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Physical and Mechanical Properties of Chemically Modified Wood

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I. INTRODUCTION

Chemical modification will be defined for this chapter as any chemical reaction between some reactive part of a wood cell wall component and a simple single chemical reagent, with or without catalyst, that forms a covalent bond between the two components. This excludes in situ polymerizations of monomers in the lumen structure of the wood and those reactions that result in cell wall–penetrating polymer systems that do not result in any cell wall attachment. It is well known that lumen-filling polymer treatment results in large improvements in mechanical properties, but these are mainly a result of the properties of the new polymer introduced [1].

To the extent possible, this chapter will deal with the changes in physical properties of wood as a direct result of chemical modification of the cell wall and not as a result of the reaction conditions used to modify the wood. For example, strong acid or base catalysts used in some chemical modification reaction systems will result in a reduction in physical properties due to hydrolysis or recrystallization of cellulose and these changes are not due to the

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chemical added to the cell wall. The actual change in physical properties that resulted from the chemical modification may be small but masked by the massive changes due to catalyst degradation. In many cases, it is very hard to determine what changes are due to the reaction conditions and those resulting from a change in cell wall chemistry.

This chapter deals mainly with changes in the mechanical properties resulting from the chemical modification of solid wood. There has been a lot of research done on modification of wood chips/particles/fiber that is then used to make wood composite. It is difficult to determine the mechanical changes in a composite made of chemically modified furnish because the properties of the adhesive and glue line influence the mechanical properties more than or as much as changes resulting from the chemical modification. Changes in gluability of chemically modified wood will be discussed as well as some changes in mechanical properties of wood composites.

II. CELL WALL CHEMISTRY AND MECHANICAL PROPERTIES

Any change in the chemistry of the wood cell wall polymers results in a change in physical and mechanical properties of the wood. These properties can vary from simple color change in the wood to major changes in modulus properties, work to maximum load, brittleness, hardness, wet strength, wet stiffness, impact strength, compressive strength, gas permeability, density, and moisture sorption. Improvements in dimensional stability and biological resistance and decreases in moisture sorption have been the motivation for much of the research done over the years in the chemical modification of wood [2–4]. Changes in cell wall moisture content resulting from chemical modification have a very large influence on mechanical properties.

A. Relationship Between Cell Wall Moisture Content and Mechanical Properties

Changes in the moisture content of the wood cell wall have a major effect on the mechanical properties of wood [5]. At moisture contents from oven-dry (OD) to the fiber saturation point (FSP), water accumulates in the wood cell wall (bound water). Above the FSP, water accumulates in the wood cell cavity (free water) and there is no tangible strength effect associated with a change in free water content. However, at moisture contents between OD and the FSP, water does affect strength. Increased amounts of bound water interfere with and reduce hydrogen bonding between the polymers of the cell

wall, which decreases the strength of wood. The approximate relationship between cell wall moisture content and strength is shown in Tables 1 and 2 [6]. It can be seen from the data in these tables that the moisture content of the cell wall has a great influence on all strength properties of wood.

Table 1 shows that fiber stress at proportional limit, work to proportional limit, and maximum crushing strength are the mechanical properties most affected by changing moisture content by only + 1% below the FSP. The change in mechanical properties from green to ovendry are shown in Table 2.

B. Effect of Chemical Modification on Cell Wall Moisture Content

Table 3 shows the changes in cell wall equilibrium moisture content (EMC) for several types of chemically modified wood. It can be seen that all bonded chemicals lower the EMC of the wood by about half that of nonreacted wood

Table 1 Approximate Change in the Mechanical Properties of Clear Wood When Subjected to Change in Moisture Content

Property	Changes per 1% change in moisture content (%)
Static bending	
Fiber stress at proportional limit	5
Modulus of rupture	4
Modulus of elasticity	2
Work to proportional limit	8
Work to maximum load	0.5
Impact bending	
Height of drop causing complete failure	0.5
Compression parallel to grain	
Fiber stress at proportional limit	5
Maximum crushing strength	6
Compression perpendicular to grain	
Fiber stress at proportional limit	5.5
Shear parallel to grain	
Maximum shearing strength	3
Hardness	
End	4
Side	2.5

Table 2 Relationship Between Some Mechanical Properties of Wood and Moisture Content

Property[a]	Moisture content				
	Green	19%	12%	8%	Oven-dry
	Douglas fir				
Modulus of rupture	62	76	100	117	161
Compression parallel to grain	52	68	100	124	192
Modulus of elasticity	80	88	100	108	125
	Aspen				
Modulus of rupture	61	75	100	118	165
Compression parallel to grain	50	67	100	126	199
Modulus of elasticity	73	83	100	111	137

[a]All values are expressed as a percentage of property at 12% moisture content.

at each RH tested except propylene oxide. EMC is not reduced as much at equal weight gains with propylene oxide as it is with acetylation or crosslinking with formaldehyde. This may be due to the fact that a new hydroxyl group is formed at the bonding site during the addition of the propyl group. Table 4 shows the FSP of control and acetylated aspen flakes. The FSP for acetylated aspen is reduced over 65% at about 17 weight percent gain (WPG). The data given in Tables 4 and 5 demonstrate the great decreasing effect of chemical

Table 3 Equilibrium Moisture Content of Control and Chemically Modified Pine

Chemical	Weight percent gain	Equilibrium moisture content at 27°C			
		0% RH	35% RH	60% RH	85% RH
Control	0	1.0	5.0	8.5	16.4
Acetic anhydride	20.4	0.7	2.4	4.3	8.4
Formaldehyde	3.9	0.4	3.0	4.2	6.2
Propylene oxide	21.9	1.4	3.9	6.1	13.1
Butylene oxide	18.7	1.3	3.5	5.7	10.7

Table 4 Fiber Saturation Point of Control and Acetylated Aspen Flakes

Weight percent gain (%)	Fiber saturation point (%)
0	46
8.7	29
13.0	20
17.6	15

Table 5 Changes in Volume of Southern Pine upon Drying and Modification with Propylene Oxide or Acetic Anhydride

A	B	B − A		C	C − B	C − A
Green volume (cm^3)	Oven-dry volume (cm^3)	*V* (%)	Weight percent gain (%)	Volume after modification (cm^3)	*V* (%)	*V* (%)
			Propylene oxide			
57.0	53.1	−6.9	15.9	56.0	+5.3	−1.7
59.0	53.1	−10.0	21.1	59.0	+10.0	0
59.0	55.1	−6.7	41.0	61.0	+9.7	+3.3
			Acetic anhydride			
23.3	21.1	−9.4	3.9	23.0	+8.3	−1.3
23.7	21.3	−10.1	22.8	23.7	+10.1	0

modification on the moisture content of the modified cell wall, which translates to major effects on strength properties.

C. Effect of Chemical Modification on Wood Volume

Table 5 shows the increases in cell wall volume resulting from reaction of southern pine with either propylene oxide or acetic anhydride. The oven-dry wood volume after modification with either of these chemicals at approximately 20 WPG is equal to the original wood green volume. This means that chemically modified wood has fewer fibers per centimeter than nonmodified wood. This means that if equal cross-sections of control and modified wood are used for mechanical tests there will be fewer fibers to test in the modified

wood as compared to the control. Since the cross-section of equal-sized control specimen contains about 10% more fibers than the modified specimen, the mechanical properties of the control should be higher than that of modified wood. Because of differences in volume and fibers per cross-section, it is difficult to compare properties between control and modified wood and make conclusions unless the difference in properties are very large.

III. CHANGES IN MECHANICAL PROPERTIES OF SOLID WOOD RESULTING FROM CHEMICAL MODIFICATION

There are always some changes in physical properties of wood when it is subjected to a chemical treatment. Vibrational properties change depending on the type of modification [7] and the moisture content of the modified cell wall [8] as do acoustical properties [9]. There is usually a color change associated with modification that may vary from almost no change to major, usually darkening, changes. The most dramatic mechanical property improvement in chemically modified wood over nonmodified wood is in wet strength and wet stiffness properties. Creep properties due to moisture sorption are also greatly reduced in chemically modified wood [10].

Most mechanical properties reported in the United States are done according to standards described by the American Society of Testing and Materials (ASTM). The standard method for testing small, clear specimens of timber (ASTM D143) calls for the test to be conducted at 65% RH. Control specimens have a moisture content of approximately 9% (Table 3), whereas specimens reacted with acetic anhydride or formaldehyde have a moisture content of only about 4% and specimens reacted with butylene oxide 6%. This means that the standard test is run on specimens of different moisture contents. Because of the great effect of moisture content on mechanical properties (Table 1), it is misleading or even invalid, to compare strength properties of control and modified wood since they were tested at different moisture levels.

Despite the problems associated with differences in fibers per cross-section and cell wall moisture content, the literature is full of comparisons of strength properties of control vs. modified wood. In many cases, the test procedure was not given or the RH used in the test was not reported, so that it is even harder to compare results from one author to another. The following is a summary of the literature on mechanical properties of chemically modified wood. However, as was stated earlier, it is hard to make comparisons or draw

conclusions due to differences in testing procedures, levels of modification, relative humidities of the tests, and the use of various hard and softwoods.

A. Changes Due to Esterification

Reacting wood with acetic anhydride to about a 15–20 WPG gives an increase in density from about 5% to 20% [11 – 13]. The color of very light woods (such as pine, maple, oak) usually darkens and the color of dark woods (such as walnut, cherry, teak) usually lightens due to acetylation. Permeability of gases decreases in acetylated wood as compared to control [14]. Shear strength parallel to the grain decreased from 12% to 24% [13]. In static bending tests, modulus of elasticity (MOE) varied from – 6% to + 2% [13, 15], modulus of rupture (MOR) varied from –8% to + 17 depending on the wood tested [13, 16, 17], fiber stress at proportional limit increased from + 7% to 20% [13], and work to proportional limit increased from 25% to 42% [13]. Ball hardness increased from 22% to 31% [13] and Brinell hardness increased tangentially 25% and radially 20% [17]. Impact strength varied from – 13% to + 16 [12,18]. Compressive strength perpendicular to the grain increased by 22–31% [13], and compressive strength parallel to the grain increased by 10% [16]. Wet compression strength at proportional limit increased by 93–144% [12,18]. Toughness varied from –7% to + 17% [11,13]. Work to failure decreased by 5–12% and tensile strength by decreased 1–4% [4]. Elongation at break in tension varied from –17% to + 42% [11]. All of these values are comparing acetylated wood to controls.

Other anhydrides, such as propionic, butyric, and phthalic, have been reacted with wood [18, 19] but no data have been published on mechanical properties of these modified woods.

Isocyanates have also been reacted with wood to give high levels of dimensional stability, but no mechanical tests have been conducted [20].

B. Changes Due to Crosslinking

Wood reacted with formaldehyde with an acid catalyst results in crosslinking between two hydroxyl groups in the cell wall polymers. The mechanical properties of formaldehyde-treated wood are all reduced from those of untreated controls. Toughness and abrasion resistance are greatly reduced [21-23], crushing strength and bending strength are reduced by about 20% and impact bending strength are reduced up to 50% [24] in specimens reacted to 4–7 WPG. The loss in toughness properties is directly proportional to the

gain in dimensional stability as measured in antishrink efficiency (ASE). For example, a specimen with a 60% ASE is equal to a 60% loss in toughness [21].

A definite embrittlement is observed in formaldehyde-treated wood that may result from the short crosslinking unit (O–C–O) formed between formaldehyde and fibrils in the cell wall. If the inner carbon unit were longer, there would be more flexibility in this unit and the embrittlement should be reduced.

Part of the loss in strength properties of formaldehyde may come from hydrolysis of cellulose by the strong acid catalyst.

C. Changes Due to Etherification

Wood has been reacted with propylene oxide and mechanical properties determined [25]. Maple specimens were reacted to 20–22 WPG and subjected to standard ASTM tests. The following is a summary of the results comparing propylene oxide–modified specimens to controls. MOE was decreased by 14%, MOR decreased by 17%, fiber stress at proportional limit reduced by 9%, maximum crushing strength decreased by 10%, radial hardness increased by 5%, tangential and longitudinal hardness remained unchanged, and the diffusion coefficient increased by 29%.

D. Changes Resulting in Cellulose Recrystallization and/or Matrix Modification

Because cellulose is the major component in strength properties of wood [6,23], if this polymer is depolymerized due to severe reaction conditions, or recrystallized, major changes in the mechanical properties of wood will result. The cellulose matrix of hemicelluloses and lignin can also be modified resulting in plasticization, which also causes major changes in mechanical properties. In some cases these changes result from the use of a strong oxidizing acid catalyst and the loss of mechanical properties is not desired. In other cases, the matrix is modified or the cellulose recrystallized in an attempt to make films and thermoplastics out of whole wood. In this case, the lowering of mechanical properties is not considered a loss because the wood structure is partially or totally lost in the new product.

Shiraishi and coworkers have had a 10-year program on wood molding at Kyoto University in Japan [26]. Their approach renders the entire wood structure thermoplastic through chemical modification of wood meal, which means that lignin and hemicelluloses are modified and the cellulose is decry-

stallized and modified. Their work has emphasized esterification of wood using trifluoroacetic acid (TFAA). Themoplasticity of esterified wood was found to depend on the acyl group, the method of preparation, and the degree of substitution. They found that as the size of the aliphatic group is increased the melting temperature of the modified wood at 3 kg/cm^2 is decreased.

Matsuda and Ueda [27] also extensively investigated the esterification of wood in order to make a totally thermoplastic material. They esterified wood with a solvent by simply heating wood meal with succinic anhydride for 3 h at temperatures greater than 60°C. The wood meal was readily molded at 180°C under a pressure of 570 kg/cm^2 for 10 min. The moldability of various esterified woods decreased in the following anhydride order: succinic anhydride > maleic anhydride > phthalic anhydride.

Hon and Ou [28] also produced a thermomoldable product by benzylation of wood powder. The degree of substitution was varied by changing the reaction alkalinity, temperature, and time. Sodium hydroxide concentrations greater than 25% were necessary to obtain a high weight gain, presumably because of the need to swell the lignocellulosic substrate. Different species showed variation in reaction rates. The thermoplasticized woods exhibited good melting properties and were readily moldable into bulk materials or extruded into films and sheets. A wide range of glass transition temperatures, from 66°C to 280°C, was observed for the benzylated wood and was largely dependent on weight gain. The molded and extruded products exhibited acceptable mechanical strength for structural engineering applications.

The lignin and hemicellulose matrix can also be thermoplasticized using various anhydrides such as maleic or succinic anhydrides [29]. If a nondecrystallizing reaction condition is used, it is possible to chemically modify the lignin and hemicellulose but not the cellulose. This selective reactivity has been shown to occur if uncatalyzed anhydrides are reacted with wood fiber [30]. This research is intended to produce wood veneers that are thermoformable.

IV. CHANGES IN MECHANICAL PROPERTIES OF WOOD COMPOSITES RESULTING FROM CHEMICAL MODIFICATION

It is very hard to compare data from chemically modified wood to data from composites made from chemically modified wood. There are so many variables in composite manufacturing alone that introducing a new chemical composition variable makes analysis even more complicated and tenuous.

Size, geometry, and orientation of particles in the composite has a great influence on mechanical properties as does density, type and level of adhesive used, volubility of resin in carrier liquid, ability of resin to penetrate into wood, moisture content, defects, wood species used, and many other variables.

A. Adhesion and Adhesives

The mechanism of adhesion is also an important factor in failure analysis in composites [31]. Some adhesives work due to a physical entanglement of the resin into the wood structure whereas others require a free hydroxyl group on one of the cell wall polymers to participate in a chemical reaction with the resin. Substitution of hydroxyl groups was shown to decrease adhesion between chemically modified veneers due to the loss of hydroxyl functionality [32]. Resins that are water-soluble and depend on a hydrophilic substrate for penetration will be less efficient in chemically modified wood due to the decreased hydrophilic nature of the cell wall resulting from modification [33].

Many different types of adhesives have been studied in the gluing of chemically modified wood, especially acetylated wood [34]. The adhesion of 18 thermoplastic and thermosetting adhesives was reduced by the level of acetylation, some to a minor degree and others to a severe degree. Many adhesives were capable of strong and durable bonds at a low level of acetylation (8 WPG) but not at higher levels (14–20 WPG). Most adhesives contained polar polymers, and all but four were aqueous systems, so that their adhesion was diminished in proportion to the presence of the nonpolar and hydrophobic acetate groups in acetylated wood. Thermosetting adhesives were capable of high shear strengths in the dry condition. With the exception of an acid-catalyzed phenol-formaldehyde adhesive, thermosetting adhesives that were hot-pressed became mobile and tended to overpenetrate the wood because of the limited capacity of the acetylated wood to sorb water from the curing bond line. The abundance of hydroxyl groups in a highly reactive resorcinol adhesive permitted excellent adhesion at room temperature despite the limited availability of hydroxyl groups in acetylated wood.

An emulsion polymer-isocyanate adhesive, a crosslinked polyvinyl acetate adhesive, a resorcinol-formaldehyde adhesive, a phenol-resorcinol-formaldehyde adhesive, and an acid-catalyzed phenolic-formaldehyde adhesive developed bonds of high shear strength and wood failure at all levels of acetylation in the dry condition. A neoprene contact bond adhesive and a moisture-curing polyurethane hot-melt adhesive performed as well on acetylated wood as untreated wood in tests of dry strength. Only a cold-setting resorcinol-formal-

dehyde adhesive and a phenol-resorcinol-formaldehyde adhesive, along with a hot-setting acid-catalyzed phenolic adhesive, developed bonds of high strength and maximum wood failure at all levels of acetylation when tested in a water-saturated condition [34].

B. Lumber Laminates

Vick and others [35] studied the dry and wet shear strength of acetylated spruce and pine lumber laminates. Using four different types of adhesives, they found that a resorcinol-formaldehyde adhesive on acetylated spruce showed a decreased (15%) dry shear strength but an increased (33%) wet shear strength as compared to controls. Similar results were found for an emulsion polymerisocyanate resin. A phenol-resorcinol-formaldehyde adhesive showed an increased (18%) dry shear strength and a greatly increased (52%) wet shear strength comparing acetylated laminates to controls. A polyvinyl acetate adhesive gave about the same increase in dry (20%) shear strength as in wet (28%) conditions comparing acetylated laminates to controls. Similar trends were found for pine laminates. Acetylation greatly reduced the swelling that occurs during the water soaking tests, so that there was essentially no stress fracturing in the acetylated wood. Nonacetylated wood developed many water-related stress fractures during the water soaking tests. A very high level of wood failure occurred in both acetylated and nonacetylated wood showing that the mechanism of failure was not due to the failure of the glue line.

C. Veneer Composites

The first work to compare mechanical properties of chemically modified wood to a composite made from chemically modified veneers was done in 1950 by Tarkow and Stamm [11]. They made a nine-ply parallel-laminated composite of 16-mm rotary-cut Sitka spruce veneers that had been acetylated to 28 WPG and compared properties to a control laminate and control and acetylated single veneers. A hot-press phenolic resin was used as an adhesive in the laminated composite. while acetylated single veneers had a MOE, in tension parallel to the grain, of + 10% greater than control veneers, the acetylated laminate MOE was lower than controls by 21%. Ultimate strength, in tension, of acetylated single veneers was 10% higher than controls but was 14% lower in the acetylated laminate than controls. These results show that mechanical properties were lowered due to the adhesion between veneers. In the control laminates, 97% of the failure of the glue line occurred in the wood whereas only 89% of the failure occurred in the wood for acetylated veneers.

In general, other properties of the acetylated laminates showed, improved properties over control laminates. For example, acetylated laminates in compression tests parallel to grain showed a 3% increase in stress at proportional limit, 8% increase in ultimate strength, 6% decrease in MOE, and 21% increase in deformation at failure as compared to controls. In compression tests perpendicular to the grain, acetylated laminates showed a 30% increase in stress at proportional limit, 47% increase in ultimate strength, 21% increase in MOE, and 10% increase in deformation at failure as compared to controls.

D. Flakeboards

In flakeboards made from acetylated aspen flakes, internal bond (– 36%), MOR (– 34%), and MOE (– 11%) were decreased as compared to boards made from nonacetylated flakes [33,36]. The adhesive used was a water-soluble phenol-formaldehyde at a solids content of 6%. Electron micrographs showed that fragmentation, intercell, intrawall, and transwall separations increased as the level of acetylation increased. These defects were found only in the outermost layer of surface flakes and were not considered to influence the mechanism of failure of the glue line. In these studies, 75–90% of the failures during test occurred in the glue line of the acetylated wood and not in the wood, showing that adhesive failure was the main mechanism of failure [33]. In unacetylated flakeboards, more than 95% wood failure occurred. The reduced moisture content of the acetylated flake made them less compressible and required higher press pressures to make the flakeboards [36].

Using an isocyanate resin, at 3% solids content, on the same aspen flakes as described above, dry internal bond strength decreased by only 2%, MOR decreased by 23%, and MOE decreased by 15% on boards made from acetylated flakes as compared to boards made from nonacetylated flakes [37]. This is just one more example of how the type and level of the resin used influenced composite board mechanical properties.

E. Fiberboards

There have been several studies on the changes in mechanical properties of fiberboards made from chemically modified wood fiber. Hardboards made from control and acetylated hemlock fiber using 7% phenyl-formaldehyde adhesive were tested. In static bending, MOR was reduced by 23% and MOE reduced by 16% in acetylated boards as compared to control boards [38]. Tensile strength parallel to the surface was reduced by 5% but there was no

change in the tensile strength perpendicular to the surface in acetylated boards compared to controls.

Fiberboards made from control and acetylated aspen fiber were made using 8% phenol-formaldehyde resin and tested in static bending. MOR increased by 15% and MOE increased by 40% in acetylated fiberboards compared to controls [39]. The acetylated boards were reported to have a more uniform density and a more consolidated surface as compared to controls.

Finally, changes in mechanical properties have been conducted on control and acetylated mixed wood (MW) fiber and recycled old newspaper (ONP) fiber. The boards were made using a phenol-formaldehyde resin at several different levels of resin solids. Comparing data generated in static bending tests on fiberboards made using 7% resin, MOR was reduced by 4% and MOE increased by 3% for boards made from acetylated ONP compared to controls, and MOR was reduced by 35% and MOE reduced by 21% for boards made from acetylated MW compared to controls [40]. It is interesting to note that in the same tests MOR was reduced by 55% and MOE reduced by 63% comparing control WF to ONP fiberboards whereas MOR was reduced by only 12% and MOE by 34% comparing acetylated WF to ONP.

V. CONCLUSIONS

If there are no major disruptions, depolymerizations, or recrystallization of cell wall polymers during the reaction chemistry to modify solid wood, there are no statistical differences in mechanical properties of chemically modified wood as compared to nonmodified wood. There are so many differences in moisture levels, specific gravities, and fibers per unit cross-section in control vs. modified woods that no definitive conclusions can be made. If the reaction chemistry used to modify solid wood does result in major disruptions, depolymerizations, or recrystallization of cell wall polymers, then there are major statistical differences between control and chemically modified solid wood. Losses in mechanical properties can vary from large decreases in all properties to complete loss of cell wall structure and wood is converted to a thermoplastic film.

In both solid wood and wood composites, the major improvement in mechanical properties resulting from chemical modification is in wet strength and wet stiffness.

There are so many variables to consider in wood composites that is it also difficult to draw conclusions on the effect of chemical modification on mechanical properties of composites. The size, geometry, and orientation of

flakes, particles, fibers, and so forth in the composite has a great influence on mechanical properties as do variations in specific gravity throughout the boards, stratification of fines in boards, type and level of adhesive used, ability of the resin system to penetrate into wood, moisture content during testing, defects/gaps arising in the board forming process, hydrophilic/hydrophobic nature of the cell wall, availability of reactive hydroxyl groups in the modified accessible cell wall polymers, and wood species used. As was concluded for solid wood, there does not seem to be any statistical difference in mechanical properties between wood composites made from chemically modified and nonmodified furnish except in wet strength properties.

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