

SURFACE FREE ENERGY OF BLUE-STAINED SOUTHERN PINE SAPWOOD FROM BARK BEETLE-ATTACKED TREES

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(Received December 2012)

Abstract. Blue-stained wood cut from bark beetle-attacked southern pine has a lower economic value than unstained wood. Wood composite products containing blue-stained wood may offer an opportunity to recover some lost timber value. This study investigated the surface-free energy of blue-stained wood. Southern pine sapwood samples with and without blue stain from both green and kiln-dried sources were obtained. Dynamic contact angle analyses were performed using three probe liquids: ethylene glycol, formamide, and deionized water. Surface-free energy was determined by applying the geometric mean model using two-liquid pairs with deionized water. The polar forces were higher across all wood types and in water–ethylene glycol vs water–formamide. Surface-free energy of air-dried blue-stained sapwood was lower than all other wood types. However, kiln-dried blue-stained sapwood had a higher surface-free energy than all other wood types. These results were indicative of a tree’s wound response to bark beetle

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attack, the volatilization of naturally occurring hydrocarbons in southern pine sapwood, and the resulting increase in wood permeability caused by blue-stained fungal colonization across the sapwood. However, improvements in wetting observed for kiln-dried blue-stained sapwood may lead to cost and quality issues in wood composite manufacturing associated with overdrying and overpenetration of an adhesive.

Keywords: Blue-stain, bark beetle, dynamic contact angle, southern pine, surface-free energy, wood composite.

INTRODUCTION

Bark beetles, such as the southern pine beetle (SPB) (*Dendroctonus frontalis* Zimmermann), have historically been the major insect threat to the economically important southern yellow pines (*Pinus* spp.) in the southeastern US (Cook and Hain 1987). Forest landowners and managers most recently dealt with an SPB outbreak during the late 1990s, and the regional economic impact was estimated to be more than \$1 billion (Nowak et al 2008). Southern yellow pine in the southeastern US comprises nearly half of the total softwood lumber supply produced nationally (Haygreen and Bowyer 1996). Therefore, impacts of SPB outbreaks have the ability to disrupt residential and commercial construction markets on a national scale.

SPBs initially overcome the oleoresin defenses in pines through mass attacks and inoculation of a variety of fungi on the host (Nebeker et al 1993). One constituent of this fungal complex, a blue-stain fungus (*Ophiostoma minus* [Hedgc.] Syd. & P. Syd.), is the most prolific in the early stages of host colonization. Moisture losses in bark beetle-attacked southern pine trees can approach 52% within 1 mo of visible foliage chlorosis, and specific gravity (SG) decreases of 16% can occur within 6 mo (Barron 1979). Solid-sawn lumber has been produced from timely salvaging of SPB-attacked trees, albeit with some losses in lumber recovery (Sinclair and Ifju 1979). Decreases in grade associated with SPB-attacked timber are not caused by the presence of blue-stained fungi, which do not deteriorate wood (Schirp et al 2003; Valiev et al 2009). Rather, these grade decreases are usually related to untimely salvage and use strategies that result in strength decrease via subsequent activities of wood-destroying borers and their associated decay fungi (Barron 1979).

Wood decay fungal fruiting bodies have been identified in SPB-attacked timber as early as the end of the third month following tree death (Barron 1979). Southern pine grading rules restrict the presence of decay in structural lumber because loadings potentially would not meet design specifications. Blue-stain alone is permitted within the southern pine grading rules for structural light framing, joists, and planks (Sinclair and Ifju 1979). However, decay in the early stages can be difficult to detect during visual inspection in the presence of heavy staining (Sinclair et al 1979). Toughness, the mechanical property most sensitive to the presence of blue-stain fungi, can significantly decrease in as little as 2 mo (Sinclair et al 1979). The value of bark beetle-attacked trees to lumber manufacturers therefore diminishes greatly in a short period of time (Levi and Dietrich 1976).

A portion of the decreased value of blue-stained southern pine timber may be recoverable through manufacturing wood composite products, which can be engineered to overcome a number of wood quality limitations. The recent massive outbreak of mountain pine beetle (MPB) (*Dendroctonus ponderosae* Hopkins) in the western US and Canada has led to numerous recent studies regarding suitability of blue-stained lodgepole pine (*Pinus contorta* Douglas ex Loudon) for use in wood composite products. Use of blue-stained wood in composite products is highly dependent on an understanding of the resulting alterations in wood permeability and diffusion rates. Increases in wood permeability and diffusion rates are commonly reported with blue-stained portions of bark beetle-attacked trees, which can significantly affect usability (Cai and Olivera 2008). Blue-stained wood from MPB-attacked lodgepole pine is suitable for the manufacture of cement-bonded particleboard and

wood–plastic composites (Chang and Lam 2009, 2010; Chang et al 2010). Also, increases in stiffness and strength properties have been observed in veneer and plywood (Wang and Dai 2008; Wang et al 2008).

Wood composite products manufactured from SPB-attacked timber have received limited attention, especially in recent decades. Kelly et al (1982) investigated the effect of SPB-attacked southern pine wood mixed with wood from healthy trees for particleboard production. Significant increases were reported in modulus of rupture, internal bond, and screw withdrawal when SPB-attacked wood was used at a 25 or 100% mixture compared with control (unstained) wood. Also, significant decreases in 2- and 24-h thickness swell and water absorption were reported at both furnish levels. These changes were found using wood from trees that had been dead for 3 and 27 mo. No proposed explanations, such as increased permeability, better resin penetration, or higher compaction ratio, were suggested for the observed benefits. Also, no research has quantified the surface property changes of wood subjected to SPB fungal associates, such as *O. minus*.

Wettability of materials can be determined by dynamic contact angle (DCA) analysis and the subsequent calculation of surface free energy (SFE). DCA measures the downward force of a wood sample hanging perpendicular to the liquid's surface. This accounts for the entire surface area of a sample by taking into consideration the variable topography of the wood's surface on a microscopic scale (Son and Gardner 2004). Using liquids with known surface properties to measure DCA allows SFE of the wood surface to be determined by use of one of several models (Gardner et al 2000). The geometric mean, a combination of Young's equation (Young 1805), Girafalco and Good's geometric mean law (Girafalco and Good 1957), and Fowkes' equation (Fowkes 1962), describes the sum of the physical and chemical molecular interactions occurring between the liquid and wood surface. The physical, or nonpolar, interactions are explained by dispersion forces,

whereas the chemical, or polar, interactions are expressed as polar forces (Gardner 1996; Wålinder and Gardner 2000).

An adhesive must adequately wet the wood surface by penetrating into the micropore structures of the wood cell wall to achieve an optimum wood-adhesive bond (Bryant 1968). This intimate contact is critical for adhesive bonding strength and wood composite durability. The wetting of wood occurs through the wicking of liquids by capillary forces (Wålinder and Johansson 2001). The greater the movement of a liquid into voids and along rays and channels, the more wetting will occur.

Sixty percent of US forest products manufacturing occurs in the South. This production depends on wood from southern pines (Prestemon and Abt 2002). A better understanding of the work of adhesion at the resin–wood interface may offer new economic outlets for southern pine timber affected by bark beetle attack, particularly those not suitable for solid-sawn lumber. The objective of this study was to examine the potential effects of blue-stain fungal attack on surface properties of the affected southern pine sapwood. SFE of air- and kiln-dried wood with and without blue-stain was calculated using DCA analysis.

MATERIALS AND METHODS

Ten wafers of southern pine sapwood measuring $25.0 \times 25.0 \times 7.0$ mm (tangential [T] \times radial [R] \times longitudinal [L]) along with 10 miniature beams measuring $5.0 \times 15.0 \times 150.0$ mm (T \times R \times L) were randomly selected from those cut from each of the following wood sources: 1) three unstained green boards (25.0 mm \times 200.0 mm \times 2.4 m); 2) three unstained kiln-dried boards (25.0 mm \times 200.0 mm \times 2.4 m); 3) three blue-stained kiln-dried boards (25.0 mm \times 200.0 mm \times 2.4 m); and 4) three 1.2-m-long bolts (with comparable rings per inch) from the butt logs of three SPB-attacked trees in Talladega National Forest in central Alabama. The lumber (excluding the three bolts sawn from SPB trees) was obtained from a mill in Choctaw

County, MS. Green boards were removed from the sawmill production line in 10-min intervals to ensure that they were milled from separate trees. Kiln-dried boards were obtained from different stacks at the mill, which were dried using a conventional southern pine drying schedule (105°C dry bulb, 55°C wet bulb for approximately 20 h). Sample bolts were collected from three different SPB-attacked trees within 2 m of visible foliage chlorosis, and there were no obvious signs or symptoms of woodborers or decay fungi. Visual inspections were conducted to ensure that all samples were cut from sapwood and that no pith was present. Following machining, the wafers and miniature beams were placed in a conditioning chamber at $24 \pm 2^\circ\text{C}$ and $55 \pm 5\%$ RH until constant mass was attained, thus air-drying the green samples.

Mean wood porosity was determined for each wood type using wafers and following the oven-dry method of Usta (2003) based on $SG = 1.54$ for wood cell walls. Wafers sawn from each wood source were individually measured in radial, tangential, and longitudinal planes three separate times to obtain green volumes. Wafers were then placed in a convection oven at $103 \pm 2^\circ\text{C}$ for 24 h to obtain oven-dry mass. SG was then calculated for each wood type. The amount of wood cell wall material (K) was measured as a function of each wafers' SG ($K = SG/1.54$). Porosity was then determined as ($P = 1 - K$). Also, wood from each type was ground in a Wiley mill to pass a size 20 mesh screen. Wood meal and distilled water were mixed together in 10-mL beakers in a 1:1 ratio. pH of the wood meal was taken 24 h later using a glass electrode, which had been calibrated prior to measurement with a buffer solution. Ten replicates were performed for each test. Means were tested using analysis of variance and Tukey's honestly significant difference test at $\alpha = 0.05$.

A Stanley (New Britain, CT) No. 90 FJ Bullnose plane was used to machine 10 fresh strips with target dimensions $0.25 \times 5.0 \times 15.0$ mm ($R \times T \times L$) from each of the 10 miniature beams for each wood type. Five strips from each beam were randomly selected and placed into a sealed bag

for DCA analysis of each wood type. Actual dimensions (width and thickness) of each DCA specimen were measured three times using calipers, averaged, and recorded at the time of DCA measurement.

Three probe liquids with known surface tensions, ethylene glycol (Fisher Scientific, Fair Lawn, NJ), formamide (Arcos Organics, Morris Plains, NJ), and deionized water, were used as standards (Table 1) (Wu et al 1995). Forty milliliters of each probe liquid were measured and placed serially on the moving stage of a Thermo Cahn (Newington, NH) DCA 322. Wood strips were randomly selected from a wood type and hung perpendicularly to the liquid and counterbalanced to ± 1 mg. The moving stage raised the liquid at a rate of $264 \mu\text{m/s}$, which was greater than the adequate threshold required for wood (Gardner et al 1991). When the wood and liquid made contact, "zero depth of immersion" was registered and force data were gathered to a depth of 4.0 mm. WinDCA software (Cahn instruments, Inc., Madison, WI) calculated the advancing and receding dynamic contact angles using a buoyancy correction factor. The porous structure and hydrophilic nature of wood can lead to liquid absorption, which can significantly affect the receding contact angle (De Meijer et al 2000). Therefore, only the advancing DCA was applied to the SFE calculation (Scheikl and Dunky 1998; Gindl et al 2001, 2004). Five replications were performed per wood type, each with a different wood strip, and averaged per probe liquid. Natural variation caused by early/latewood ratios existed among the samples. DCA accounts for this difference by immersing the entire sample into a probe liquid, whereas static contact angle measurements, by placement of a single droplet, cannot account for this variability.

Table 1. Properties of probe liquids used for the advancing contact angle measurements (mJ/m^2) (Wu et al 1995).

Probe liquid	γ_L^d	γ_L^p	γ_L
Water	21.8	51.0	72.8
Formamide	39.0	19.0	58.0
Ethylene glycol	29.0	19.0	48.0

The geometric mean model was applied using paired liquid combinations with water to determine the dispersive and polar components of SFE for each wood type:

$$\frac{(1 + \cos \theta) * \gamma_L}{2 * (\gamma_L^d)^{1/2}} = (\gamma_S^d)^{1/2} + (\gamma_S^p)^{1/2} * \left(\frac{\gamma_L^p}{\gamma_L^d}\right)^{1/2} \quad (1)$$

where θ represented mean advancing contact angle of each wood type in a liquid, γ_L represented total surface tension of a probe liquid, γ_L^d and γ_S^d represented dispersive forces of a liquid and wood, and γ_L^p and γ_S^p represented polar forces of a liquid and wood. The unknown parameters of each wood type, $(\gamma_S^d)^{1/2}$ and $(\gamma_S^p)^{1/2}$, were solved using a simple linear regression model:

$$Y = \beta_0 + \beta_1 X + \varepsilon \quad (2)$$

with $\frac{(1+\cos\theta)\gamma_L}{2(\gamma_L^d)^{1/2}}$ representing Y , $(\gamma_S^d)^{1/2}$ the intercept, $(\gamma_S^p)^{1/2}$ the slope, and $\left(\frac{\gamma_L^p}{\gamma_L^d}\right)^{1/2} X$ in the model, respectively. The intercept, $(\gamma_S^d)^{1/2}$, and slope, $(\gamma_S^p)^{1/2}$, of each line were then squared to determine the dispersive and polar components of the wood types. Summing the two determined total SFE of each wood type. All analyses were performed in SAS 9.1.3 (SAS Institute 2003).

RESULTS AND DISCUSSION

Mean SG, porosity, and pH for each wood type are given in Table 2. SG and porosities of the two control treatments did not significantly differ; both were within ± 0.01 ($\pm 0.6\%$). Blue-stained wood types differed 0.06 in SG and 4.7% in porosity. Air-dried, blue-stained wood

was significantly denser ($F_{3,36} = 253.33$, $p < 0.0001$) and less porous ($F_{3,36} = 130.39$, $p < 0.0001$) than all other wood types. However, kiln-dried, blue-stained southern pine was significantly less dense and more porous than all other wood types. SG (oven-dry weight/green volume) of unextracted southern pine is known to be affected by the extractives content (subsequently discussed) of the wood (Koch 1972). Because the air-dried blue-stained wood was obtained from trees in the early stages of foliage chlorosis, wound response of these trees to bark beetle attack may have led to an increase in SG.

The average advancing DCA of each wood type in the probe liquids is listed in Table 3. Dispersive forces, γ_S^d , polar forces, γ_S^p , and total SFE, γ_S , for each liquid pair of the four wood types are shown in Table 4. Defect-free southern pine contact angle samples typically have a greater degree of variation when obtained along the grain with hand tools compared with those obtained with electrically powered devices (Stehr et al 2001). Minute imperfections along the grain caused by a hand tool may have contributed to some contact angle variability in this study.

Total SFE ranged from 49.0–58.8 mJ/m² for water–formamide and 51.5–64.4 mJ/m² for water–ethylene glycol. Kiln-dried blue-stained wood had the higher SFE in each liquid combination. Air-dried blue-stained wood had a lower SFE than all other wood types. Kiln-dried and air-dried controls had the second and third highest SFE in each instance. Higher SFE occurred in the water–ethylene glycol combinations than in the water–formamide combinations.

Polar forces ranged from 37.0–50.2 mJ/m² in water–formamide combinations and 46.2–61.2 mJ/m² in water–ethylene glycol combinations.

Table 2. Mean (standard error) specific gravity, porosity, and pH of the four wood types.^a

Wood type	Specific gravity	Porosity (%)	pH
Air-dried SPB ^b blue-stain	0.53 (0.003) A	65.1 (0.253) C	4.90 (0.007) B
Air-dried control	0.49 (0.003) B	68.1 (0.095) B	5.34 (0.062) A
Kiln-dried control	0.50 (0.003) B	67.5 (0.200) B	4.76 (0.009) C
Kiln-dried blue-stain	0.47 (0.003) C	69.8 (0.126) A	4.73 (0.011) C

^a Capital letters indicate significantly different means within columns at $\alpha = 0.05$.

^b SPB, southern pine beetle.

Table 3. Advancing contact angles of wood types in three probe liquids.

Wood type	Probe liquid		
	Water	Formamide Mean contact angle (standard error)	Ethylene glycol
Air-dried SPB ^a blue-stain	51.1 (1.15)	52.7 (0.69)	41.4 (0.70)
Air-dried control	47.0 (3.72)	42.8 (4.69)	39.3 (1.33)
Kiln-dried control	45.4 (1.85)	45.1 (1.15)	42.0 (0.78)
Kiln-dried blue-stain	40.0 (2.26)	45.8 (0.15)	34.4 (2.06)

^a SPB, southern pine beetle.

The polar forces obtained when pairing ethylene glycol (acidic probe liquid) with water were higher than those found when comparing formamide (basic probe liquid) with water within a wood type. This may indicate an overall more basic southern pine wood surface (Gardner 1996). Kiln-dried blue-stained wood had the highest polar component within each liquid pair. The higher polar forces in the water–formamide pair for kiln-dried blue-stained wood may also indicate more acidic sites were present relative to the other wood types. Across all wood types, the higher polar forces in the water–ethylene glycol combinations may have contributed to a lower dispersive component than that observed for the water–formamide dispersive component in each liquid pair. Overall, the dispersive forces contributed the least to total SFE in this study, ranging from 2.5–11.8 mJ/m². There was not a consistent ordering of the wood types' dispersive forces for each liquid combination.

Wood types dried with the conventional kiln schedule had higher polar components and SFE than the air-dried wood types. Increases in the SFE of kiln-dried wood have been reported up to

the glass transition temperature of lignin, 60°C, at which point the wood structure was altered and SFE decreased (Gunnells et al 1994). Structural degradation, however, is believed to be negligible under normal lumber drying conditions (Milota 2006). Because SFEs were higher for both stained and unstained kiln-dried wood types in this study, excessive drying conditions did not appear to have been present.

The process of kiln-drying removes some water and extractives from southern pine, including hydrocarbons known collectively as volatile organic compounds (VOC) (Shmulsky 2000). This occurs initially as the wood surface is heated to the wet-bulb temperature followed by the migration and removal of bound water through internal diffusion. As the surface temperature of wood increases, vapor pressure increases and additional VOCs are removed (Ingram et al 2000). Because removal of extractives increases the overall acidity of wood (Wålinder and Gardner 2000), the migration and loss of southern pine extractives, including VOCs, during the kiln-drying process may have resulted in the lower pH values observed for kiln-dried wood types (Table 2). Also, the

Table 4. Total surface energies (mJ/m²) and their components for the four wood types.

Wood type	Probe liquid pairs	γ_s^d	γ_s^p	γ_s
Air-dried SPB ^a blue-stain	Water-For ^b	9.40	39.6	49.0
	Water-EG ^b	5.30	46.2	51.5
Air-dried control	Water-For	14.4	37.0	51.5
	Water-EG	4.20	52.2	56.4
Kiln-dried control	Water-For	11.8	41.4	53.2
	Water-EG	2.50	58.4	60.9
Kiln-dried blue-stain	Water-For	8.60	50.2	58.8
	Water-EG	3.30	61.2	64.5

^a SPB, southern pine beetle.

^b For, formamide; EG, ethylene glycol.

removal of hydrophobic extractives is known to increase wettability (Gunnells et al 1994).

The presence of blue-stain in air-dried wood resulted in a lower SFE than all other wood types. This is indicative of various physical, anatomical, morphological, and chemical changes taking place within the tree once fungal inoculation occurs via bark beetle attack (Shigo and Marx 1977; Barron 1979; Blanche et al 1983; Shamoun and Levi 1985; Woo et al 2005). These factors, among others, can affect the SFE of wood (Gindl et al 2004). The nutrient-rich wood rays and ray parenchyma cells are the primary pathways of colonization by many micro-organisms (Greaves 1971). Blue-stain fungi are initially confined to the radial parenchyma tissue of the sapwood, causing a blockage of water-conducting passages through internal wounding. This results in water at first being conducted around but not through fungal-infected areas of the sapwood (Mathre 1964). It is likely that wettability of the air-dried blue-stained wood was decreased in trees following bark beetle attack as a result of blue-stained fungal inoculation and the initial wound response of the tree. This includes resin formation, which slows the rate of fungal spread but also produces abnormal levels of various extractives, including VOCs (Hodges and Lorio 1975; Tisdale et al 2003).

Kiln-dried blue-stained wood had the highest SFE of all wood types. Wood porosity, and consequently permeability, is increased with time as defensive barriers in the sapwood's radial, tangential, and longitudinal planes, such as occluded resin canals and pit membranes, are degraded by blue-stained fungi (Whitney 1971; Ballard et al 1983; Tisdale et al 2003). The increase in wood permeability caused by *O. minus* infection therefore allows increased liquid movement across the grain (Greaves 1971). Mean wood porosity highly correlated with SFE in this study, although statistical significance was moderate (water–ethylene glycol $r = 0.90$, $p = 0.1093$; water–formamide $r = 0.91$, $p = 0.0941$). Cai and Olivera (2008) concluded that increased permeability of blue-

stained lodgepole pine was caused by 1) rupturing of the ray parenchyma cells; 2) rupturing of the pit membranes; 3) checking in the middle lamella; and 4) openings in the aspirated pits. Therefore, the higher SFE of kiln-dried blue-stained wood compared with other wood types can be explained by a rougher and more variable surface (Young 1976) caused by blue-stain fungal infection.

Results reported in this study for kiln-dried blue-stained wood, although promising, warrant some consideration for bark beetle-attacked timber. Because of the South's high temperatures and humidity, bark beetle-attacked timber has a relatively short period of utility. Use of blue-stained wood in composite products may lead to excessive tool wear, fines generation, and uneven drying during production. Over-drying can lead to excessive moisture uptake from the adhesive affecting resin flow. Also, heating the wood surface above safe tolerances leads to surface inactivation, decreasing wettability (Christiansen 1990). The drying schedule used in this study was for lumber, which is considerably less severe than those for wood composite manufacturing. Any overpenetration of the adhesive can lead to irreversible thickness swelling, first at press opening and second when the composite is exposed to moisture (Byrne et al 2005).

CONCLUSIONS

Kelly et al (1982) reported increases in various wood composite structural properties for SPB-killed southern pine as a portion of furnish; however, no explanations were given for the observed benefits. Our results indicate that 1) air-dried blue-stained wood from bark beetle-attacked trees had a lower SFE than all other wood types; and 2) kiln-dried blue-stained wood had a higher SFE than all other wood types. These changes may be explained by increased wood porosity, and consequently SFE, as a result of a rougher and more variable surface caused by bark beetle blue-stain fungal infection and subsequent exposure to kiln-drying. These

findings hold promising implications for the use of bark beetle-attacked timber in wood composite manufacturing. Further research is needed to examine the effect of drying processes on wettability of bark beetle-attacked timber.

ACKNOWLEDGMENTS

This research was funded by the USDA Forest Service Forest Health Protection, the Southern Wood to Energy group, the Mississippi Agricultural and Forestry Experiment Station, and the Forest and Wildlife Research Center, Mississippi State University. We thank Southeastern Timber Products of Ackerman, MS, for donating the lumber for this project.

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