United States
Department of
Agriculture

Forest Service

Forest
Products
Laboratory

General
Technical
Report
FPL-50

Water Repellency and Dimensional Stability of Wood

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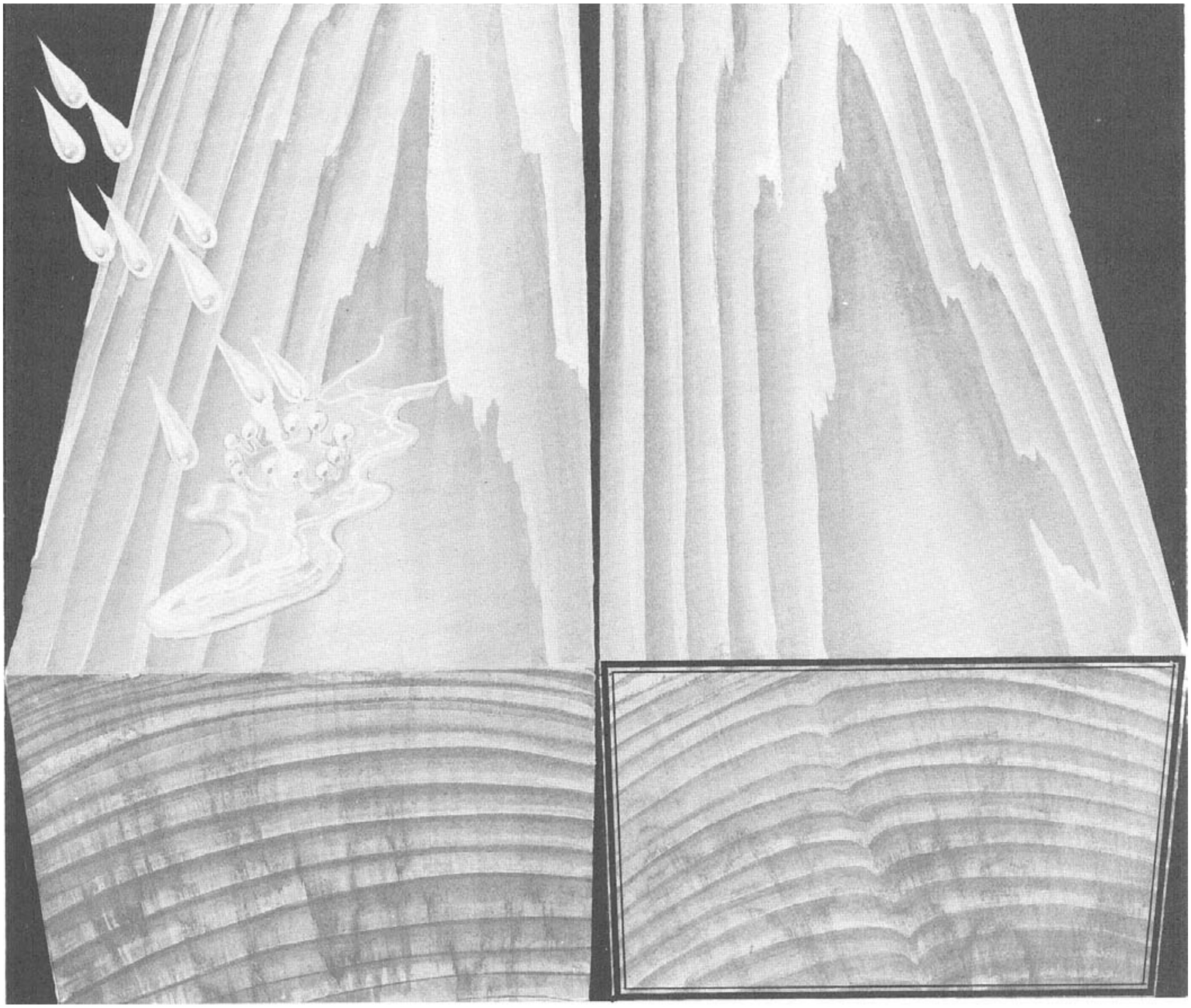

Abstract

A discussion of the interaction between wood and water makes clear the distinction between water repellency of wood (a rate of change) and dimensional stability (a level of equilibrium). A review of methods of treating wood follows, leading to comparison of their effectiveness, description of test procedures to evaluate treatments, and discussion of deficiencies of the tests commonly used, first with regard to water repellency, then to dimensional stability, with final consideration of future research. Figures, numerous tables (introducing some new data), and an extensive bibliography illustrate the discussion.

Keywords: Water repellency, dimensional stability, moisture uptake, contact angle, test conditions, treatments, wood, review, definitions.

December1985

Rowell, Roger M.; Banks, W. Bart. Water repellency and dimensional stability of wood. Gen. Tech. Rep. FPL-50. Madison, WI: U.S. Department of Agriculture, Forest Service, Forest Products Laboratory; 1985.24 p.

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Introduction

The terms water repellency and dimensional stability are often used interchangeably as if they were the same. However, water repellency is a rate phenomenon and dimensional stability is an equilibrium phenomenon, and confusion over these two very different concepts has led some workers to draw inaccurate conclusions from research dealing with the interaction of wood with water. This paper briefly discusses the interaction of wood with water, defines water repellency and dimensional stability, describes treatments for their improvement, evaluates test procedures for their determination in treated wood, and finally, suggests some ideas for future research in this area.

As a building/engineering material wood has advantages in being economical, low in processing energy, renewable, strong, and aesthetically pleasing. Several disadvantageous properties limit its use, however, among them the swelling and shrinking of wood as it takes up and loses moisture.

Wood/Water Interaction

The wood cell wall is mainly composed of polymers with hydroxyl and other oxygen-containing groups (fig. 1) that attract moisture through hydrogen bonding. As water is added to the cell wall, wood volume increases nearly proportionally to the volume of water added (Stamm 1964). Swelling increases until the cell wall is saturated with water. This point is called the fiber saturation point, and ranges from 20 to 50 percent weight gain (Feist and Tarkow 1967). Water, beyond this point, is free water in the void structure and does not contribute to further swelling. This process is reversible, and wood shrinks as it loses moisture below the fiber saturation point.

The cellulose and hemicelluloses (fig. 1), being more hygroscopic than lignin, are mainly responsible for moisture uptake. Sumi et al. (1964) showed that only 60 percent of the total hydroxyl groups in spruce wood are accessible to tritiated water. Stamm (1964) estimated that 65 percent of the cellulose in wood is crystalline and therefore probably not accessible to water. Browning (1963) showed that water sorption is lower in lignin than in the hemicelluloses and celluloses. For *Eucalyptus regnans* he found the fractional contribution of each cell wall polymer to the total water sorption to be cellulose 47 percent, hemicelluloses 37 percent, and lignin 16 percent. This means that lignin (to a small degree), the hemicelluloses (all are noncrystalline in nature), the noncrystalline portion of cellulose, and the surfaces of the cellulose crystallites are responsible for moisture uptake by wood.

The amount of swelling that occurs in wood because of hygroscopic expansion is dependent on the density of the wood (Stamm 1964). The percent volumetric swelling, V, is a function of the dry density, d (in g/cc), and fiber saturation point, K_f (in cc/g percent), so that

$$V=K_f d \tag{1}$$

This equation determines the approximate volumetric swelling of wood passing from an ovendry state to fiber saturation point, and the approximate volumetric shrinkage of wood passing from fiber saturation point to ovendry. Deviations from this relationship are usually found in species high in natural extractives.

Wood is an anisotropic material, which means it swells and shrinks to a different extent in the three anatomical directions–longitudinal (vertical direction), tangential (parallel to annual growth rings), and radial (perpendicular to the annual growth rings). The dimensional changes in the longitudinal direction are generally very small. Maximum tangential swelling and shrinking varies between 2 and 15 percent depending on the density of the wood. Radial changes are smaller, usually about 40 to 70 percent of tangential swelling (Forest Products Laboratory 1974).

Swelling of wood in contact with moisture exerts very large forces. Stamm (1964) determined the theoretical swelling pressure for wood to be 1,630 atmospheres or 24,000 lb/in.^{2}. Tarkow and Turner (1958), measuring the swelling of compressed yellow birch, found less than half this theoretical pressure. Nevertheless, when wood in use swells, the forces developed can cause serious damage. The ancient Egyptians used this swelling force to quarry large stones by drilling holes for the desired size rock, driving dry wooden stakes into these holes, then adding water which caused the wood to swell and crack the rock from the face of the mountain.

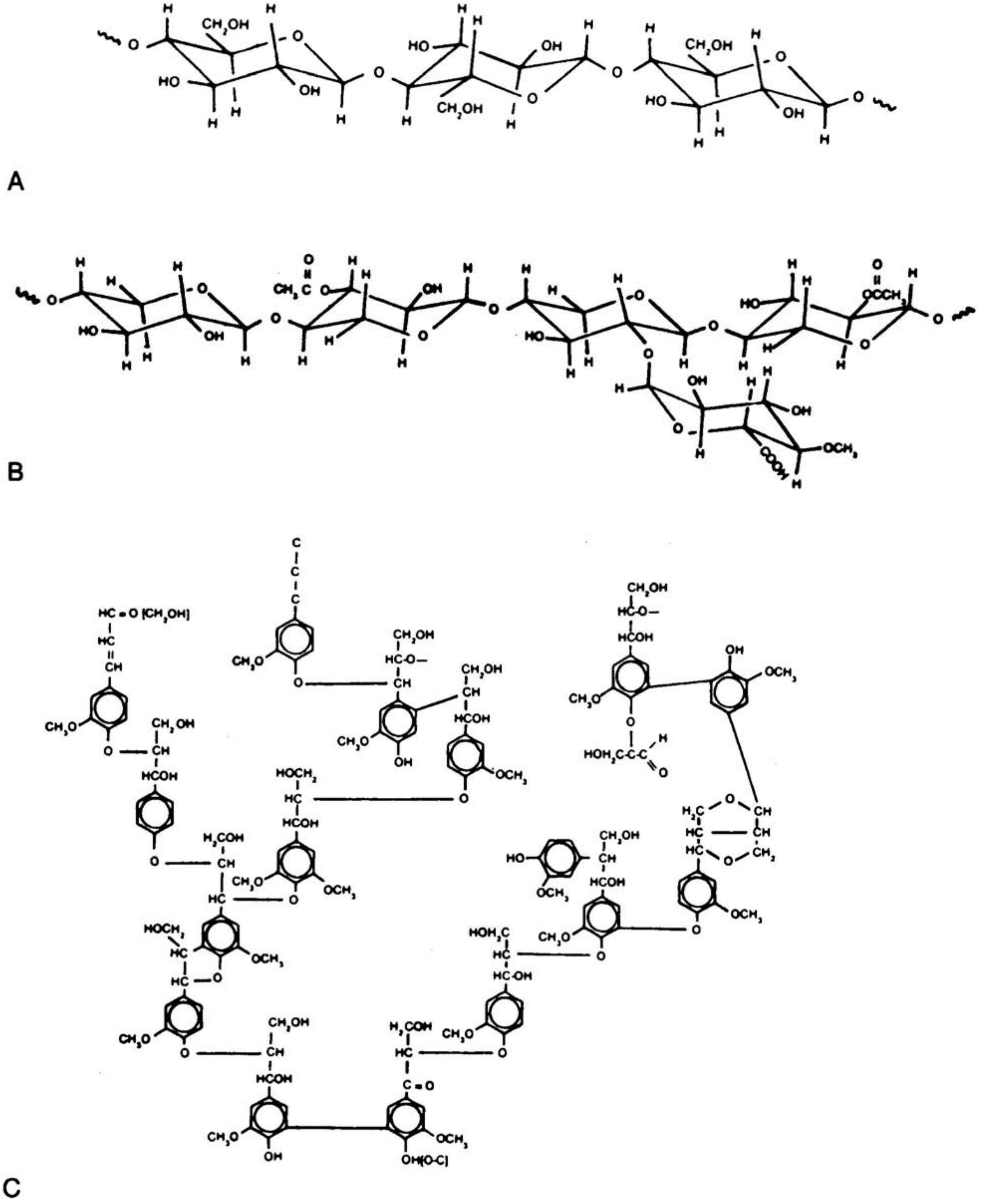

Figure 1.—Major polymer components of wood: (A) partial structure of cellulose, (B) partial structure of a hemicellulose, o-acetyl-4-O-methyl-glucuronoxylan, (C) partial structure of softwood lignin. (ML85 5009)

Treatments to Reduce Wood/Water Interactions

Treatments to reduce the tendency of wood to take on moisture and change dimensions can be broken into two categories: water repellents and dimensional stabilizers. Even though these two are often used synonymously, they are in fact completely different approaches to moisture control in wood. The effectiveness of water-repellency treatment can be defined as the ability of a treatment to prevent or control the rate of liquid water uptake. Dipping in wax or silicone oil are treatments of this type. In contrast, the effectiveness of dimensional stabilizing treatment can be defined as its ability to reduce or prevent the swelling and shrinking of wood resulting from moisture pickup. Dimensional stability depends on the extent rather than the rate of water uptake. Figure 2 shows a typical swelling versus time plot for untreated and treated wood specimens. The upper curve shows how an untreated specimen quickly takes up water and swells to the maximum extent. The lower curve shows a theoretical plot of a treated wood specimen. The reduced rate of water uptake indicates increased repellency while the reduced extent of swelling indicates greater stability. Thus, this treatment seems effective in improving both water repellency and dimensional stability.

Treatments for water repellency and dimensional stability can be described as one of three types shown in figure 3. A Type I treatment reduces the rate of moisture uptake, but in time the extent of swelling is nearly the same as untreated wood. Such a treatment would improve water repellency but not dimensional stability. A Type II treatment reduces the extent of swelling but not the rate at which the wood takes up moisture. Such a treatment would improve dimensional stability but not water repellency. A Type III treatment reduces both the rate of swelling and the extent of swelling. Such a treatment would improve water repellency and dimensional stability.

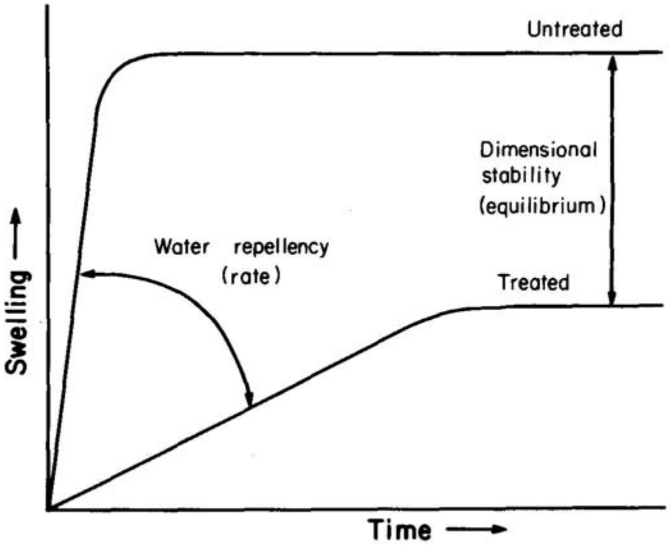

Figure 2.–Hypothetical swelling time plot for water-repellency and dimensional stability treatments. (ML85 5011)

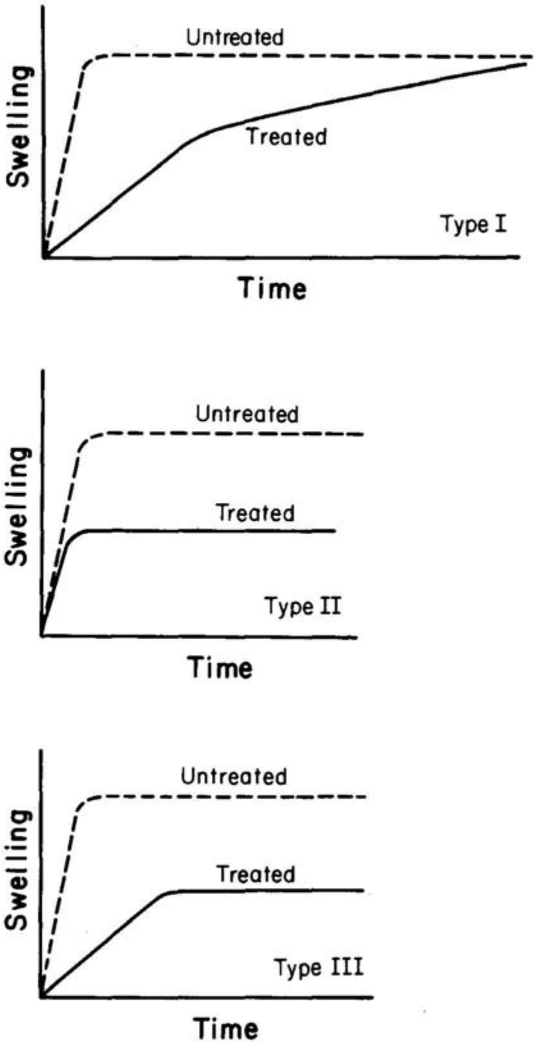

Figure 3.–Hypothetical treatments of wood for: water repellency (Type I). dimensional stability (Type II), water repellency and dimensional stability (Type III). (ML85 5013)

Water Repellency and Moisture Diffusion Processes

Cohesion and Adhesion

An attractive force exists between a solid and a liquid in contact with it. The net value of this force is governed by the relative magnitudes of the cohesive forces within the liquid and the adhesive forces generated between the liquid and solid. Where the adhesion of liquid to solid is equal to or greater than the cohesion of the liquid, a drop of liquid implanted onto the solid spreads spontaneously. That is, the angle between solid and liquid at the solid/liquid/air interface, termed the "contact angle," is zero. If the liquid/solid adhesion is less than the liquid cohesion, an applied liquid droplet does not spread but stands on the surface making a finite contact angle with it (fig. 4). The magnitude of the contact angle increases as the magnitude of the adhesive forces relative to the cohesion of the liquid decreases (Adam 1963). These relationships are expressed algebraically in equation (2), often termed the Young Equation, below:

$$\gamma_S = \gamma_{SL} + \gamma_L \cos\theta \tag{2}$$

where

γ_s = surface tension of the solid

γ_L = surface tension of the liquid

γ_{SL} = liquid/solid interfacial tension

θ = contact angle.

Capillary Pressure and the Kelvin Equations

Wood is a capillary porous medium. The pore structure is defined by (a) the lumina of the cells and (b) the cell wall openings (pits) interconnecting them. The primary routes for liquid penetration into wood are provided by these capillaries (Bailey 1913; Behr et al. 1969; Buro and Buro 1965; Erickson and Balatinez 1964; Petty 1970; Smith and Banks 1971; Wardrop and Davis 1961). Except in the unique case where the contact angle (θ) is equal to 90° ($\cos\theta = 0$), any liquid contained in a cylindrical capillary of uniform bore has a curved surface. The pressure difference (P_c), often called the capillary pressure, across this curved surface is given by the following relationship, derived from the Kelvin equation (Adam 1963):

$$P_c = \frac{-2\gamma_L \cos\theta}{r} \tag{3}$$

where

γ_L = liquid surface tension

θ = liquid/solid contact angle

r = capillary radius

The pressure gradient set up by the pressure difference acts in the sense that liquid is forced into the capillary spontaneously for values of q less than 90°. Conversely, where θ is greater than 90°, external pressure larger than P_c must be applied to force liquid into the capillary. Although wood structure departs significantly from a simple cylindrical capillary model, the general principles of capillary penetration into its structure hold and the magnitude of P_c remains functionally related to the cosine of the contact angle (Adam 1963).

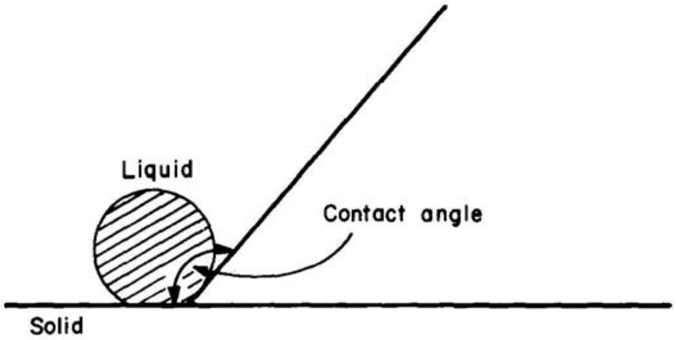

Figure 4.–Contact angle between a liquid drop and a solid surface. (ML85 5008)

In systems involving water as the liquid phase, surfaces forming contact angles less than 90° are said to be hydrophilic, whereas those giving rise to contact angles greater than 90° are said to be hydrophobic or water repellent.

The chemistry of surfaces giving rise to these properties is fairly well understood. Those surfaces presenting polar functional groups, especially those capable of forming hydrogen bonds with water, tend to be hydrophilic. For example, it has been shown that solid stearic acid ($CH_3(CH_2)_{16}COOH$), prepared so that part of the exposed surface consists of carboxyl groups, makes a contact angle with water of about 50° (Adam and Jessop 1925). This relatively low value may result from the availability of carboxyl groups able to form "strong"hydrogen bonds with water, and thus relatively strong adhesion.

In contrast, surfaces consisting of nonpolar moieties, especially methyl groups, tend to be strongly hydrophobic. Where the stearic acid (above) was allowed to solidify from the liquid phase, exposing primarily the nonpolar carbon chains (because long-chain fatty acids tend to solidify in laminae, each layer consisting of two monolayers with the polar (carboxyl ends) sandwiched in the middle of the sheet), the contact angle made with water ranged from 100° to 105° (Adam and Jessop 1925).

From the structural formulas of the linear and branched polymers in the cell wall (fig. 1) it can be seen that these polymers are rich in primary and secondary alcoholic hydroxyl groups, many of them capable of hydrogen bonding with water. Even lignin which is less hydrophilic than the cellulose or hemicellulose has considerable affinity for water (Christensen and Kelsey 1959). A good indication of the strength of the affinity of wood for water is given by the magnitude of the heat of sorption and swelling pressure associated with the interaction (Skaar 1972).

Sorption Isotherms and Fiber Saturation Point

The moisture content (MC) of wood tends toward equilibrium with that of the surrounding atmosphere. Thus for any relative humidity (RH) and air temperature, there is a matching wood equilibrium moisture content (EMC). Moisture sorption isotherms for wood result. These are characteristically sigmoid in form with the EMC corresponding to 100 percent RH ranging from 20 to 50 percent and lying around 28 to 30 percent for many species (Stamm 1964; Feist and Tarkow 1967). At this moisture content (MC = fiber saturation point) the cell wall appears to hold its maximum amount of sorbed water, while the cell lumens remain essentially water-free. Water that is sorbed by the cell wall causes the wood to swell. Further moisture beyond fiber saturation is located within the cell voids held by capillary forces and causes no additional dimensional change. The fiber saturation point of species rich in cell wall extractives occurs at MC's less than 28 to 30 percent, with correspondingly less associated dimensional change. Skaar (1972) fully discussed these effects.

Penetration of Wood by Water

For wood to swell from the dry state, water or some other swelling agent must enter the cell wall. Entry may result from mass flow or diffusion of water vapor into the cell lumens and diffusion from there into the cell wall, or from a diffusion of bound water entirely within the cell wall (Stamm 1959a, 1960a). In most situations, both processes probably occur (Stamm 1960b). Banks (1973) showed that penetration by mass flow followed by diffusion into the cell wall is a much more rapid process than either vapor-phase or bound-water diffusion.

Wood is much more permeable in the longitudinal than in the radial or tangential directions (Banks 1972). Miller and Boxall (1984) showed that, because of this anisotropy, longitudinal flow paths are of major importance in the wetting, in service, of wood exposed to the weather. These workers maintained the end grain surfaces of simulated millwork in contact with a damp substrate. They observed MC to a distance of 120 to 150 ml from such surfaces in excess of fiber saturation after 24 hours exposure.

Physical and Chemical Bonding of Hydrophobes to the Wood Cell Wall

Up to the present, water repellents applied to wood commercially have been almost exclusively of the simple nonchemically bonded type, mainly based on paraffin waxes (Feist 1982; Feist and Mraz 1978a,b; Gray and Wheeler 1959; Stalker 1972; U.S. Department of Agriculture 1969).

These substances are applied, usually by immersion or vacuum impregnation, as solutions in a light organic solvent (National Woodwork Manufacturers' Association 1981a). Formulations contain, in addition to the hydrophobe, fungicidal, resinous, and sometimes insecticidal substances (Feist and Mraz 1978a). The resin probably acts as a binding agent, binding the weakly adhesive hydrophobe to the interfibril spaces at the surface of the cell wall (Borgin and Corbett 1970). A combination of resin and paraffin wax gives a better combination of waterproofing and over-painting properties than either component used alone (Hilditch 1965; Richardson 1965). After impregnation, the carrier solvent is allowed to evaporate leaving the water-repellent substances deposited on the external and on some of the internal surfaces of the treated wood. An idealized model of water-repellent-treated wood is an envelope of cells evenly coated with a hydrophobic layer, surrounding an untreated core (fig. 5); because the surfaces of cells within the treated zone are hydrophobic ($\theta > 90°$), liquid water is unable to penetrate unless an external pressure greater than the capillary pressure P_c is applied. Even if the ideal visualized by this model were achieved, such treatments are unlikely significantly to reduce the rate at which water is sorbed from the vapor phase or to affect the rate of bound-water diffusion through the cell wall, once access to the wall has been gained (Banks 1973). Furthermore, Banks and Carragher (1984) recently demonstrated that in practice the deposit is laid down unevenly because of capillary forces operating during solvent evaporation. Consequently, even within the treated envelope, relatively large areas of the cell walls lack deposit. These water-repellent substances seem to be bonded to the cell wall only by relatively weak Van der Waal forces, a hypothesis supported by the work of Razzaque (1982), who demonstrated that the deposits were removed quantitatively by extraction with petroleum solvents.

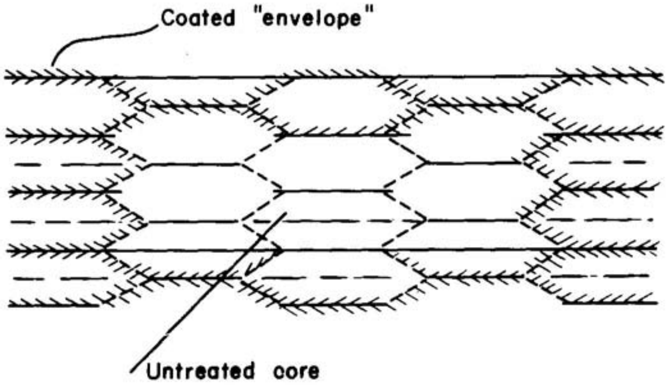

Figure 5.–Hypothetical water-repellent treatment for wood. Hydrophobic layer (envelope) surrounding an untreated core. (ML85 5012)

In spite of these limitations several workers (Banks and Voulgaridis 1980; Feist and Mraz 1978a,b; Purslow and Williams 1978) have shown that treatment of wood with such water repellents gives significant control of water uptake for a reasonable period of time. Work by Banks and Voulgaridis (1980) indicates that the eventual loss of water-repellent effectiveness may be associated with failure of the bond between cell wall and deposit resulting largely from degradation of the wood surface. This idea is supported by Razzaque (1982), who showed that the deposit remained hydrophobic after the treated wood had lost its water repellency. One way to overcome this effect may be to bond the hydrophobe chemically onto the cell wall. A number of reactions are known enabling the bonding of organic substances to wood (Rowell 1975). Among the reagents allowing such reactions, epoxides and isocyanates are particularly interesting since they do not lead to the generation of unwanted byproducts or to serious degradation of the treated wood (Rowel1 1982b). The chemistry of these reagents is discussed later in this paper. Treatment of wood with hydrophobic reagents containing one or other of these functional groups may lead to chemical bonding of the water repellent onto the cell wall. Such techniques have been applied successfully over a considerable period of time in the textile industry (Baird 1963; Mehtra 1975).

Impregnation and Pore Structure of Wood

The pore structure of wood relative to fluid conduction has been studied by microscopic and electron microscopic techniques (Bailey 1913; Bailey and Preston 1969; Bauch et al. 1972; Behr et al. 1969; Comstock 1970; Côté 1963; Wardrop and Davis 1961) and by fluid permeability methods (Bolton and Petty 1977; Comstock and Côté 1968; Petty 1970; Petty and Preston 1969; Smith and Lee 1958; Smith and Banks 1971;). From these studies a generally accepted view of the cellular geometry and the nature of intercellular connections has emerged. In the case of softwoods (gymnosperms), some 95 percent of the volume of wood is occupied by longitudinal tracheids interconnected mainly through their radial walls by bordered pits. The remaining volume is occupied by ray tissue. It can be inferred from microscopic and permeability data that fluids are able to move from tracheid to tracheid, and to some extent between tracheids and rays, via the pit system. The extent to which continuously interconnected pathways exist varies with species (Bramhall 1971; Banks 1970b).

Hardwood (angiosperm) pore structure is considerably more complex. In these species the major longitudinal conducting elements are the vessels and to a lesser extent the rays (Greaves 1974; Smith and Lee 1958). The extent to which mass flow can occur between these cells and surrounding fiber tissue is not generally established.

Impregnation of wood with solvent-borne water-repellent solutions involves flow through the cellular structure discussed briefly above. Enormous variation is observed between and within species in the depth to which fluid penetrates and in the pattern of distribution within the penetrated zone. In permeable softwoods virtually all cells may be impregnated by a solvent-borne system to a considerable depth. In less permeable softwoods, complete cellular penetration may occur to a depth of only a few centimeters longitudinally and 1 or 2 mm laterally and may be restricted beyond this zone to latewood cells only. In impermeable hardwoods, the penetration depth achieved, even by a pressure process, may amount to only a millimeter or so in the fiber direction. However, in permeable hardwoods liquids may easily travel distances of several meters longitudinally and a few centimeters laterally (Redding 1971). It is known that vessels and rays provide the primary flow routes in hardwoods, but the extent to which liquids are able to disperse from these cells into surrounding fibers, by mass flow or diffusion processes, remains unknown. Such redistribution varies from species to species according to Greaves (1974), Greaves and Levy (1978), and Levy and Greaves (1978). In addition to variation between species, marked variation occurs within species especially in association with the structural and chemical differences between heartwood and sapwood (Redding 1971; Smith and Lee 1958).

As a result of this variability, the degree of water-repellent penetration achieved by a given process varies greatly both within and between species. Furthermore, Banks and Carragher (1984) recently showed that the observed variability in cell and intercellular geometry leads to marked solution redistribution during solvent evaporation after treatment. These various distribution factors may strongly influence the performance of water repellents in service.[1]

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[1] Carragher, I. G. Unpublished data. Department of Forestry and Wood Science, University College of North Wales, Bangor; 1983.

Evaluation of Water-Repellent Treatments

Limitations of Water-Repellent Treatment

Water-repellent treatments are applied to wood principally to prevent or reduce the rate of liquid water flow into the cellular structure. Usually treatments involve the deposition of a thin layer of a hydrophobic substance onto external and to some extent internal cell lumen surfaces of wood. As discussed above, we believe that such applied deposits cannot interfere to any significant extent with the movement of water into wood by vapor-phase or bound-water mechanisms. Given time, wood treated with water repellent (i.e., a Type I treatment) and exposed to water will swell to the same extent as untreated wood. EMC remains unaltered. If the wood-water contact angle remains greater than 90°, free water may be excluded from cell lumens for long periods. In practice, however, environments are never perfectly temperature-stable; consequently, once the surrounding tissue is at fiber saturation, water is likely to condense within cell spaces. Moreover, Good and Kotsidas (1979) have shown that the contact angle made by a liquid on a polymer surface decreases as the polymer swells. Banks and Voulgaridis (1980) and Rak (1975) observed this effect in the wood-water system. For these several reasons, we expect that water-repellent-treated wood exposed to liquid water for a prolonged period will not only swell to the same extent as similar but untreated material, but will also accumulate free water in the cell lumens, and therefore eventually attain an MC in excess of fiber saturation.

Water transport into wood occurs rapidly by mass flow, capillarity processes and relatively slowly by diffusion. In service, exposed to weather, wood is subjected to fluctuating wet and dry conditions. Under such conditions, unpainted wood in the absence of any treatment tends to gain and lose water rapidly in sympathy with climatic changes, with associated rapid dimensional change leading to checking and splitting (Banks 1971; Purslow and Williams 1978). Where longitudinal surfaces are painted, water gaining access through untreated end grain (by way of split joints) tends to accumulate (Purslow and Williams 1978), leading to a condition increasingly favoring fungal decay (Carey 1980). Treatment with a water repellent reduces the rate of water uptake by the rapid capillarity process and in turn reduces the magnitude of MC changes incurred during exposure and may reduce the proportion of time that exposed wood is susceptible to fungal decay.

Initial Water-Repellency Determination

Water repellency is related to the tendency of a surface to resist interaction with water, and the contact angle made by water with a surface is the most direct indicator of the resistance to such interaction. Consequently, several workers (Borgin 1961; Gray and Wheeler 1959) have suggested contact angle measurement as a guide to the water repellency of treated wood. More recently, Rak (1975) modified the contact angle technique and examined the rate of change of contact angle with time as a guide to water-repellent effectiveness. Although both the contact angle and its rate of decrease provide good indications of water repellency, the technique suffers a number of disadvantages. Contact angles are usually measured by the “sessile drop” or “tilting plate” method, both described by Adam (1963). The former involves implantation of a small drop of water onto the surface, the contact angle being measured microscopically. In the latter tilting plate method, a flat, treated wood surface is prepared and is immersed in clean water. The surface is then tilted until the position is found at which the wood-water interface is not curved. Both methods make the assumption (unjustifiable for wood) that the prepared surfaces are smooth and flat. In fact, because of its cellular nature, a wood surface can never be smooth, and the anisotropy of wood usually ensures that even a carefully prepared flat surface will become curved to some extent with change in MC. Furthermore, with both techniques a relatively small area of surface is sampled in any test. As a result, many replicate measurements must be made on each wood specimen to obtain a reliable estimate of the average.

In addition to these practical shortcomings, contact angle tests have a more fundamental weakness. Contact angles can be measured only on exposed external surfaces. However, in a capillary porous material such as wood, the nature of the internal pores may well determine the extent to which water is absorbed. Using careful techniques, one can split specimens and then examine microscopically the contact angle made by water with capillary surfaces thus exposed. The process is tedious; it involves destruction of the test specimen, so that changes induced by exposure cannot be followed on a single test piece; to evaluate the water repellency imparted by water-repellent treatment, therefore, most workers have used empirical techniques based on estimation of the decrease in rate of water uptake or cell wall swelling.

Browne and Schwebs (1944) studied several techniques, showing that the water-repellent performance of a series of water repellents was ranked similarly whether judged by water uptake or swelling. At that time (1944) the sole aim of applying water repellents to wood was to enhance dimensional stability, and, for this reason, and because change of dimension is easily automated, Browne and Schwebs favored reduction in swelling rate as the parameter of interest. Nowadays, we believe other factors besides rate of dimensional change may be important in assessing water repellency. For example, although dimensional change is a reasonable guide to the rate of water uptake to fiber saturation, it gives no guide to performance at moisture levels beyond this.

Yet, water repellents may play an important role in reducing the rate of biodegradation (Carey 1980; Feist and Mraz 1978a,b; Verral 1965), presumably by reducing the period of time during which wood is wet enough to sustain attack by microorganisms; premature paint failure has been clearly associated with excessive wetting of the underlying wood (Forest Products Laboratory 1974, Miller and Boxall 1984); bacterial attack is favored by the presence of free water, and a certain amount of free water is necessary for optimal fungal growth.

Whichever method of evaluation is chosen, the comparison of treated with untreated material is based on the change (in dimension or water absorbed) upon exposure to water for a defined time. Water repellency (liquid water) is usually expressed as water-repellent effectiveness (WRE):

$$\mathrm{WRE} = \frac{\mathrm{Dc} - \mathrm{Dt}}{\mathrm{Dc}} \times 100 \qquad (4)$$

where

Dc = swelling (or water uptake) of control during exposure in water for ‘t’ minutes

Dt = swelling (or water uptake) of treated specimen, also for ‘t’ minutes.

As we have emphasized, water repellents usually do not significantly alter the dimensions or water sorption observed at equilibrium. Therefore, the measured WRE varies between 0 and 100 percent depending on the time the test specimens are exposed to water.

For a given exposure time, considerable variation in WRE may result from variations in specimen geometry and in permeability to water of the wood species. In addition, Banks (1971) found the temperature and purity of the water used for the immersion or spray procedure could markedly affect the results obtained. When measuring dimensional change during water immersion, he suggested using as the test parameter the time taken to attain 50 percent of the equilibrium dimensional change (T-1/2). This measure appeared to give wider discrimination between different water repellents and had the advantage that it might be related to the diffusion coefficient for water flowing into wood (Banks 1973).

To ensure good reproducibility of swelling or water uptake tests, conditions must be closely specified and carefully adhered to. Various specifications of such test procedures exist (Banks 1970a; National Woodwork Manufacturers’ Association 198la; Oliver 1963; U.S. Department of Agriculture 1969). These methods are devised to assess the water repellency imparted to freshly treated and conditioned, exposed end-grain surfaces. To ensure maximum susceptibility to water uptake, specimens are prepared from a permeable sapwood with as large a relative end-grain surface as possible. The U.S. Federal Specification (U.S. Department of Agriculture 1969) and U.S. Industrial Standard (National Woodwork Manufacturers’ Association 1981a,b) calls for specimens 40 by 250 mm in cross section, 6 mm long, cut from the sapwood of ponderosa pine. In Britain, the preferred species is Scots pine sapwood and the specified dimensions are 25- by 25-mm cross section, 6.5 mm long, in the fiber direction (Banks 1970a; Oliver 1963). Voulgaridis (1980) has demonstrated that the efficiency of many water repellents applied to these waferlike specimens falls off rapidly with exposure to the weather or to simulated weathering procedures. Voulgaridis and Banks (1983) have shown this very rapid rate of loss of effectiveness to be a relatively superficial effect. The loss of performance which is significant in the thin wafer specimens after a short period of exposure is less significant in larger specimens (geometrically more related to commercially sized wood artifacts) (Feist and Mraz 1978a,b; Miniutti 1961). Thus, we need to take account of the effects of specimen geometry on measurements of water repellency by water uptake or dimensional change. In addition, we must recognize that some water-repellent materials and methods of treatment may be more resistant to weathering than others. Testing the performance of a water repellent only on freshly treated and conditioned specimens may give a misleading guide to long-term performance; therefore, evaluation should include some assessment of the resistance of treatments to weathering, especially to exposure to repeated wetting and drying.

A few workers have evaluated water-repellent performance by field testing, the earliest being Miniutti (1961). Feist and Mraz (1978b) monitored their trials by subjective assessment of appearance and extent of biodegradation, while Purslow and Williams (1978). more objectively, followed MC changes. Results of both suggest that water-repellent treatments retain a protective effect for a period of several years; in the American trial (Feist and Mraz 1978b), the effect persisted for more than 20 years.

Water Soak Tests

As discussed earlier, the test methods most commonly used for evaluating water-repellent treatments are based on the measurement of water uptake or dimensional change of specimens immersed in water. Table 1 compares measurements of water uptake and of swelling, obtained using sapwood wafer specimens 25- by 25-mm cross section, 6.5-mm length (fiber direction). We see that the two techniques rank the treating solutions in similar order of effectiveness. Experience shows this to be generally true in testing resin/wax formulations (Stalker 1972), although the swelling method appears to give better discrimination between solutions of varying effectiveness. Nevertheless, absolute WRE values depend strongly on the test conditions specified. Deviation from such specifications may lead to change not only in the absolute values of water absorption and corresponding swelling test data, but also of their relative values. (Table 5 shows this effect.)

Contact Angle Measurements

We characterize the water repellency of a surface by the contact angle made with water at that surface.

The contact angles reported in table 2 were measured as soon as possible (within a few seconds) after immersion in water. The values are all significantly greater than 90°, indicating highly water-repellent surfaces, and this is reflected in the relatively high WRE values. However, clearly no correlation exists between contact angle and WRE values. The contact angles are closely clustered (130° to 136°), whereas the WRE values show considerable spread (64 to 88 pct).

Because the contact angle made by a liquid on a surface into which it is sorbed decreases with time of contact, initial contact angle is less satisfactory as a guide to water-repellent effectiveness than would be some parameter that took account of initial contact angle and its rate of decrease.

However, data gathered by Voulgaridis (1980) show no clear correlation between water uptake and such transformed contact angle data for freshly treated and conditioned specimens (table 3).

The data of tables 2 and 3 indicate that the hydrophobic effect (contact angle) only partly controls the water resistance of specimens treated with resin/wax; possibly, irrespective of its hydrophobic character, the deposit may also contribute by blocking the pores. Water soak tests may give a better practical guide to performance than those based on contact angle determination.

Table 1.–Comparison of water-repellent effectiveness by water absorption and swelling techniques (30-min immersion in water)

Formulation	Concentration	Water-repellent effectiveness[1]	
		Water absorption	Swelling
	Pct	---------*pct*---------	
Modified resin[2] Paraffin wax	5 0.5	83	74
Modified resin[2] Unrefined wax	5 0.5	20	3
Resin ester[3] Paraffin wax	8 2	85	85

[1] Calculation from equation (4).

[2] Resin specification from Stalker (1972).

[3] Resin specification from Banks, W. B.

Unpublished data. Department of Forestry and Wood Science, University College of North Wales, Bangor; 1969.

Table 2.–Comparison of initial water-repellent effectiveness and contact angle (Voulgaridis 1980)

Treating solution	Concentration	Water-repellent effectiveness	Contact angle[1]
	Pct	*Pct*	*Deg*
Alkyd resin/ Paraffin wax	10 0.5	78	130
Hydrocarbon resin/ Paraffin wax	10 0.5	64	131
Rosin ester/ Paraffin wax	10 0.5	68	136
Coumarone resin/ Paraffin wax	10 0.5	88	132

‘Contact angle measured by “tilting plate” method using transverse wood surfaces.

Table 3.–Comparison of initial water-repellent effectiveness and contact angle ‘θ’ rate of decrease data (Voulgaridis 1980)

Treating solution	Concentration	Water-repellent effectiveness	Time taken for ‘θ’ to fall to 90°
	Pct	*Pct*	*Min*
Alkyd Paraffin wax	10 0.5	78	425
Hydrocarbon resin Paraffin wax	10 0.5	64	416
Rosin ester Paraffin wax	10 0.5	68	460
Coumarone resin Paraffin wax	10 0.5	88	450

Control of Test Conditions

Wood is strongly anisotropic, longitudinal permeability being much greater than lateral, so that the ratio of exposed transverse to longitudinal surface has a marked effect on rate of water sorption and swelling. Stamm (1959a) showed that moisture movement into and along the cell wall is an activated diffusion process. Rate of diffusion into the cell wall and accompanying swelling are therefore temperature dependent. Choong (1965) showed the diffusion coefficient for moisture flow in wood increases with increasing cell wall MC. Furthermore, diffusion rate increases with the magnitude of the moisture gradient across the diffusion substrate (the wood cell wall). For these reasons, the initial MC of the test specimens may be expected to have a marked and complex effect on swelling rate. Tables 4 to 6 clearly illustrate the importance of controlling all these diffusion variables in carrying out water-repellency tests.

Temperature affects WRE values obtained by water absorption but affects results obtained by swelling techniques more, as we might expect, because swelling is an activated diffusion process brought about by water diffusion into the cell wall. Capillarity mechanism, however, is less temperature sensitive and plays a significant part in water uptake, so that at one temperature a given solution may appear more efficient in terms of swelling rate and at some other temperature more efficient in terms of water uptake.

It is generally recommended that prior to testing, specimens should be conditioned at 60 to 65 percent RH and 20 to 25° C. This brings wood to an MC around 12.5 percent. Table 6 shows that in this region the test is fairly sensitive to deviation from the specified MC. Wood exhibits marked sorption hysteresis (Stamm 1964), and the direction from which the conditioned MC is approached (by either adsorption or desorption) has a significant influence on the MC attained at equilibrium and therefore on the estimated WRE value (Banks 1971).

Taken together, these results emphasize the need to work closely to specified test conditions to achieve reliable measurements.

Table 4.–Effect of specimen geometry on measured water repellency[1] (original data)

Specimen length	Ratio of transverse to longitudinal surface area (T/L)	Time to half swell (mean of 5 replicates)
mm		*Min*
3.2	4.0	40
6.4	2.0	108
12.7	1.0	245
25.4	0.5	518

[1] All specimens treated by immersion with same water-repellent solution. Results are expressed as time to attain half maximum swell, because the longer control specimens were still swelling rapidly after 30 min, making precise water-repellent efficiency estimation difficult.

Table 5.–Effect of immersion water temperature on water-repellent effectiveness[1]

Temperature of immersion water	Water absorption (a)	Swelling (b)	(a/b)
°*C*	---------------- *Pct*	----------------	
5	80	85	0.94
15	77	76	1.01
20	74	68	1.09
30	58	35	1.66

[1] Based on wafer specimens, 25- × 25-mm cross section, 6.5-mm fiber direction length. Specimens treated by 30-sec immersion in a common water-repellent solution.

Banks, W. B.; Carragher, J. G. Unpublished data. Department of Forestry and Wood Science, University College of North Wales, Bangor; 1983.

Table 6.–Effect of wood moisture content on water-repellent effectiveness[1] (original data)

Specimen initial moisture content	Water-repellent effectiveness	
	Solution 1	Solution 2
Pct	- - - - - - - - - - *Pct*	- - - - - - - - - -
7	80	76
9.5	82	73
12.5	19	72
16	85	77
20–25	87	86

[1] 30-min water immersion tests using 6.5-mm-length wafer specimens. Solutions 1 and 2 are commercial water repellents of high and medium water-repellent effectiveness, respectively.

Effect of Simulated Weathering on Water-Repellent Effectiveness

With the short (~6-mm length) specimens generally used in water-repellency testing, the effectiveness of treatments is often extremely sensitive to specimen exposure to water. For example, table 7 (Voulgaridis 1980) shows that the effectiveness of 4 solutions, 2 based on resin alone and 2 on a resin/wax blend, decreases very significantly over a period of 40 wet/dry cycles (each cycle consisting of 12 hr water soak at 20 °C, 6 hr drying at 30 °C, 6 hr conditioning at 20 °C, and 65 pct RH).

A similar effect is reflected in the change in contact angle brought about by simulated weathering (table 8) (Voulgaridis 1980).

As a guide to test methodology, these data emphasize the need for care in the handling of test specimens. It is of paramount importance not to allow specimens to get wet prior to testing for true initial performance of wafer specimens. At the same time, the data suggest that with wafer specimens, any initial high value of WRE may be ephemeral. If this test fairly reflects performance loss in practical exposure conditions, standard test procedures may need to be revised.

Table 7.—Effect of simulated weathering on water-repellent effectiveness (Voulgaridis 1980)

Formulation	Concentration	Time to half swell after following 'wet/dry' cycles			
		0	5	20	40
	Pct	*Min*			
Control		2.7	2.2	1.2	0.8
Alkyd resin	10	7.0	3.9	2.0	1.6
Hydrocarbon resin	10	12.5	6.2	3.5	1.8
Alkyd plus paraffin wax	10 0.5	22.4	13.4	5.7	3.7
Hydrocarbon plus paraffin wax	10 0.5	37.1	28.8	13.3	6.9

Table 8.–Effect of simulated weathering on the contact angle of wood surfaces treated with a resin/wax water repellent (Voulgaridis 1980)

Number of wet/dry weathering cycles	Initial contact angle	Time for contact angle to fall to 90°
	Deg	*Min*
0	123	55
10	101	1.5
20	95	0.5
60	84	–

Dimensional Stability

In contrast to water repellency which is a measure of the rate of moisture pickup, dimensional stability is the measure of the extent of swelling resulting from moisture pickup. A variety of terms have been used to describe the degree of dimensional stability given to wood by various treatments. The most common term, antishrink efficiency or ASE, is misleading because it seems to apply to shrinking and not to swelling. The abbreviation is acceptable because ASE might stand also for antiswelling efficiency; this usage requires a statement whether the values were determined during swelling or shrinking, and, in addition, because both are possible, whether they were obtained in liquid water or water vapor.

Changes in wood dimensions as a result of moisture uptake can be measured as a single dimensional component, i.e. tangential, radial, or longitudinal alone, but are usually measured volumetrically taking into account all three dimensional changes. Calculations for dimensional stability are:

$$S = \frac{V_2 - V_1}{V_1} \times 100 \qquad (5)$$

where

S = volumetric swelling coefficient

V_2 = wood volume after humidity conditioning or wetting with water

V_1 = wood volume of ovendried sample before conditioning or wetting

or

$$S = \frac{V_2 - V_1}{V_2} \times 100 \qquad (6)$$

where

S = volumetric swelling or shrinking coefficient.

Then

$$ASE = \frac{S_2 - S_1}{S_1} \times 100 \qquad (7)$$

where

ASE = reduction in swelling or shrinking efficiency resulting from a treatment

S_2 = treated volumetric swelling or shrinking coefficient

S_1 = untreated volumetric swelling or shrinking coefficient.

Test Methods for Dimensional Stability

The test conditions for determining the efficiency of a treatment to reduce dimensional changes depend on the treatment as well as the application of the treated product. For water-leachable treatments, a test method based on water vapor is usually used. Humidity tests are also applied to products intended for indoor use. Nonleachable treatments and products intended for exterior use. are usually tested in liquid water.

For changes in wood dimensions resulting from changes in humidity, the test must be continued long enough to ensure that final equilibrium swelling has been reached.

A typical humidity test is run by ovendrying the specimen, measuring the tangential, radial, and longitudinal dimensions, and calculating the volume. The specimen is then placed in a controlled humidity room and dimensions measured periodically until the specimen reaches equilibrium. Equations (5) and (7) are used to calculate ASE and the test humidity range specified in the results.

This constitutes a single-cycle humidity test. For a series of humidity cycles the specimens are ovendried after the humidity test and shrinkage values calculated. This cycle may be repeated many times to show the efficiency of the treatment when exposed repeatedly to extremes of humidity.

If a liquid water test is used on a leachable chemical treatment, then a single water swelling test may be run. Because the treating chemicals are leached out during this swelling test, however, permitting no further testing of the specimen, leachable treatments are usually tested in a series of humidity tests.

Nonleachable treatments are usually tested for dimensional stability by water soaking. The test consists of determining the ovendry volume of the test specimen, then submerging the specimen in distilled water and removing entrapped air in a vacuum desiccator. Vacuum is continued for 30 minutes, released for 1 hour, reapplied for 30 minutes, then released for 24 hours. Fresh distilled water is then added and this process is continued for 4 to 7 days. The S and ASE are then determined from equations (5) and (7). A series of water soaking and drying cycles give the best indication of the efficiency of a nonleaching treatment (Rowell and Ellis 1978). This repeated water soaking and ovendrying test is very severe and may result in specimen checking and splitting. It should only be used to determine the effectiveness of treatments for outdoor applications under the harshest of conditions.

Since determination of dimensional stability is based on a comparison between an untreated and a treated sample, it is critical that the treated sample come from the same source as the control. The blocks can serve as their own controls if they are subjected to a soaking-drying cycle prior to treatment. The S value for pure southern pine earlywood is 6 to 9, while pure latewood is 17 to 20 (Rowell 1978). The average swelling coefficient for a sample, therefore, depends on the proportion of latewood to earlywood. If a control is used to compare with a treated sample that differs in percent of latewood, then the values obtained for ASE are nearly useless.

Specimen size and geometry are important in dimensional stability tests. If the specimen size is too small, errors in measurement can result in standard deviations so large the ASE values are meaningless. Because the tangential dimension changes the most during swelling and shrinking, a specimen with maximum tangential dimension is best. To increase the rate of swelling and get the specimen to EMC as quickly as possible, specimen must have minimal longitudinal dimension. The actual size of test specimens depends on the maximum capacity of the testing apparatus.

Most test measurements are done with a flatbed micrometer, which makes it important that the grain direction be parallel to one face. If the grain is not parallel, the wood, as it swells in the test, goes out of square, and flatbed caliper measurements on such samples are inaccurate.

Dimensional Stability Treatments

We consider many ways to control dimensional changes in wood, ranging from species and geometry selection, to cross lamination and reducing hygroscopicity, to various chemical methods.

Species Selection

Dimensional changes can be minimized by selection of wood species that have a low coefficient of moisture expansion (Forest Products Laboratory 1974). Many products have been made of certain species to take advantage of a low coefficient of expansion. For example, teak has been used in boatbuilding for many years because the coefficient of moisture expansion for teak is half that of many other species.

Geometry

Because radial swelling and shrinking is 40 to 70 percent less than tangential swelling and shrinking, some dimensional stability can be achieved by minimizing tangential grain direction in a wood product. Quartersawing lumber, for example, produces a board with maximum radial and minimum tangential grain direction. This technique has been applied to products such as flooring and siding (cladding) to minimize expansion and contraction during changes in humidity.

Cross Lamination

Wood swells in water 30 to 100 times more in the radial and tangential directions than in the longitudinal direction. Because of this anisotropy, if wood veneers are glued with grains perpendicular to one another, each ply is mechanically restrained from swelling. This principle has been used for years as the basis of product stability in plywood. Whether the cross-laminated structure is stable to water vapor or liquid uptake depends on the water stability of the glueline. Plywood swells in the thickness direction but is very stable in the two cross-ply longitudinal directions as long as the glueline holds.

Hygroscopicity Reduction

A treatment that reduces the tendency of wood to take on water may result in a reduction not only in the rate but in the extent of water uptake and thus in a reduction in swelling and shrinking of wood.

Optimal treatment of this sort would remove the hydroxyl groups on the cell wall polymers and thus remove the sites for hydrogen bonding to water. Removal of hydroxyl groups is theoretically possible through reductive reactions; however, the treatment would destroy the wood.

Heat treatment reduces the hygroscopicity of wood, Heating wood in the absence of oxygen to temperatures up to 350 °C for a short time results in a 40 percent reduction of swelling in liquid water (Seborg et al. 1953; Stamm et al. 1946a,b). The treated wood, Staybwood, can also be obtained by heating at lower temperatures for longer periods of time. Stamm (1956b) suggests that thermal degradation of the hemicellulose component of the wood cell wall is the mechanism imparting a reduction in hygroscopicity. Hemicellulose, the component most susceptible to thermal degradation, is the most hygroscopic of the cell wall polymers; its degradation products polymerize under heat to produce a water-insoluble polymer. In humidity tests from 30 to 90 percent RH, ASE values as high as 65 percent can be obtained by heating wood at 320 °C for 1 hour (Stamm et al. 1946a,b).

If, along with heat, pressure is applied to the wood, a product known as Staypak is produced. Water absorption is reduced about 60 percent when wood is pressed at 2,000 lb/in.^2 at 300 °C for 5 minutes. The internal stresses of compression are somewhat relieved due to the flow of lignin.

Chemical Treatments

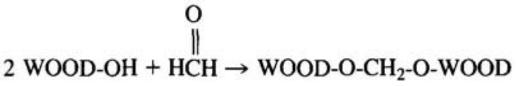
Moisture Uptake Reduction

External and internal coatings, while effective in some cases as water-repellent treatments, give only a small amount of dimensional stability. Dipping wood in a solution of 1 percent wax in a solvent gives it ASE values of 2 to 3 percent. Probably, the wax physically covers some accessible hydroxyl group, excluding moisture penetration.

Treatment of wood with methyl methacrylate followed by heat or radiation polymerization provides a lumen filling or internal coating (Meyer 1977, 1984). Very little chemical enters the cell wall, so the treatment causes little or no wood swelling. Meyer (1977, 1984) achieved polymer loadings over 100 weight percent gain (WPG). WPG is the weight gain due to chemical addition determined from the original ovendried weight of the specimen. Because the lumen structure is filled, the rate of water uptake is greatly reduced.[2] Some dimensional stability is also achieved because some water-accessible sites have been blocked. ASE values of 10 to 20 percent have been reported (Rosen 1976; Siau and Meyer 1966).

Chemical Crosslinking

If structural units of the wood cell wall are chemically bound together (crosslinked), the bonds restrain the units from swelling when moisture is present much as the physical bonds of cross lamination do in plywood. One of the most widely studied chemical systems for crosslinking is the reaction between wood cell wall hydroxyls and formaldehyde (Rowell 1978):



$$2\ \text{WOOD-OH} + \text{H}\overset{\overset{\displaystyle O}{\|}}{\text{C}}\text{H} \rightarrow \text{WOOD-O-CH}_2\text{-O-WOOD}$$

Crosslinking can take place between hydroxyl groups on the same or different cellulose, hemicellulose, and lignin polymers. The reaction is usually catalyzed by strong acids.

Low levels of formaldehyde cross linking provide very high ASE values. Tarkow and Stamm (1953) obtained ASE value in liquid water at 3.1 WPG an ASE of 47 percent, at 4.1 WPG 55 percent, at 5.5 WPG 60 percent, and at 7 WPG an ASE value of 90 percent (see also Stamm 1959b).

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[2] Rowell, R. M.; Ritter, L. A. Unpublished data. Madison, WI: U.S. Department of Agriculture, Forest Service, Forest Products Laboratory; 1982.

Bulking Treatments

The mechanism by which bulking improves dimensional stability is by occupying accessible sites to the exclusion of water.

By far the greatest amount of research for dimensional stability of wood has been carried out in the area of cell wall bulking treatments (Rowell 1984). If chemicals enter the cell wall, their volume fills the space that water would occupy if the wood absorbed water. In order to occupy all the sites accessible to water, the cell wall treating chemical must have a molecule as small as water and be able to swell as much as water. Rowell and Ellis (1978) showed that cell wall treatment causes an increase in wood volume directly proportional to the theoretical volume of chemical added. With increasing added chemical the volume of wood increases to about 25 WPG, at which point the treated volume is approximately equal to green volume (Rowell et al. 1976). When this bulked wood comes into contact with water, very little additional swelling can take place.

We consider three classes of bulking treatments: nonbonded and leachable; nonbonded and nonleachable; and bonded and nonleachable.

Nonbonded-leachable.– If wood is soaked in aqueous solutions of salts or sugars and then dried, the solutes are deposited in the cell wall. Solutions of manganese, sodium, barium, magnesium, and lithium chloride, potassium thiocyanate and potassium iodide swell wood beyond the water-swollen size especially above pH 8 (Stamm 1934). Treating wood with sucrose or invert sugar (glucose-fructose mixture) gives ASE values up to 70 percent (swelling test in water vapor) (Stamm 1937). Both the salts and sugars are very soluble in water and are easily leached if the treated wood comes into contact with water. These chemicals also make the wood even more hygroscopic, so the wood is usually finished with two coats of varnish to seal the chemical in the wood.

Another nonbonded-leachable treatment utilizes polyethylene glycol (PEG) (Stamm 1956a, 1959c). In this case, the wood is usually treated in a green condition and the PEG is exchanged for the cell wall water. The treatment most often used is a 30 percent by weight solution of PEG-1000. The green wood is soaked in this solution for a length of time depending upon the thickness of the sample. This treatment is also hygroscopic, and two coats of polyurethane varnish are usually applied to help seal in the PEG and help seal out water. Maximum ASE values in swelling tests in water vapor of 82 percent are achieved at PEG loadings of 45 WPG. The addition of a water-soluble phenolic resin to the PEG solution can improve the ASE value (Stamm 1956a).

Nonbonded-nonleachable.–Treatment of wood with aqueous solutions of phenol-formaldehyde resin-forming compounds gives a bulked product where the chemicals are not attached or bonded to the cell wall components but form insoluble polymers which will not leach out in water. Usually thin veneers are treated to 25 to 35 percent content of resin-forming materials on the basis of the weight of dry untreated wood. After uniform diffusion of the resin into the cell wall structure, the wood is partially dried and the resin is then cured at about 150 °C for 30 minutes (Stamm and Seborg 1943). The resulting cured product, commonly termed “impreg,” can then be laminated with practically all kinds of adhesives; however, water-insoluble types are usually used. Thin veneers are treated because the treating time varies directly with the square of the thickness. Green veneer, 1/32 inch thick, can be treated by soaking 1 to 2 hours, whereas green veneer, 1/8 inch thick, requires about a day. At weight gains of 25 to 40 percent resin, the treated impreg will have an ASE of 60 to 70 percent in both liquid and water vapor (Stamm and Seborg 1943, 1936). Impreg has been used for pattern wood dies because of its dimensional stability (Seborg and Vallier 1954).

When phenol-resin-treated wood is highly compressed during curing of the resin, a product commonly known as “compreg” is made. Compreg with a specific gravity of 1.3 to 1.4 containing 30 percent phenol-formaldehyde resin is achieved by applying pressures of 1,000 to 1,200 lb/in.^{2} and a temperature of 150 “C (Seborg and Stamm 1945; Stamm and Seborg 1944).

In a 24-hour water-soaking dimensional-stability test, compreg has an ASE of about 95 percent. The rate of water pickup is so slow that complete swelling equilibrium of a 112-inch sample is not complete in a year at room temperature (Stamm 1948). For this reason, results from short-term low-temperature water-soaking test data have little meaning for compreg.

If styrene or methyl methacrylate is impregnated into wood using a swelling solvent system instead of the nonswelling system described earlier, the monomers do enter the cell wall. Polymerization by radiation gives a wood polymer composite with grafted copolymers bulking the cell wall (Laidlaw et al. 1967). Using either dioxane-water or methanol-water as solvent, Ellwood et al. (1972) obtained maximum ASE values of 70 percent in a 90 percent RH test at polymer loadings of 80 to 100 WPG. Using a mixture of styrene (60 pct) and acrylonitrile (40 pct) at 30 WPG they found maximum ASE values of about 60 percent in liquid water tests and 50 in humidity tests at 85 percent RH.

One interesting nonbonded-nonleachable treatment that has received little recent attention is to replace cell wall water by waxes. In a two-phase process, Stamm and Hansen (1935) removed water from a thin green wood sample by boiling the sample in an organic solvent that had a boiling point higher than that of water. They used ethylene glycol monoethyl ether (Cellosolve) which has a boiling point of 135 “C and is soluble in water. The glycol replaced the water in the wood with little change in wood volume. They then replaced the glycol in the wood by a molten wax whose melting point was higher than the boiling point of Cellosolve. They found that because of its molecular size, the wax cannot occupy all the sites of potential interaction between water and cell wall polymers, but it may replace up to 80 percent of the original bound water. Some part of the wax ends up in the lumen structure, so they used much more wax than the amount needed to replace cell wall water. After one wax boiling, they achieved reductions in swelling of about 30 percent in humidity tests at 90 percent RH, and, by increasing the number of wax distillations to nine, obtained ASE values up to 80 percent. However, because the process is used with thin veneers and it interferes with gluing and finishing, its commercialization was not undertaken.

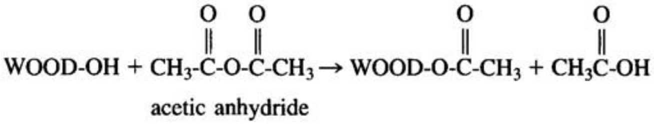
Bonded-nonleachable. – Through chemical reactions it is possible to add an organic chemical to the hydroxyl groups on wood cell wall components. This type of treatment may reduce the hygroscopicity of the wood as described earlier and also bulk the cell wall with a permanently bonded chemical.

Many reactive chemicals have been used to chemically modify wood (Rowell 1975, 1984). For best results chemicals used should be capable of reacting with wood hydroxyls under neutral or mildly alkaline conditions at temperatures at or below 120 °C. The chemical system should be simple and must be capable of swelling the wood structure to facilitate penetration. The complete molecule should react quickly with wood components yielding stable chemical bonds and the treated wood must still possess the desirable properties of untreated wood.

Chemicals used to react with wood cell wall hydroxyl groups can be divided into two classes: those that react with a single hydroxyl group (single-site addition) and those that react with a hydroxyl group and then polymerize (polymerizing addition).

Single-Site Addition

Acetylation.– The most studied of all the chemical modification treatments for wood has been acetylation.



$$\text{WOOD-OH} + \underbrace{\text{CH}_3\text{-}\overset{\text{O}}{\overset{\|}{\text{C}}}\text{-O-}\overset{\text{O}}{\overset{\|}{\text{C}}}\text{-CH}_3}_{\text{acetic anhydride}} \rightarrow \text{WOOD-O-}\overset{\text{O}}{\overset{\|}{\text{C}}}\text{-CH}_3 + \text{CH}_3\overset{\text{O}}{\overset{\|}{\text{C}}}\text{-OH}$$

The best acetylation condition is uncatalyzed acetic anhydride in xylene at 100 to 130 °C (Goldstein et al. 1961).

Acetylation to 20 to 25 WPG shows an ASE of 70 percent in liquid water swelling tests (Koppers Acetylated Wood 1961; Rowell 1982a; Tarkow et al. 1950). Southern pine weathered for 12 months shows a slight decrease in acetyl content, but a reduction in ASE from 78 to 64 percent (Koppers Acetylated Wood 1961). The anhydride reaction produces acetic acid as a byproduct during acetylation which results in a 50 percent loss of reagent. Since the reaction is a single hydroxyl site modification, maximum acetyl substitution is achieved at about 25 WPG. Longer reaction times do not increase the level of modification.

Other anhydrides have been reacted with wood. Propionic and butyric anhydrides in xylene without catalyst react with wood, but at a slower rate than acetic anhydride (Goldstein et al. 1961). Trifluoroacetic anhydride has also been used in esterification, but was found to result in no more than surface modification (Ami et al. 1961). Phthalic anhydride in dimethylformamide gives an initial ASE of 75 percent, but on water soaking the ASE drops to 50 percent (Risi and Arseneau 1958).

Methylation.– Reaction of wood with dimethyl sulfate or methyl iodide results in methylation of the cell wall polymers (Rudkin 1950).

$$\text{WOOD-OH} + \underbrace{\text{CH}_3\text{I}}_{\substack{\text{methyl}\\\text{iodide}}} \rightarrow \text{WOOD-OCH}_3 + \text{HI}$$

A single methylation results in a low WPG of added methoxyl groups, but repeated methylation can result in WPG of up to 20. At WPG of 17 to 20, ASE values of 55 to 60 percent are achieved.

Alkyl chlorides.–In the reaction of alkyl chlorides with wood, hydrochloric acid is formed as a byproduct. Because of this, a great deal of wood degradation takes place during the reaction.

$$\text{WOOD-OH} + \underbrace{\text{R-Cl}}_{\substack{\text{alkyl}\\\text{chloride}}} \rightarrow \text{WOOD-O-R} + \text{HCl}$$

Reaction of wood with an alkyl chloride in pyridine (Kenaga et al. 1950; Kenaga and Sproull 1951) or with aluminum chloride gives a high initial ASE but, on drying and resoaking, the effects of alkylation are lost (Risi and Arseneau 1957a). In the alkyl chloride-pyridine case, the ASE is not caused by the formation of alkyl ethers in cellulose or lignin, but by the formation of alkyl pyridinium-chloride polymers which have the effect of bulking, but, being water soluble, are easily leached out.

Risi and Arseneau (1957b) studied other alkyl chlorides, namely, crotyl chloride and n- and t-butyl chlorides (1957c) catalyzed with pyridine. These treatments produced, again, only temporary ASE along with severe degradation caused by the liberated hydrochloric acid.

Aldehydes. — Acetaldehyde (Tarkow and Stamm 1953) and benzaldehyde (Tarkow and Stamm 1953; Weaver et al. 1960) have been reacted with wood using either nitric acid or zinc chloride catalysts. Good ASE was achieved with acetaldehyde, but an ASE of only 40 percent with benzaldehyde.

Difunctional aldehydes (dialdehydes) have been tried with zinc chloride, magnesium chloride, phenyl dimethylammonium chloride, and pyridinium chloride as catalysts (Weaver et al. 1960). Glyoxal, glutaraldehyde, and α-hydroxyadipaldehyde all show ASE of 40 percent with WPG of 15 and the highest ASE (50 pct) at 20 pct WPG. With these three compounds, crosslinking is a possible cause of ASE, as in the reaction with formaldehyde; however, with the low ASE observed at high WPG, it is clear that bulking is the mechanism for the ASE achieved.

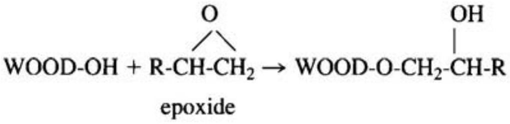
Chloral (trichloroacetaldehyde) with no catalyst gives a 60 percent ASE at 30 WPG (Weaver et al. 1960). After 15 weeks at 70 percent RH, however, all weight gain is lost as well as the ASE. This shows a very unstable, perhaps reversible, bond formation.

Phthalaldehydic acid in acetone catalyzed with p-toluenesulfonic acid gives ASE of 40 percent at WPG of 34 (Weaver et al. 1960). Kenaga (1957) claims a 50 to 70 percent ASE when phthalaldehydic acid or its derivatives are cured at 100 °C uncatalyzed for 16 to 24 hours.

Other aldehydes and related compounds have been tried either alone or acid catalyzed (Weaver et al. 1960). Compounds such as N,N'-dimethylolethylene urea, glycol acetate, acrolein, chloroacetaldehyde, heptaldehyde, o- and p-chlorobenzaldehyde, furfural, p-hydroxybenzaldehyde, and m-nitrobenzaldehyde all achieve ASE by a bulking mechanism and not by low-level crosslinking. At WPG of 15 to 25, the highest ASE reported is 40 percent.

Polymerizing Addition

Epoxides.– The reaction between epoxides and wood can be catalyzed under mildly basic conditions (Rowell and Gutzmer 1975).

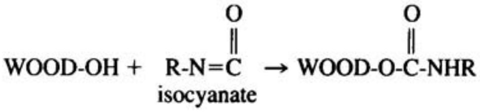


$$\text{WOOD-OH} + \underset{\text{epoxide}}{\text{R-}\overset{\text{O}}{\overset{/\backslash}{\text{CH-CH}_2}}} \rightarrow \text{WOOD-O-CH}_2\text{-}\overset{\text{OH}}{\overset{|}{\text{CH}}}\text{-R}$$

A new hydroxyl group is formed during the reaction which can then react with another reagent epoxide group and polymerization can occur. Because of the ionic nature of the reaction and the availability of alkoxyl ions in the wood components, chain transfer probably results. The chain length of the polymer is probably short as a result of chain transfer. The simplest epoxide, ethylene oxide, catalyzed with trimethylamine has been used as a vapor-phase treatment. At WPG of 20, McMillin (1963) recorded a 60 percent ASE in swelling tests in liquid water. Liu and McMillin (1965) claimed an ASE of 82 percent with a WPG of 10 for the same process or with propylene oxide. Under similar conditions, Aktiebolag (1965) reported a WPG of 22 gave less than 1 percent tangential and radial shrinkage. Using an oscillating pressure rather than a constant pressure system with ethylene oxide and trimethylamine, Barnes et al. (1969) found an ASE of 42 in swelling tests in liquid water for WPG of 11. Reactions of wood with propylene oxide, butylene oxide, and epichlorohydrin catalyzed with triethylamine show ASE of 70 percent at WPG of 22 to 25 (Rowell and Gutzmer 1975).

As WPG increases, the ASE value increases up to about 35 WPG (fig. 6). At about 35 WPG, the ASE starts to drop (Rowell et al. 1976). Above this WPG the volume of added chemical becomes great enough to rupture the cell wall. Electron micrographs of wood clearly show the splits in the cell wall at high chemical loadings (Rowell et al. 1976). The epoxides react and swell the cell wall and continue to do so until the cell wall must rupture to accommodate more reacting chemical. Water can then interact with newly exposed ruptured cell wall component hydroxyl groups causing the wood to superswell above green volume causing the ASE to drop.

Isocyanates.– In the reaction of wood hydroxyls with isocyanates, a nitrogen-containing ester is formed.



$$\text{WOOD-OH} + \underset{\text{isocyanate}}{\text{R-N=}\overset{\text{O}}{\overset{\|}{\text{C}}}} \rightarrow \text{WOOD-O-}\overset{\text{O}}{\overset{\|}{\text{C}}}\text{-NHR}$$

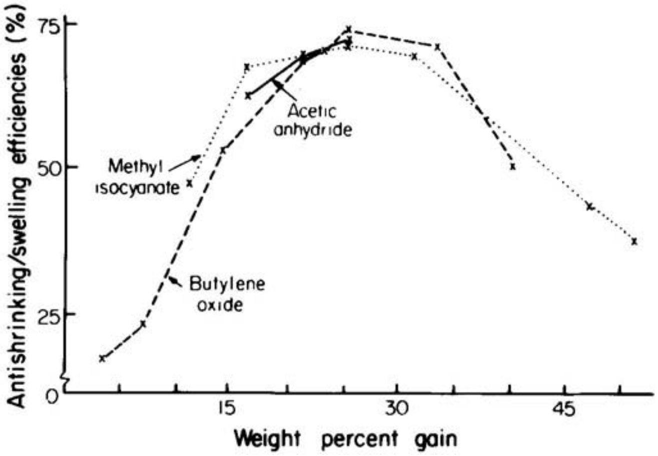


Figure 6.–Dimensional stabilization of wood as a function of bonded chemical weight gain. (ML85 5010)

An example of this type is the reaction of wood with phenylisocyanate in dimethylfonnamide (DMF) (Baird 1969). Veneers impregnated with DMF and exposed to vapors of phenylisocyanate gave a product with an ASE of 77 percent. Reaction of wood with ethyl-, n-butyl-, and t-butyl isocyanates in DMF gave an ASE of 67 percent at 31 WPG (Clermont and Bender 1957). A polymerization reaction was observed only when the reaction took place in DMF.

Methyl isocyanate reacts with wood without a catalyst (Rowell and Ellis 1979). As with the epoxides, as the WPG increases so does the ASE up to a maximum of about 35 WPG and 70 percent ASE in liquid water tests for swelling (fig. 6). Above this level of chemical bonding, rupturing of the cell wall takes place causing the ASE to fall (Rowell and Ellis 1979).

Acrylonitrile.– When acrylonitrile is reacted with wood in the presence of an alkaline catalyst, cyanoethylation occurs.

$$\text{WOOD-OH} + \underset{\text{acrylonitrile}}{\text{CH}_2\text{=CH-CN}} \rightarrow \text{WOOD-O-CH}_2\text{CH}_2\text{CN}$$

With sodium hydroxide catalyst WPG's up to 30 have been achieved. At this level, the wood has an ASE in liquid water of 60 percent.[3] Cyanoethylation using ammonium hydroxide as a catalyst gives an ASE of 80 percent at a WPG of 26. Hot water extraction of this treated wood, however, leaches out all of the chemical and the dimensional stability is lost (Baechler 1959). This outcome may result from the reaction of acrylonitrile with the ammonia catalyst forming water-soluble polymers in the cell wall.

Reacting wood with 25 percent acrylonitrile in methanol followed by exposure to 10^7 rads of ionizing radiation gives an ASE of only 40 percent at a WPG of 29 (Kenaga 1963). The low value of this ASE may be caused by the acrylonitrile reacting with the methanol and forming polymers in the lumen rather than in the cell wall.

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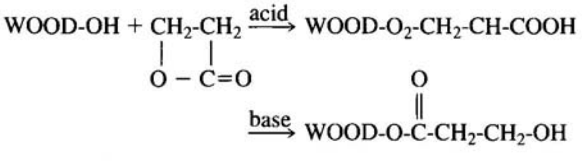
[3] Ellis, W. D.; Rowell, R. M. Unpublished data. Madison, WI: U.S. Department of Agriculture, Forest Service, Forest Products Laboratory; 1980.

β-Propiolactone.– The reaction of β-propiolactone with wood is interesting in that different products are possible depending on the pH of the reaction. Under acid conditions, an ether bond is established with the hydroxyl group along with the formation of a free acid end group. Under basic conditions, an ester bond is formed with the hydroxyl giving a primary alcohol end group.



$$\text{WOOD-OH} + \underset{\text{O}-\text{C=O}}{\text{CH}_2\text{-CH}_2} \xrightarrow{\text{acid}} \text{WOOD-O}_2\text{-CH}_2\text{-CH-COOH}$$

$$\xrightarrow{\text{base}} \text{WOOD-O-}\overset{\overset{\text{O}}{\|}}{\text{C}}\text{-CH}_2\text{-CH}_2\text{-OH}$$

β-propiolactone

Southern pine wood has a pH of approximately 5, and uncatalyzed b-propiolactone reacts with this wood to give a carboxyethyl derivative (Goldstein et al. 1959). High concentrations of b-propiolactone cause a very high degree of swelling, resulting in delamination and splitting. At a 30 WPG, the treated wood has an ASE of 60 percent in a water swelling test (Goldstein 1960).

Evaluation of Dimensional Stability Treatments

Rowell and Ellis (1978) used multicycle water-swelling tests to compare the effectiveness of chemical treatments with propylene oxide, butylene oxide, acetic anhydride, acrylonitrile and methyl isocyanate. Table 9 shows the results of a double cycle (OD to wet to OD to wet to OD) water leaching and swelling test. Acetylation with acetic anhydride gives the most consistent results. In this case, ASE remains constant during two soaking/drying cycles, indicating strong bonding with little or no loss of chemical during the leaching cycles. Propylene and butylene oxides and methyl isocyanate specimens show an ASE of about 70 percent on the first wetting cycle. Because of a small loss of chemical upon leaching, ASE drops during the second wetting cycle. The ASE value calculated from the first wet volume to the second ovendry volume is almost always lower than any subsequent ASE values. The reason for this is simple. The new ovendry volume is smaller than the original ovendry volume because chemical that had bulked the original specimens has been leached out during the wetting cycle. In the case of acrylonitrile, ASE value for the first wetting cycle is high, but upon redrying, the ASE value is negative. We infer that the bulking chemical in the original ovendry sample has now been leached out, and the new ovendry volume is close to or smaller than the volume of the ovendry sample before chemical modification. The negative ASE value probably results from the degradative effects of the catalyst and heat on cell wall components. In almost all tests described for water swelling, a soaking time of 1 week is used. Table 9 refers to test specimens of a size, 20 by 20 by 8 mm (radial x tangential x longitudinal), permitting easy access of the water into the interior of the specimens. Within 24 hours, the volumetric swelling coefficient reaches, within experimental error, the value it will have after 7 days (table 10). Although soaking beyond 24 hours does not increase the swelling coefficient, it does influence the amount of material leached out.

Table 9.–Volumetric swelling and shrinking coefficients (S) and antishrink/antiswell efficiency (ASE) as determined by the water-soaking method (Rowell and Ellis 1978)

Treatment	WPG[1]	S_1[2]	ASE_1[3]	S_2[4]	ASE_2[5]	Weight loss[6]	S_3[7]	ASE_3[8]	S_4[9]	ASE_4[10]	Additional weight loss[11]
						Pct					*Pct*
Propylene oxide	0	15.8	–	15.8	–	<0.6	15.9	–	15.9	–	2.3
	29.2	6.0	62.0	9.0	43.8	0.4	7.8	50.9	7.9	50.3	1.7
Butylene oxide	0	13.6	–	12.4	–	< .5	12.4	–	12.9	–	2.4
	26.7	3.5	74.3	5.5	55.6	1.6	5.0	59.7	5.4	48.1	3.0
	27.0	3.6	73.5	5.7	54.0	1.6	5.2	58.1	5.6	56.6	2.6
Acetic anhydride	0	13.8	–	13.3	–	<.2	13.6	–	13.3	–	<.2
	16.3	5.1	63.0	5.1	61.7	<.2	5.3	61.0	5.3	60.2	<.2
	21.1	4.2	69.6	4.1	69.2	<.2	4.3	68.4	4.6	65.4	<.2
	22.5	4.1	70.3	3.8	71.4	<.2	4.0	70.6	4.1	69.2	<.2
Acrylonitrile	0	14.1	–	13.9	–	<.5	14.0	–			<.2
NH_4OH	26.1	2.7	80.9	15.3	negative	21.7	14.4	negative			.9
NaOH	0	20.3	–	16.8	–	<.2	16.7	–			<.2
	25.7	10.5	48.3	18.8	negative	13.5	17.5	negative			1.2
Methyl isocyanate	0	14.0	–	13.8	–	<.5	13.7	–	13.5	–	4.5
	10.9	7.2	48.3	8.1	40.4	<.2	8.3	39.4	8.9	34.1	4.5
	21.6	5.5	60.4	6.6	52.0	<.2	6.5	52.6	6.3	53.3	3.9
	26.0	4.2	69.7	5.1	62.8	<.2	4.8	65.0	5.3	60.7	4.3
	29.9	4.7	66.4	6.0	56.8	1.6	4.8	65.0	5.3	60.7	4.1
	41.5	3.2	77.1	8.1	41.3	9.7	8.0	41.6	7.6	43.7	1.4

[1] Weight percent gain. Samples recorded at 0 WPG are controls.

[2] Volumetric swelling coefficient determined from initial ovendry (OD) volume and first waterswollen volume, equation (S_1).

[3] Antiswell efficiency based on S_1, equation (6).

[4] Determined from first waterswollen volume and reovendrying.

[5] Antishrink efficiency based on S_2.

[6] Percent weight loss based on the difference between initial OD weight and OD weight after first soaking.

[7] Determined from reovendry volume and second waterswollen volume

[8] Antiswell efficiency based on S_3.

[9] Determined from second waterswollen volume and second reovendrying.

[10] Antishrink efficiency based on S_4.

[11] Additionalweight loss based on OD weight

Table 10.–Rate of liquid water swelling of southern pine at 22°C (Rowell and Ellis 1978)

Treatment	Weight percent gain	Volumetric swelling coefficient, S			
		1 day	2 days	4 days	7 days
Control	0	18.9	18.9	19.0	19.0
	0	15.8	15.8	15.8	15.8
Acetic anhydride	21.1	4.1	4.1	4.2	4.1
Propylene oxide	29.2	6.1	6.1	6.0	6.0

Water Repellency of Some Dimensional Stabilization Treatments

Up to this point, we have considered evaluations of treatment to increase either water repellency or dimensional stability. This section deals with evaluation of treatments that might be considered both water repellents and dimensional stabilizers.

Typical water-repellency tests have been carried out on wafer specimens (20- × 20-mm cross section, 5 mm longitudinal) treated by a dimensional stabilizing technique. They are included here as a guide to the water-repellent behavior apparently imparted by treatments intended for dimensional stability. Performance varies with species and with variation in treatment schedule. Additionally, performance depends to a large extent on the detail of the test applied to the treated specimen. The treatments evaluated are:

(1) Lumen fill–methyl methacrylate

(2) Nonbonded-leachable — PEG

(3) Bonded, bulking — butylene oxide and acetylation

The authors have evaluated these treatments by the 30-minute swelling test, in which water absorption and contact angle (transverse surface) were measured after 30 minutes soak. The swelling tests were carried out twice, before simulated weathering and after water soak for 24 hours followed by drying at 40 to 50 °C and reconditioning at 65 percent RH, 20 °C. Contact angles were measured by the sessile drop method (described earlier) on specimens conditioned at 65 percent RH, 20 °C following the swelling and water sorption tests. Data deriving from the various test procedures are summarized in table 11.

The authors have tested the several treatments for both water repellency and dimensional stability. Each treatment in table 12 was carried out under one set of conditions on a single wood species. We ran the tests in triplicate, and each cited value is a mean of the three separate determinations. They are presented to illustrate distinctions between dimensional stabilization and water-repellency, characteristic of the various treatments discussed. Although methyl methacrylate (lumen fill) impregnation is often regarded as a stabilization treatment, the small wafer test shows it to perform well neither as a dimensional stabilization nor as a water-repellent treatment. With larger pieces of wood, we would expect the lumen plugging effect to slow the rate of water uptake and show water repellency.

The PEG-treated specimens initially performed well. They were dimensionally quite stable, and this stability apparently led also to a high level of water-repellent effectiveness (WRE), measured by the swelling technique. After a fairly mild weathering treatment (24-hr water soak at 25 °C, followed by drying at 40 to 50 °C), however, effectiveness decreased very significantly. This falloff in performance resulted from loss of PEG (which does not bond chemically to the wood) by water leaching. The WRE assessed by water uptake was nonexistent; even before leaching, this technique probably has a low water-repellency value, because the impregnant material (polyethylene glycol) is itself hygrophilic.

Both butylene oxide and acetylation treatments gave effective dimensional stability. In contrast to the PEG treatment, because these stabilizing agents are chemically bonded to the cell wall, their performance is little affected by the leaching/weathering treatment. WRE appears high when measured by the swelling technique. This water-repellent effectiveness occurs probably because the cell wall is significantly stabilized, however, rather than because the treatments slow down capillary penetration to any large extent.

This explanation is supported by the water-uptake/water-repellency test data, which show both treatments to perform badly, giving WRE values of only 20 to 25 percent.

Finally, table 12 shows that none of the stabilization treatments tested imparts any lasting hydrophobic character to the treated wood surfaces. The contact angles measured for these surfaces are never higher than the untreated controls, and in all cases the implanted drops spread (i.e. contact angle = 0°) within 5 minutes.

Table 11.–Water-repellent performance of stabilization treatments (original data)

Treatment	Water-repellent efficiency			Contact angle (after weathering)	
	Swell before weathering	Swell after weathering	Water uptake (after weathering)	(1)	(2)
	Pct			*Deg*	
Untreated				125	70
Methyl methacrylate	15	35	23	95	60
Polyethylene glycol	73	8.5	0	115	100
Butylene oxide	52	62	25	0*	0*
Acetylated	70	76	20	120	100

[1] Measured immediately after drop implantation.

[2] Measured 1 min after drop implantation.

*Drop spread almost instantaneously.

Table 12.–Tests for both water repellency and dimensional stability from a single treatment (original data)

Treatment	Antishrink[1] efficiency		Water-repellent[2] effectiveness			Contact angle	
	1	2	Swell (1)	Swell (2)	Water uptake (3)	Initial	After 5 minutes
	Pct		*Pct*			*Deg*	
Untreated							
Methyl methacrylate	2	2	18	25	23	95	0
Polyethylene glycol	70	20	73	10	0	115	0
Butylene oxide	75	74	52	62	25	10	0
Acetylated	75	70	70	76	20	120	0

[1] After 1 and 2 water-soaking tests.

[2] (1) determined by water swelling, (2) same test after weathering, (3) determined by water uptake.

Conclusions and Recommendations for Future Research

Two basic types of treatment protect wood from the effects of moisture: One reduces the rate of water vapor or liquid water absorption but does not reduce the extent of swelling to any great degree; the other reduces the extent of swelling and may or may not reduce the rate of water absorption.

The effectiveness of a treatment in reducing the rate of moisture absorption is expressed as water repellency, while effectiveness in reducing the extent of swelling is expressed as antiswell or antishrink efficiency. Four criteria guide the selection of treatment for either: environment of the product, type of protection needed, degree of protection needed, and cost.

If a product is to be used outside in contact with liquid water and requires a high degree of dimensional stability, a nonleachable, perhaps even bonded, treatment will be needed. The cost of such a treatment may be quite high. If, however, the product will be subjected to changes in RH in an indoor environment, a leachable treatment for dimensional stability might be satisfactory at a much lower price. If very rigid tolerances are required in a product–as in pattern wood dies–a treatment with very high ASE value is needed. If only a moderate degree of dimensional stability is satisfactory, for such products as painted millwork, a less rigorous treatment will suffice.

If a product needs to repel liquid water or slow the rate of absorption, then a water-repellent treatment will suffice. The millwork industry, for example, uses a dip treatment in a solution containing a small amount of wax to achieve a moderate degree of water repellency. They would, no doubt, like a higher degree of water repellency or even dimensional stability, but only if the extra cost were recoverable in the marketplace. In contrast, musical instrument makers require a very high degree of water repellency or dimensional stability, and the value of the final instrument may be able to absorb a high cost to accomplish the desired result.

We offer several general recommendations for research in the area of water repellency and dimensional stability of wood.

(1) Research in the fundamental principles of wood-moisture relationships. This includes the role of each cell wall component in moisture uptake and movement, hydrophilic and hydrophobic nature of the wood microstructure, mechanism of adsorption and absorption of moisture, moisture movement in refractory species, and contribution of extractives. A breakthrough in moisture control in wood may not come until basic research has been completed and the results are fully understood.

(2) Research on stabilizing reconstituted wood products such as fiberboard, ,particleboard, flakeboard, and chipboard. These products account for an increasing proportion of the market, and very little is known about controlling moisture uptake in these products. Processes responsible for thickness swelling and shrinking in these products need study, along with the occurrence of irreversible changes. With fiberboards, different approaches than serve for solid wood may be needed to achieve a high degree of water repellency and/or dimensional stability.

(3) Research on the dimensional stability of new and different wood species coming into the market. Very little research has been done in dimensional stability of tropical woods, hardwoods, and second-growth softwoods in either solid or reconstituted wood products. Especially in respect of the tropical woods, the effects on dimensional stability of growth patterns, density, extractives, wax, and inorganic content need to be determined so that these resources can be utilized to their highest potential.

(4) Finally, a definitive market evaluation of the potential use of water-repellent-treated and dimensional-stabilized wood is needed. Wood is renewable and lower in processing energy cost than plastics, metals, or ceramics, and, if more data were available on cost effectiveness, might find new (or reestablish old) markets, as competing materials become more costly. There may be some very large markets for wood with a moderate degree of water repellency or dimensional stability, but they have not yet been given proper consideration.

In addition to these studies of the woody substrate, investigations of ways to modify wood need to be undertaken. Considerable research effort is expended to evaluate the performance of improved resin systems applied to wood. When resin systems fail, degradation of the wood surface or the wood-coating interface is usually involved in addition to any breakdown in the applied material. Although new and perhaps more effective resins or coating systems may be found, this line of research is more likely to lead to an improvement in moisture control than to fundamental change leading to a totally new technology.

New technology in water repellency is more likely to come from studies in wood-hydrophobe interactions. Relatively little chemical reagent should be required for effective water repellency if the hydrophobe is permanently bonded and well distributed. A high level of effectiveness should be obtained by the modification of external surfaces only. Apart from the economy of such a treatment, the technique is attractive in avoiding the problem of penetration and reagent-substrate accessibility.

Dimensional stabilization may be best achieved by new technology in crosslinking chemistry or bulking reactions. Crosslinking of cell wall components is a very interesting system; however, formaldehyde-treated wood suffers a definite embrittlement, caused perhaps by the short inflexible crosslinking unit of the -O-C-O- type. If the inner carbon chain unit were longer, there would be more flexibility in this unit and the embrittlement should be reduced. Although increased flexibility might reduce embrittlement, however, it would also reduce the dimensional stability by allowing structural units to expand more upon wetting. An interesting possibility is the use of reactive difunctional chemicals with longer carbon chains to achieve crosslinking. For example, difunctional epoxides or isocyanates of 4 to 10 carbon atoms reacted under mild conditions might achieve the' desired dimensional stability through crosslinking, without the accompanying embrittlement or loss of mechanical properties.

For dimensional stability for outdoor applications. cell wall reactive systems (for nonleachability) deserve further study. Careful examinations need to be done of bulking efficiency. hydrophobic efficiency, reaction efficiency, and penetration and distribution of bonded chemicals. None of the chemicals used so far penetrate as well, or as completely, as water. Because the hydroxyl oxygen is not removed during the reaction, an oxygen atom is still available to hydrogen-bond with water. We may be able to overcome this by bonding hydrophobes to that oxygen and thus repelling water from the site.

Complete dimensional stability of wood, i.e. ASE of 100 percent, has only been accomplished through the process of petrification. Chemical treatments are unlikely ever to achieve this level of treatment. Fortunately, for most applications, a lower level of stability suffices.

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☆ U. S. GOVERNMENT PRINTING OFFICE:1985/641-044/20015

The Forest Products Laboratory (USDA Forest Service) has served as the national center for wood utilization research since 1910. The Laboratory, on the University of Wisconsin-Madison campus, has achieved worldwide recognition for its contribution to the knowledge and better use of wood.

Early research at the Laboratory helped establish U.S. industries that produce pulp and paper, lumber, structural beams, plywood, particleboard and wood furniture, and other wood products. Studies now in progress provide a basis for more effective management and use of our timber resource by answering critical questions on its basic characteristics and on its conversion for use in a variety of consumer applications.

Unanswered questions remain and new ones will arise because of changes in the timber resource and increased use of wood products. As we approach the 21st Century, scientists at the Forest Products Laboratory will continue to meet the challenge posed by these questions.

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