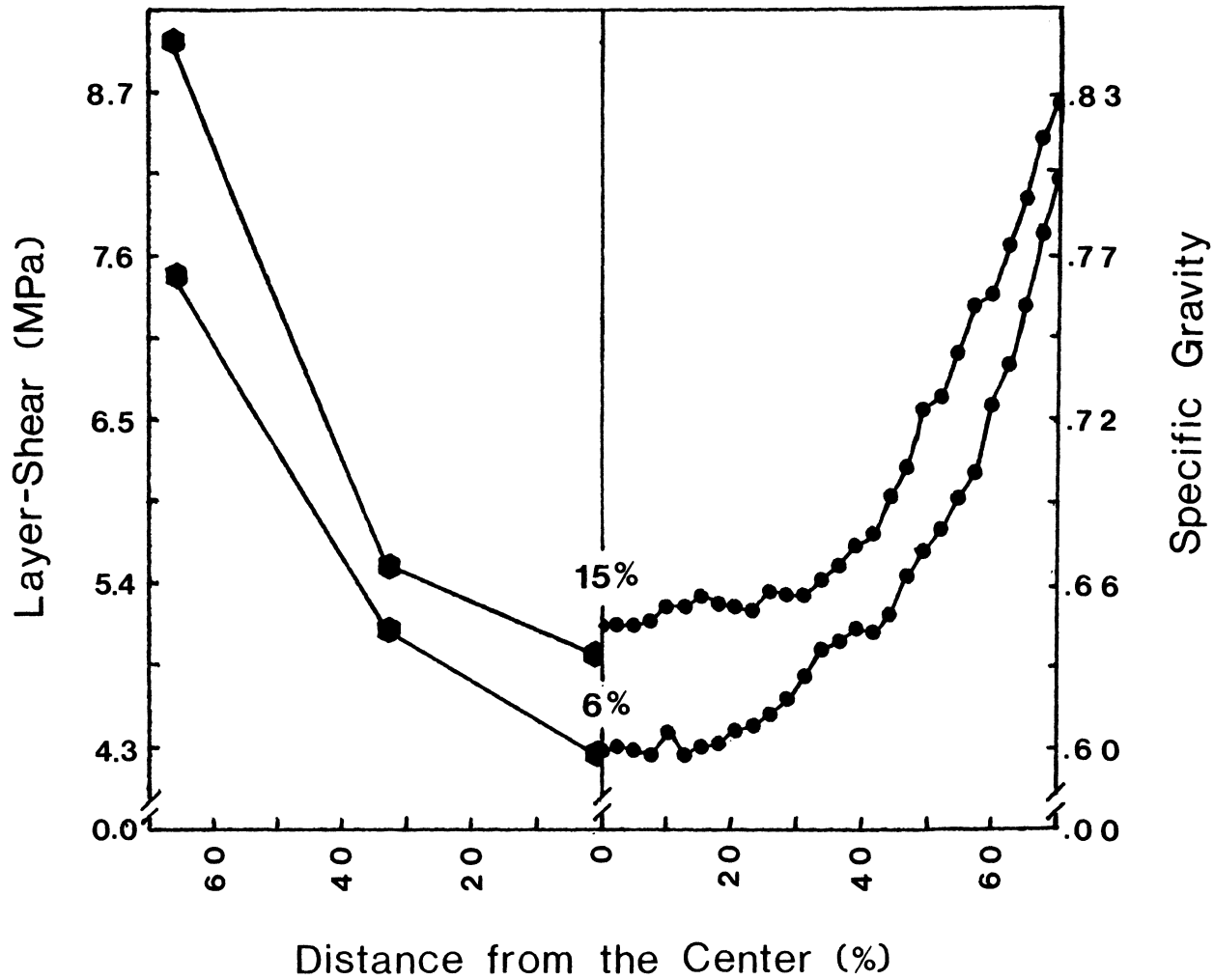


Figure 12: Layer-shear and specific gravity plotted versus distance from the panel center for the treatments of 1 minute press closing time, 190°C (375°F), and 6 and 15 percent moisture content.

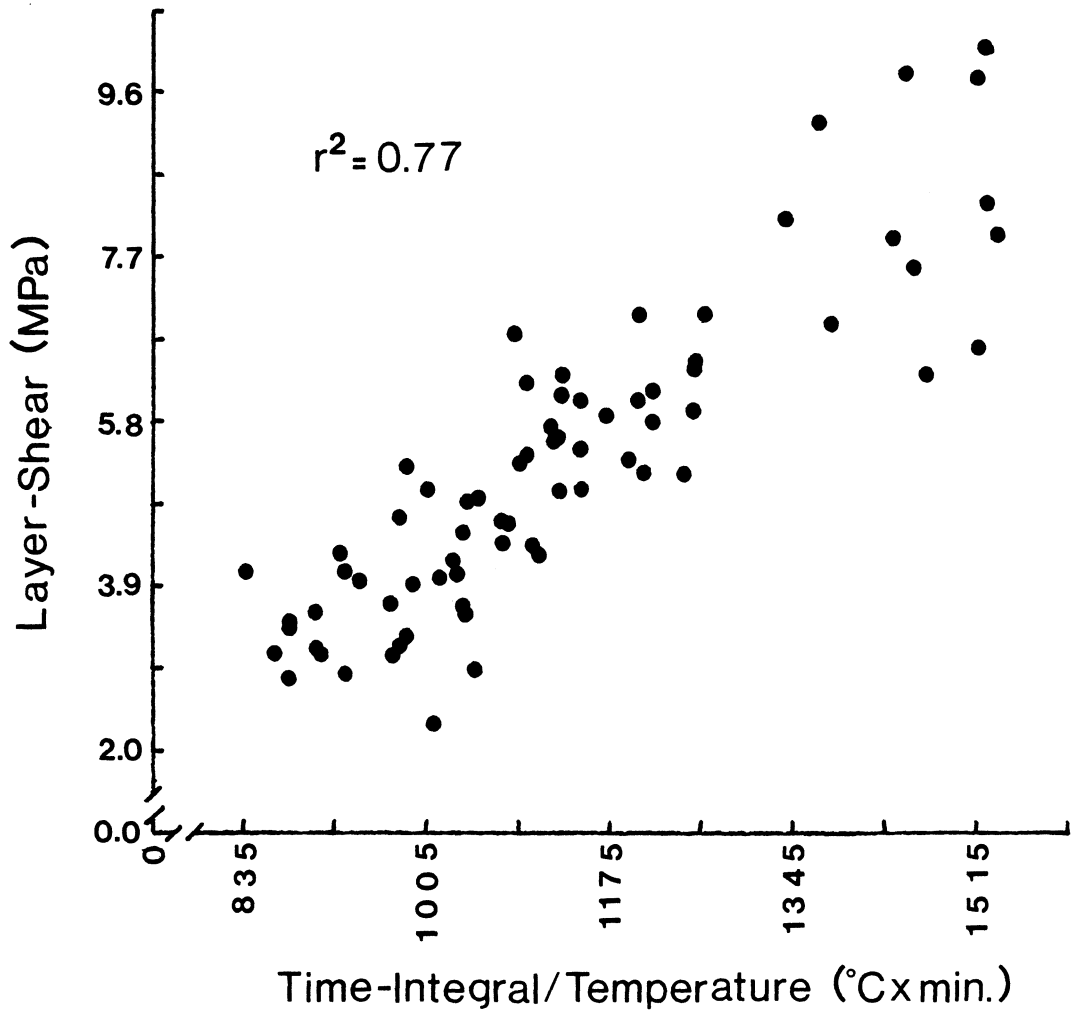
layer-shear at this location.

None of the possible two-way interactions influenced the intermediate layer-shear values at the 0.05 level. The interaction of moisture content with press closing time was, however, significant in relation to both the core and face values obtained for layer-shear. The interaction of moisture content with temperature was also significant in relation to the face layer-shear values.

The quality of the adhesive bond and, therefore, the layer-shear strength, is a function of the local conditions of heat, moisture and pressure surrounding each bonding site. The environmental history of each layer-shear location was compared to the corresponding layer-shear value for the purpose of relating bond quality to localized environmental conditions. Statistical correlations and least squares multiple regression were employed to provide these relationships.

Initial correlations indicated a strong relationship between the 72 shear values and the corresponding time-integrals of temperature. Figure 13 illustrates the scatterplot of these two variables, with an r-squared value of 0.77. This correlation associates over 77 percent of the variability observed in layer-shear with the variability in the time-integral of temperature. A much weaker correlation was found for the dependence of shear on the time-integral of gas pressure with an r-squared value of 0.33. The correlation to the actual press closing time also yielded a low r-squared value of 0.03.

Exhaustive regression and correlation of various combinations of these and other parameters were completed to provide the best possible multiple linear regression. These three variables were retained when



multiple linear regression was performed using a stepwise technique with entry and exclusion levels of 0.05. The summary of the regression relationships, including model and partial r-squared values along with the estimates of the parameter coefficients, are listed in Table 11. The results of this analysis indicate an r-squared of 0.90, and the actual temperature, moisture content, and pressure history are statistically significant in determining the shear strength at various layers through the flakeboard panels. Table 11 also includes the results of a similar stepwise regression procedure used to analyze specific gravity as a function of environmental history.

#### Resin Penetration

Microscopic examination of a limited sample of specimens from these panels using a technique similar to that employed by Furuno, et al. (1983) provided a qualitative evaluation of the resin/wood interphase. The visual examination of the fluorescent micrographs produced using this technique yielded three significant observations. The penetration of the phenol-formaldehyde resin into the yellow poplar flakes was observed to be increased by pressing at a higher moisture content. The penetration also appeared to be greater at the core than at the face of the panel. Both of observations are believed to be related to the environmental history specific to the particular location through the panel thickness. The environmental history influences the depth and uniformity of penetration by affecting the viscosity and degree of cure of the resin.

Table 11: Summary of regression results for dependent variables shear and specific gravity.

Variable	Dependent Variable Shear			Dependent Variable Specific Gravity		
	Parameter Estimate	Partial $r^2$	Model $r^2$	Parameter Estimate	Partial $r^2$	Model $r^2$
Intercept	-5535772	-----	-----	0.207187	-----	-----
Temperature Time-Integral	7927	0.778	0.778	0.000272	0.744	0.744
Gas Pressure Time-Integral	2124	0.095	0.872	0.000088	0.082	0.826
Platen Pressure Time-Integral	-----	-----	-----	0.000004	0.047	0.873
Actual Press Closing Time	598707	0.024	0.896	0.006242	0.011	0.884

The third significant observation concerning the resin penetration characteristics in flakeboard was that the resin is estimated to have been present at 95 percent of the interfaces between flakes. The limited nature of this study prohibits concrete conclusions based on these observations, but this finding is in agreement with the findings of Bolton, et al. (1985). The establishment of a contiguous network or matrix of resin on the wood surfaces would provide the highest mechanical properties and provide for the modelling of flakeboard as a particulate composite.

#### Summary and Conclusions

The evaluation of the time-integrals of temperature, gas pressure, and platen pressure indicated that the pressing parameters provide both simple and interactive influences on the environmental history of a location through the thickness of a mat. The time-integral of platen pressure was observed to be a function of the plasticization of the wood and was increased by an increase in press closing time, or a decrease in moisture content or temperature. The time-integral of gas pressure was believed to be a function of the evaporation and gaseous expansion of water, and was increased by increased moisture content and temperature, but was unaffected by the press closing time. The time-integral of temperature was increased by an increase in platen temperature and decrease in press closing time but was unaffected by moisture content, which is due to a heat of vaporization effect.

Values obtained for internal bond were also influenced by the main

parameters and by the interaction of the parameters. Internal bond was increased by a higher platen temperature, by a lower moisture content, and by a faster press closing time. The interaction of moisture content with platen temperature and with press closing time provided a further influence of the level of moisture on internal bond. In addition, the penetration of the resin was qualitatively observed to be increased at higher moisture content.

The specific gravity profile of the panel was found to be influenced by press closing time, platen temperature, and initial mat moisture content. Decreased press closing time facilitated formation of a U-shaped density profile, and the profile became more uniform at slower closing times. Increased temperature or moisture content resulted in an upward shift of the entire profile. This phenomenon was believed to be a function of the elastic recovery (springback) of the panel thickness due to various levels of wood plasticization.

The values obtained for layer-shear increased consistently from the core to the face while the coefficient of variation decreased consistently in the same direction. Good correlation between layer-shear and specific gravity, as well as a similar parameter dependency was observed. Each layer of shear testing was influenced by platen temperature, moisture content, and press closing time through either a simple or interactive relationship. Regression of layer-shear based on the various time-integrals indicated the significant influence of the environmental history and a strong correlation with temperature history.

These findings lead to the following conclusions concerning the

layer properties of flakeboard panels:

1. The parameters of platen temperature, initial mat moisture content, and press closing time directly and interactively determine the environmental history specific to a particular plane through the thickness which significantly influences the strength at the location and the distribution of strength through the panel.
2. The layer-shear and specific gravity at a particular plane through the thickness of a flakeboard panel are developed through mechanisms which are similarly dependent on the local environmental history and especially dependent on the temperature history.
3. The mechanism of layer-shear failure is through adhesive and wood fracture because flake deformation inhibits failure strictly in the adhesive component. The technique of layer-shear testing therefore provides a measure of bond quality and wood densification.
4. The results obtained from examination of in situ resin penetration in flakeboard were inadequate to establish a relationship between resin penetration and layer-shear.

## SUMMARY OF CONCLUSIONS

The evaluation of the penetration of phenol-formaldehyde resin into aspen and douglas-fir, and of the layer properties of yellow poplar flakeboard as both were influenced by hot-pressing parameters yielded the following conclusions:

1. The uniformity of resin penetration is influenced more by the natural variability of wood than by temperature, moisture content, time, or pressure.
2. The pressing parameters of temperature, moisture content, and time influence the extent of resin penetration by controlling the viscosity of resin. Pressure influences penetration by providing a mechanism to control hydrodynamic flow. The interactions of moisture content with pressure and temperature with time also influence resin penetration by affecting the viscosity, with the time-temperature interaction providing the larger influence.
3. The parameters of platen temperature, initial mat moisture content, and press closing time directly and interactively determine the environmental history specific to a particular plane through the thickness which significantly influences the strength at the location and the distribution of strength through the panel.

4. The layer-shear and specific gravity at a particular plane through the thickness of a flakeboard panel are developed through mechanisms which are similarly dependent on the local environmental history and especially on the temperature history.
5. The mechanism of layer-shear failure is through adhesive and wood fracture because flake deformation inhibits failure strictly in the adhesive component. The technique of layer-shear testing therefore provides a measure of bond quality and wood densification.
6. The results from examination of in situ resin penetration in flakeboard were inadequate to establish a relationship between resin penetration and layer-shear.

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APPENDIX A  
EXPERIMENTAL DATA FOR RESIN PENETRATION

Table A1: Summary of average area of penetration and coefficient of variation of phenol-formaldehyde resin into aspen flakes pressed at 105°C (221°F), 4% moisture content, and 2.07 MPa (300 psi) of pressure.

Specimen #	2 Minute Pressing Time		5 Minute Pressing Time	
	Resin * Penetration (u <sup>2</sup> )	Coefficient of Variation (%)	Resin * Penetration (u <sup>2</sup> )	Coefficient of Variation (%)
1	6262	29.8	3853	57.8
2	5378	40.7	3991	21.8
3	5324	22.6	4378	48.1
4	6867	32.7	2782	30.3
5	4969	37.5	2693	41.4
6	3569	49.8	3342	25.9
7	4520	48.7	4169	35.0
8	3142	34.9	3138	41.9
9	5111	25.7	3902	56.9
10	4564	36.7	4564	46.7

\* Values represent the average of 24 adjacent penetration segments along a single, continuous bondline.

Table A2: Summary of average area of penetration and coefficient of variation of phenol-formaldehyde resin into aspen flakes at 155°C (311°F) and 4% moisture content.

Specimen #	2.07 MPa Pressure 2 Min. Press Time		2.07 MPa Pressure 5 Min. Press Time		6.89 MPa Pressure 5 Min. Press Time	
	Resin * Penetration ( $\mu^2$ )	C. V. (%)	Resin * Penetration ( $\mu^2$ )**	C. V. (%)	Resin * Penetration ( $\mu^2$ )	C. V. (%)
1	3920	40.7	4653	38.8	3338	47.3
2	8818	26.5	4653	28.5	4458	26.6
3	5009	34.1	4400	41.7	6174	22.5
4	5493	36.9	4022	36.1	3667	49.0
5	7111	18.9	3964	41.8	4667	26.6
6	4756	43.9	6809	27.3	4013	33.5
7	3813	32.8	5391	27.8	5520	27.9
8	3342	30.2	4836	50.2	3089	38.8
9	4489	38.6	4231	37.2	4916	50.8
10	4493	49.3	-----	-----	4364	30.9

\* Values represent the average of 24 adjacent penetration segments along a single, continuous bondline.

\*\* One specimen slide was lost for this set of pressing parameters. All calculations made using this cycle were based on 216 individual area of resin penetration readings.

Table A3: Summary of average area of penetration and coefficient of variation of phenol-formaldehyde resin into aspen flakes pressed at 155°C (311°F) and 15% moisture content for 5 minutes.

Specimen #	2.07 MPa Platen Pressure		6.89 MPa Platen Pressure	
	Resin * Penetration (u <sup>2</sup> )	Coefficient of Variation (%)	Resin * Penetration (u <sup>2</sup> )	Coefficient of Variation (%)
1	3053	26.5	6671	36.2
2	5316	44.1	9062	19.0
3	4409	19.2	2689	40.9
4	6009	28.2	3929	34.3
5	7671	33.4	4867	31.3
6	7316	26.8	5622	20.6
7	4342	19.7	4476	39.3
8	4440	38.0	4729	35.7
9	4342	25.1	4893	44.2
10	3653	47.4	5484	28.0

\* Values represent the average of 24 adjacent penetration segments along a single, continuous bondline.

Table A4: Summary of average area of penetration and coefficient of variation of phenol-formaldehyde resin into Douglas-fir earlywood and latewood flakes pressed at 155°C (311°F), 4% moisture content, and 2.07 MPa of pressure for 5 minutes.

Specimen #	Earlywood		Latewood	
	Resin * Penetration (u <sup>2</sup> )	Coefficient of Variation (%)	Resin * Penetration (u <sup>2</sup> )	Coefficient of Variation (%)
1	5502	30.7	2462	40.1
2	9733	22.9	1604	17.8
3	7333	23.7	2142	22.1
4	8111	27.2	1604	34.6
5	5329	29.0	2733	31.9
6	4796	22.9	1329	29.6
7	5982	22.9	2204	30.6
8	6804	21.0	4076	19.1
9	5462	18.9	2684	29.3
10	6227	24.2	2507	45.9

\* Values represent the average of 24 adjacent penetration segments along a single, continuous bondline.

**APPENDIX B**  
**EXPERIMENTAL DATA FOR PANEL MANUFACTURE AND TESTING**

Table B1: Summary of temperature time-integrals and distance of temperature probes from the center plane of the panel, for the face and core probes.

Panel #	Panel Surface*		Face		Core	
	Temp-Time Integral (°C x min.)	Dist. to Core (cm)	Temp-Time Integral (°C x min.)	Dist. to Core (cm)	Temp-Time Integral (°C x min.)	Dist. to Core (cm)
1	1828	0.965	1340	0.668	1082	0.081
2	1828	0.991	1336	0.610	1073	0.041
3	1815	0.973	1374	0.668	1084	0.201
4	1789	0.975	1154	0.795	1008	0.008
5	1763	0.973	1299	0.700	1012	0.079
6	1802	0.980	1190	0.579	1017	0.102
7	1802	1.013	1214	0.732	970	0.015
8	1802	0.996	1151	0.638	928	0.076
9	1789	0.998	1171	0.732	960	0.003
10	1802	0.996	1469	0.792	1093	0.008
11	1853	0.978	1316	0.602	1107	0.086
12	1828	0.980	1287	0.726	1044	0.249
13	2271	0.965	1615	0.617	1226	0.020
14	2239	0.945	1524	0.620	1239	0.119
15	2255	0.935	1684	0.726	1270	0.135
16	2207	0.970	1620	0.820	1119	0.023
17	2176	0.935	1587	0.805	1180	0.269
18	2207	0.955	1581	0.706	1152	0.061
19	2191	0.932	1394	0.617	1065	0.048
20	2223	0.963	1449	0.711	1049	0.091
21	2207	0.965	1375	0.648	1092	0.076
22	2223	0.940	1586	0.709	1219	0.058
23	2287	0.952	1690	0.681	1257	0.089
24	2271	0.947	1624	0.683	1247	0.046

\* Time-integrals for the panel surface are obtained by assuming exposure to the platen temperature for the entire pressing cycle. Distance for surface is the half thickness of the panel.

Table B2: Summary of gas pressure time-integrals and distance of gas pressure probes from the center plane of the panel, for the face and core probes.

Panel #	Face		Core	
	Gas Pressure Time-Integral (kPa x min.)	Distance from Center (cm)	Gas Pressure Time-Integral (kPa x min.)	Distance from Center (cm)
1	132	0.739	104	0.216
2	100	0.617	82	0.005
3	99	0.617	69	0.038
4	107	0.752	124	0.003
5	104	0.612	104	0.094
6	103	0.704	86	0.030
7	277	0.757	285	0.036
8	457	0.772	370	0.018
9	342	0.605	208	0.020
10	497	0.833	482	0.003
11	478	0.798	447	0.173
12	342	0.579	348	0.198
13	170	0.704	177	0.051
14	186	0.655	171	0.196
15	369	0.518	349	0.025
16	229	0.668	199	0.028
17	323	0.787	279	0.163
18	283	0.650	245	0.099
19	901	0.686	756	0.033
20	902	0.706	756	0.142
21	915	0.607	701	0.173
22	1053	0.693	992	0.066
23	1024	0.866	905	0.168
24	991	0.668	959	0.005

Table B3: Summary of the time-integrals of platen pressure for the 24 yellow poplar flakeboard panels.

Platen Temperature (°C (°F))	Initial Mat Moisture Content (%)	Press Closing Time (min.)	Panel #	Platen Pressure Time-Integral (kPa x min.)
154 (310)	6	1	1	21960
			2	18846
			3	18181
		2	4	18879
			5	18495
			6	20336
	15	1	10	12139
			11	12146
			12	11655
		2	7	13827
			8	15222
			9	13794
190 (375)	6	1	13	12286
			14	14086
			15	14205
		2	16	15261
			17	14042
			18	14701
	15	1	22	13365
			23	13026
			24	12647
		2	19	15772
			20	14328
			21	14391

Table B4: Summary of regression equations for the time-integral of temperature as a function of the square of the distance from the center plane of the panel.

Platen Temperature (°C (°F))	Initial Mat Moisture Content (%)	Press Closing Time (min.)	Panel #	Intercept (b)	Slope (a)
154 (310)	6	1	1	981	866
			2	1013	819
			3	987	885
		2	4	904	756
			5	975	796
			6	931	822
	15	2	7	878	830
			8	838	932
			9	866	838
		1	10	1041	726
			11	994	871
			12	879	917
190 (375)	6	1	13	1078	1268
			14	1112	1211
			15	1130	1189
		2	16	1042	1096
			17	1043	1137
			18	1075	1176
	15	2	19	989	1327
			20	931	1291
			21	982	1242
		1	22	1122	1148
			23	1132	1217
			24	1122	1202

Table B5: Summary of internal bond values for the 12 panels manufactured at 154°C (310°F), six specimens from each panel.

Panel #	6% Moisture Content 1 Min. Press Closing			6% Moisture Content 2 Min. Press Closing		
	1	2	3	4	5	6
Internal Bond	405	286	551	376	481	525
(kPa)	363	309	423	305	404	480
	343	285	419	441	592	367
	257	301	516	458	245	538
	431	297	699	418	617	466
	501	239	296	465	439	529
Average	383	286	484	410	463	484
C.V. (%)	21.7	8.7	28.5	14.9	29.8	13.3

Panel #	15% Moisture Content 2 Min. Press Closing			15% Moisture Content 1 Min. Press Closing		
	7	8	9	10	11	12
Internal Bond	285	315	434	348	401	439
(kPa)	174	242	479	443	370	206
	148	312	543	374	621	558
	308	368	369	360	317	612
	375	463	321	400	402	522
	289	315	191	408	376	338
Average	263	336	389	389	414	446
C.V. (%)	32.6	22.1	32.2	9.0	25.5	34.1

Table B6: Summary of internal bond values for the 12 panels manufactured at 190°C (375°F), six specimens for each panel.

Panel	6% Moisture Content 1 Min. Press Closing			6% Moisture Content 2 Min. Press Closing		
	13	14	15	16	17	18
Internal Bond	772	675	619	637	668	510
(kPa)	471	604	356	627	455	551
	478	357	827	652	753	739
	791	735	851	576	671	574
	827	607	902	418	770	736
	409	579	767	592	554	827
Average	625	593	720	584	645	656
C.V. (%)	30.5	21.7	28.2	14.7	18.7	19.5

Panel	15% Moisture Content 2 Min. Press Closing			15% Moisture Content 1 Min. Press Closing		
	19	20	21	22	23	24
Internal Bond	268	566	292	377	277	481
(kPa)	485	128	152	415	469	361
	328	315	327	534	558	581
	233	257	424	347	476	532
	212	342	53	415	276	455
	230	194	197	394	288	409
Average	293	300	241	414	391	470
C.V. (%)	35.1	50.6	55.3	15.6	32.0	17.0

Table B7: Summary of core layer-shear values for the 12 panels manufactured at 154°C (310°F), six specimens from each panel.

Panel #	6% Moisture Content 1 Min. Press Closing			6% Moisture Content 2 Min. Press Closing		
	1	2	3	4	5	6
Stress at Failure (MPa)	2.48	1.19	3.29	2.69	2.35	2.37
	3.27	3.05	4.30	2.46	2.83	3.35
	3.27	2.29	2.89	2.86	2.80	3.34
	4.54	2.08	3.54	3.98	4.05	2.56
	3.07	2.93	3.11	3.23	4.27	2.54
	2.65	2.31	2.75	3.76	2.23	3.12
Average	3.22	2.31	3.32	3.16	3.09	2.88
C.V. (%)	22.6	29.0	16.9	19.2	28.0	15.3

Panel #	15% Moisture Content 2 Min. Press Closing			15% Moisture Content 1 Min. Press Closing		
	7	8	9	10	11	12
Stress at Failure (MPa)	0.93	1.84	3.28	3.85	3.26	3.96
	2.20	3.23	2.49	4.44	3.02	2.47
	1.85	6.14	3.05	4.11	5.18	3.89
	4.92	4.25	2.61	4.99	3.91	3.96
	3.66	2.85	3.87	4.18	4.27	2.29
	3.39	6.08	3.47	5.63	3.92	4.05
Average	2.82	4.07	3.13	4.53	3.93	3.44
C.V. (%)	50.9	43.3	16.8	14.6	19.6	23.9

Table B8: Summary of core layer-shear values for the 12 panels manufactured at 190°C (375°F), six specimens for each panel.

Panel	6% Moisture Content 1 Min. Press Closing			6% Moisture Content 2 Min. Press Closing		
	13	14	15	16	17	18
Stress at Failure (MPa)	5.91	4.75	4.31	3.04	5.29	4.40
	4.58	4.00	5.36	2.74	3.85	4.58
	4.15	4.20	4.99	4.34	4.63	4.23
	4.78	3.76	5.80	3.96	5.26	4.78
	4.27	4.42	4.46	3.66	4.60	5.90
	2.67	4.43	5.14	3.66	5.56	4.02
Average	4.39	4.26	5.01	3.57	4.87	4.65
C.V. (%)	23.9	8.2	11.1	16.5	12.9	14.3

Panel	15% Moisture Content 2 Min. Press Closing			15% Moisture Content 1 Min. Press Closing		
	19	20	21	22	23	24
Stress at Failure (MPa)	5.34	4.53	5.81	6.11	9.43	4.75
	6.00	4.18	4.53	5.65	7.59	5.22
	4.11	5.81	3.39	5.12	3.99	7.47
	8.40	5.89	5.80	5.67	7.19	5.68
	2.77	3.39	4.73	5.37	3.97	5.00
	5.05	5.65	3.89	6.51	5.95	5.54
Average	5.28	4.91	4.69	5.74	6.35	5.61
C.V. (%)	35.9	21.0	21.0	8.8	33.8	17.4

Table B9: Summary of Intermediate layer-shear values for the 12 panels manufactured at 154°C (310°F), six specimens from each panel.

Panel #	6% Moisture Content 1 Min. Press Closing			6% Moisture Content 2 Min. Press Closing		
	1	2	3	4	5	6
Stress at Failure (MPa)	3.12	3.12	3.83	4.54	4.49	3.60
	3.05	3.32	4.84	3.26	3.81	3.47
	4.21	3.84	3.81	4.21	2.60	3.46
	4.25	2.82	5.12	4.98	5.02	4.51
	5.44	2.79	3.47	3.49	4.94	3.67
	5.15	1.74	3.24	3.36	3.19	3.52
Average	4.20	2.94	4.05	3.97	4.01	3.70
C.V. (%)	23.6	23.8	18.7	17.8	24.5	10.9
Panel #	15% Moisture Content 2 Min. Press Closing			15% Moisture Content 1 Min. Press Closing		
	7	8	9	10	11	12
Stress at Failure (MPa)	1.27	1.65	3.14	4.78	2.89	3.87
	2.96	3.49	4.69	3.80	4.16	4.25
	2.91	5.43	4.00	4.56	2.85	3.83
	4.60	3.96	2.27	4.46	4.18	4.66
	3.83	3.28	4.42	5.53	5.26	3.79
	3.12	3.08	3.09	4.64	2.66	5.33
Average	3.11	3.48	3.60	4.63	3.67	4.29
C.V. (%)	35.6	35.4	25.6	12.1	28.1	14.2

Table B10: Summary of intermediate layer-shear values for the 12 panels manufactured at 190°C (375°F), six specimens for each panel.

Panel	6% Moisture Content 1 Min. Press Closing			6% Moisture Content 2 Min. Press Closing		
	13	14	15	16	17	18
Stress at Failure (MPa)	5.75	5.48	5.38	3.91	5.24	4.91
	5.31	5.94	5.88	3.78	6.37	4.99
	5.41	5.27	6.66	4.52	5.13	5.89
	5.20	5.83	7.84	4.85	5.14	6.27
	5.21	5.17	5.61	4.52	5.45	5.40
	3.37	4.44	5.67	4.68	6.40	5.45
Average	5.04	5.35	6.17	4.38	5.62	5.48
C.V. (%)	16.7	10.1	15.1	9.8	10.8	9.5

Panel	15% Moisture Content 2 Min. Press Closing			15% Moisture Content 1 Min. Press Closing		
	19	20	21	22	23	24
Stress at Failure (MPa)	5.45	4.85	4.38	8.31	3.53	5.13
	6.09	3.35	4.68	7.45	7.28	5.55
	7.28	5.00	5.18	5.86	5.09	5.86
	9.92	5.81	5.81	7.87	6.06	6.59
	6.12	6.25	4.97	7.33	4.91	6.48
	5.95	4.94	4.54	5.44	4.44	6.75
Average	6.80	5.03	4.93	7.04	5.22	6.06
C.V. (%)	24.1	19.8	10.6	16.2	25.0	10.7

Table B11: Summary of face layer-shear values for the 12 panels manufactured at 154°C (310°F), six specimens from each panel.

Panel #	6% Moisture Content 1 Min. Press Closing			6% Moisture Content 2 Min. Press Closing		
	1	2	3	4	5	6
Stress at Failure (MPa)	5.77	5.97	6.97	5.50	6.37	5.84
	6.25	4.41	6.87	4.83	5.67	5.91
	6.30	4.67	5.22	5.91	5.80	6.39
	7.44	5.97	7.06	7.74	5.98	5.38
	6.34	5.91	5.62	6.52	6.00	5.87
	3.49	4.31	6.61	6.09	4.98	5.94
Average	5.93	5.21	6.39	6.10	5.80	5.89
C.V. (%)	22.2	15.8	12.2	16.2	8.0	5.5
Panel #	15% Moisture Content 2 Min. Press Closing			15% Moisture Content 1 Min. Press Closing		
	7	8	9	10	11	12
Stress at Failure (MPa)	4.66	4.92	6.00	6.24	5.48	6.03
	4.83	6.62	4.85	7.46	5.86	5.92
	3.02	5.81	5.53	6.88	7.67	6.48
	6.37	6.73	6.05	6.94	6.61	7.15
	7.29	7.58	5.18	5.90	6.03	4.66
	5.77	5.81	4.91	8.86	7.30	6.08
Average	5.32	6.24	5.42	7.05	6.49	6.05
C.V. (%)	28.1	14.8	9.7	14.8	13.2	13.5

Table B12: Summary of face layer-shear values for the 12 panels manufactured at 190°C (375°F), six specimens for each panel.

Panel	6% Moisture Content 1 Min. Press Closing			6% Moisture Content 2 Min. Press Closing		
	13	14	15	16	17	18
Stress at Failure (MPa)	6.30	6.47	7.20	6.68	8.11	9.54
	6.97	6.76	9.17	8.26	7.70	6.45
	6.17	6.43	7.20	7.14	7.20	7.52
	6.92	7.01	8.73	6.51	8.73	7.31
	6.42	7.28	8.33	7.09	9.05	7.21
	5.41	6.07	7.19	5.79	6.75	7.51
Average	6.36	6.67	7.97	6.91	7.92	7.59
C.V. (%)	9.0	6.6	11.1	11.9	11.2	13.6

Panel	15% Moisture Content 2 Min. Press Closing			15% Moisture Content 1 Min. Press Closing		
	19	20	21	22	23	24
Stress at Failure (MPa)	11.99	8.25	7.82	10.28	10.12	8.73
	11.07	10.13	10.03	11.51	10.54	11.24
	9.22	8.87	8.66	9.70	6.37	10.24
	6.70	8.14	10.01	9.33	7.38	10.76
	8.17	5.42	8.51	8.61	7.13	7.57
	11.81	8.06	10.52	9.20	8.44	12.31
Average	9.83	8.14	9.26	9.77	8.33	10.14
C.V. (%)	21.8	19.0	11.6	10.4	20.3	17.0

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