

Wood-Plastic Composites with Reduced Moisture: Effects of Chemical Modification on Durability in the Laboratory and Field

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Abstract

Although laboratory evaluations of wood-plastic composites (WPCs) are helpful in predicting long-term durability, field studies are needed to verify overall long-term durability. Field exposure can encompass numerous degradations i.e., fungal, ultraviolet light, moisture, wind, temperature, freeze/thaw, wet/dry cycling, termites, mold, etc. that traditionally are studied separately in the laboratory. Moisture sorption of WPCs is slower than in unmodified solid wood, but it affects the strength, stiffness, and ultimately the decay of the material.

The objectives of this study were to: 1) investigate several methods of reducing moisture sorption and, consequently, fungal degradation, 2) compare the effects of moisture and fungal decay on woodflour-filled and woodfiber-reinforced high-density polyethylene, and 3) perform laboratory and field evaluations of WPCs specimens. The methods used to reduce moisture were: 1) acetylation of the wood component and 2) use of a coupling agent. Acetylating wood esterifies the hydroxyl groups, making the wood more hydrophobic, dimensionally stable, and biologically durable. Cou-

pling agents are known to promote bonding between the plastic and unmodified wood fibers when added to WPCs and have been shown to reduce moisture.

Acetylating the wood component in WPCs reduced moisture sorption and related performance losses such as decay and flexural property loss in laboratory tests. Reduction in moisture sorption and decay with addition of a coupling agent were significant but not as large as acetylation. Early findings in field tests were consistent with laboratory results but considerably more time is needed to adequately assess the different formulations.

Introduction

Demand for wood-plastic composite (WPC) and plastic lumber in the United States is predicted to increase 11 percent per year through 2009 to \$3.5 billion (Staff 2006). Decking applications are expected to account for almost 40 percent of value demand in 2009. The main reasons for these gains are the removal of decking treated with chromated copper arsenate (CCA) and consumer expectations for improved performance characteristics, such as high durability, low maintenance requirements, and enhanced appearance. But, some of these improvements may still not meet consumer expectations.

It was first thought that mixing plastic and wood together would result in plastic encapsulation of wood, which would prevent moisture sorption and fungal decay. Morris and Cooper (1998), however, found evidence of fungal decay and discoloration on WPC decking in service. Since this first evidence, we have

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evaluated moisture, ultraviolet (UV), and biological degradation (Ibach et al. 2004). Shrip et al. (2007) reviewed the literature on biological degradation of WPCs and strategies for improving resistance.

In outdoor conditions, WPCs are exposed not only to biological and water degradation but also UV, thermal, chemical, and mechanical degradations. Chemical modification of wood cell walls can prevent or slow these degradation reactions (Rowell 2005). Although WPCs have slower moisture sorption than wood (Rowell et al. 2002, Wang and Morrell 2004), water affects the strength and ultimately promotes biological decay of the material (Clemons and Ibach 2002, 2004; Shrip and Wolcott 2005).

Acetylation is one of the most studied wood modification chemistries. Reaction of acetic anhydride with wood results in esterification of accessible hydroxyl groups in the cell wall with acetyl groups and reduced moisture sorption. Because moisture plays a key role in fungal decay of WPCs, it is prudent to evaluate WPCs made with acetylated wood fiber (Glasser et al. 1999, Ibach and Clemons 2002, Khalil et al. 2002, Glasser 2004, Segerholm et al. 2005). Acetylated wood-fiber-plastic composites that were compression molded had better mechanical properties and water resistance than unmodified WPC controls (Glasser et al. 1999, Khalil et al. 2002). Acetylated woodfiber-plastic composites that were injection molded and extruded had decreased moisture content (MC) and were highly resistant to brown-rot decay compared with unmodified WPC controls (Segerholm et al. 2005). The acetylated fiber in Segerholm et al.'s research was produced by grinding previously acetylated solid wood. Ibach and Clemons found that compression molded composites of acetylated ground wood fiber and high-density polyethylene (HDPE) decreased the equilibrium moisture content (EMC) and fungal decay of the WPC compared with composites made with unmodified wood or butylene- or propylene-oxide modified wood (Ibach and Clemons 2002).

Coupling agents are incorporated into WPCs to improve the compatibility and adhesion between polar wood fibers and non-polar polymers (Bledzki et al. 1998, Lu et al. 2000, Bledzki and Omar 2003). The most common coupling agents are maleic anhydride grafted to polypropylene (MAPP) or polyethylene (MAPE). WPCs containing polypropylene (PP) and the coupling agent maleated MAPP were injection molded and their mechanical properties evaluated. MAPP improved polymer-woodfiber adhesion and dispersion of particles and lowered water absorption compared with composites without the coupling agent (Ichazo et al. 2001). MAPP enhanced the tensile modulus and yield stress and the Charpy impact strength (Hristov

et al. 2004). MAPP caused greater strength increases with woodfiber composites than with woodflour composites (Stark and Rowlands 2003). MAPP with longer chain lengths and lower functionality gave the greatest benefit, supporting the chain entanglement mechanism proposed by Pickering and Ji (2004). Kazayawoko et al. (1999) attributed the improvement of mechanical properties with MAPP to the compatibilization effect, which decreases total woodfiber surface free energy, thus improving polymer matrix impregnation, fiber dispersion, fiber orientation, and interfacial adhesion through mechanical interlocking.

Both the addition of a coupling agent and acetylation of the wood component appear to offer potential for reducing moisture sorption and improving biological durability. Field tests on these composites, however, have not been conducted, and results have not been compared with laboratory fungal tests. The following research begins to address this need. The overall objective of this study was to determine moisture and biological resistance of WPCs made with chemically modified wood in both field and laboratory tests.

Experimental

Materials

The wood filler used in this study was western pine wood flour, nominal 40 mesh (420 μm), from American Wood Fiber (Schofield, WI). The southern pine wood fiber was produced by atmospheric refining at Matts Incorporated (Floodwood, MN) and then screened to -20/+40 mesh. The plastic material was HDPE from reprocessed milk bottles (Muehlstein and Co., Inc., Roswell, GA), with a melt flow index of approximately 0.7 g/10 min. The flour was modified with acetic anhydride from Aldrich Chemical Company (Milwaukee, WI). The coupling agent was maleated polyethylene (MAPE), Polybond 3009, from Chemtura Corporation (Middlebury, CT) and it contains 1 percent maleic anhydride. The lubricant was TPW-113 from Struktol Company of America (Stow, OH). It is a complex blend of modified fatty acid ester.

Acetylation of Wood Flour and Fiber

The flour or fiber was oven-dried and then boiled in acetic anhydride in a 1-L glass reactor for 4 hours. The treated flour was washed and oven-dried, and its weight gain percentage was calculated. Percentage acetyl content was determined using anion exchange high-performance liquid chromatography (HPLC) with a suppressed conductivity detector. The previously described method was followed (Ibach et al. 2000).

Profile Extrusion

Profile extrusion was performed a reconfigured 32-mm twin-screw extruder. All of the components were fed into the main feed throat. HDPE was melted and then blended with the wood flour or fiber. The material was then forced through a die into 3- by 13-mm specimens for soil block testing and 19 by 19-mm specimens for field testing. Addition of 6 percent or 7.8 percent lubricant helped prevent tearing of the material as it exited the die. Blends of six different compositions were extruded (**Table 1**).

Modified Soil Block Test

Initial oven-dried weight was determined by drying for 24 hours at 105°C in a forced-draft oven, cooling in a desiccator for 1 hour, and then weighing each specimen. Specimens were preconditioned by water soaking for 2 weeks, weighed, and moisture content (MC) was calculated. At the end of preconditioning, five specimens of each blend were air-dried for 24 hours, oven-dried for 24 hours in a forced-draft oven, cooled for 1 hour in a desiccator, and then weighed. Percentage weight loss was calculated due to water soaking.

A modified soil block test procedure based on ASTM D1413 (ASTM 1999) and outlined in Clemons and Ibach (2004) was used to evaluate the specimens. Five replicates of each blend were autoclaved wet and then placed in horizontal soil bottles under one of three fungal exposure conditions:

1. No fungus (nf)
2. *G. trabeum*, a brown-rot fungus (br)
3. *C. versicolor*, a white-rot fungus (wr)

After 12 weeks of exposure, specimens were taken out, wiped to remove fungal mycelium if present, weighed, oven-dried for 24 hours at 105°C in a forced-draft oven, cooled in a dessicator for 1 hour, and weighed again. Weight loss and MC were calculated.

Flexural Testing

Third-point flexural tests were performed on oven-dry specimens according to ASTM D790 (ASTM

1990). Initial tangent modulus, maximum strength, and work to maximum stress were determined.

Field Testing

All blends (10 stakes each, 19 by 19 by 457 mm) were installed in the ground in Saucier, Mississippi on the Harrison Experimental Forest and Madison, Wisconsin at the Valley View site following AWP Standard E7-01. Stakes were evaluated annually for decay/moisture and termites. Observations such as cracking, checking, and swelling were recorded. Thickness measurements of the stakes were taken with a Mitutoyo digimatic caliper in three locations on the stake: below the ground line, at the ground line, and above the ground line (two readings each).

All blends (10 stakes each, 19 by 19 by 229 mm) were also installed aboveground horizontally on a chromated copper arsenate (CCA)-treated wood rack and evaluated by the following criteria: color change, mold/mildew, checking/cracking, and warping/distorting. Thickness measurements of the stakes were taken (two readings each).

The Mississippi site contains a sandy loam soil with an annual rainfall of 1575 mm (62 in.) per year. The Wisconsin site contains a clay loam soil with 787 mm (31 in.) of rainfall per year.

Results and Discussion

Acetylation of Wood Flour

Acetylation of the wood flour was straightforward, except for the calculation of weight gain. There was loss of fine flour particles during refluxing and removal of the acetic acid by-product by water washing, therefore, percentage acetyl content was determined using HPLC (Ibach et al. 2000). The average acetyl content of the acetylated pine flour was 22.65 percent $\pm$ 0.01 (average of 24 batches) compared with the unmodified pine flour of 1.95 percent $\pm$ 0.00 (average of 4 samples.) The acetyl content of the acetylated pine fiber was 22.98 percent $\pm$ 0.00 (average of 13 batches)

Table 1. ~ Composition of six extruded WPC blends. All percentages are weight percent.

Modification	Wood content	Wood form	HDPE content	Lubricant content	Coupling agent content
	(%)			(%)	
Control	50	Flour	44	6	--
Acetylation	50	Flour	44	6	--
Coupling agent	50	Flour	41	6	3
Control	50	Fiber	44	6	--
Acetylation	50	Fiber	44	7.8	--
Coupling agent	50	Fiber	41	6	3

compared with the unmodified pine fiber of 1.56 percent $\pm$ 0.00 (average of 2 samples.)

Modified Soil Block Test

Figure 1 shows the weight losses found in modified soil block tests. The weight losses ranged from approximately 0 to 7 percent of the sample weight or 0 to 14 percent of the wood component assuming that all weight was lost from the wood component. The largest weight losses were found for composites containing unmodified flour or fiber and without any coupling agent. Composites made with fiber tended to have slightly higher weight losses than those containing flour. The coupling agent appeared to slightly reduce the weight loss but acetylating the wood essentially eliminated all weight loss. Inoculation of the soil bottles with brown-rot fungus resulted in more than twice the weight loss as those inoculated with white-rot which is expected due to the preference of brown-rot fungus for pine wood.

The MC of the composite specimens after the water-soaking and soil bottle tests (**Fig. 2**) generally showed similar trends as the weight loss (**Fig. 1**) emphasizing the strong connection between moisture and fungal resistance. Some reduction in MC was found when a coupling agent was added but acetylating the wood led to much larger reductions. The woodfiber composites tended to absorb more moisture than the woodflour composites. This is likely because there is greater connectivity between the fibers (i.e., percolation) as a result of their higher aspect ratio than wood flour. This allows for an easier path for moisture intrusion along the fiber network. Though

similar MCs were found whether the soil bottles were inoculated with the brown-rot or white-rot fungus, weight losses for composites exposed to brown-rot were approximately double those exposed to white-rot.

For solid wood, it is necessary to keep the MC below the fiber saturation point (FSP) (about 25% to 30% moisture) to prevent fungal decay (Carll and Highley 1999). Since HDPE does not absorb moisture, the average MC of the wood flour in the composite would be about twice that shown in **Figure 2**. This suggests that all but the composites containing acetylated wood exceed or at least approach this critical MC. Additionally, since there is the possibility of a moisture gradient through the thickness of even our thin specimens due to the very slow moisture of WPCs, it is possible that even those composites with an average MC near the FSP have portions near the surface that exceed it. Since the composites with acetylated wood are well below the FSP, no weight loss was found. **Figure 3** shows this relationship between MC and weight loss.

Flexural Testing

Figure 4 summarizes the flexural moduli of the initial specimens, as well as those after the 2-week water soak (soak) and after the water soak followed by soil bottle tests with either brown-rot (soak/br) or white-rot (soak/wr) fungi. Additionally, specimens were water soaked and then placed in soil bottles that were not inoculated with fungus (soak/nf) to investigate the ef-

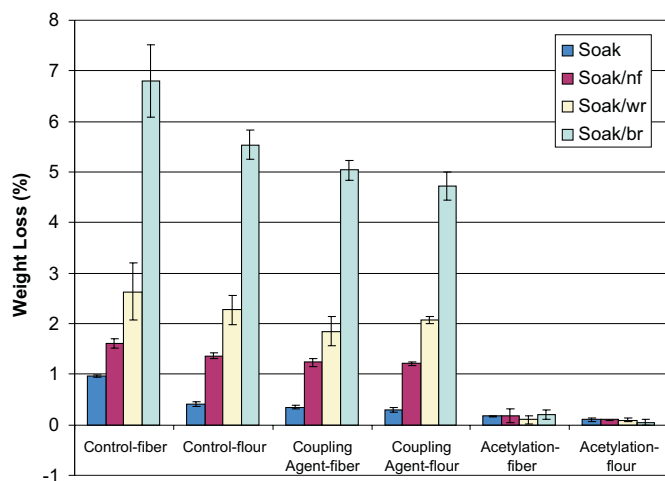


Figure 1. ~ Average percentage weight loss of six WPC blends due to water soaking and/or fungal decay after removal from a 12-week soil block test (nf = no fungus; wr = white-rot fungus, *C. versicolor*; br = brown-rot fungus, *G. trabeum*).

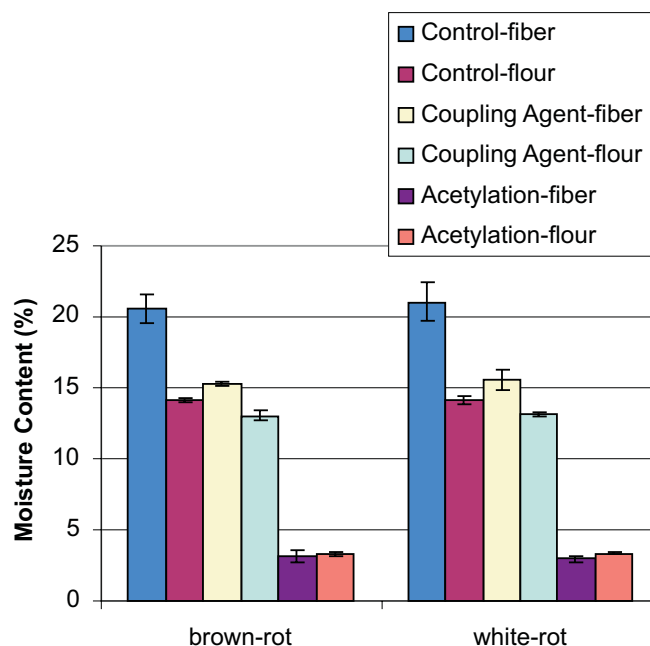


Figure 2. ~ Average MC of six blends of WPC specimens after water soaking and 12 weeks of fungal exposure (brown-rot fungus, *G. trabeum*; white-rot fungus, *C. versicolor*).

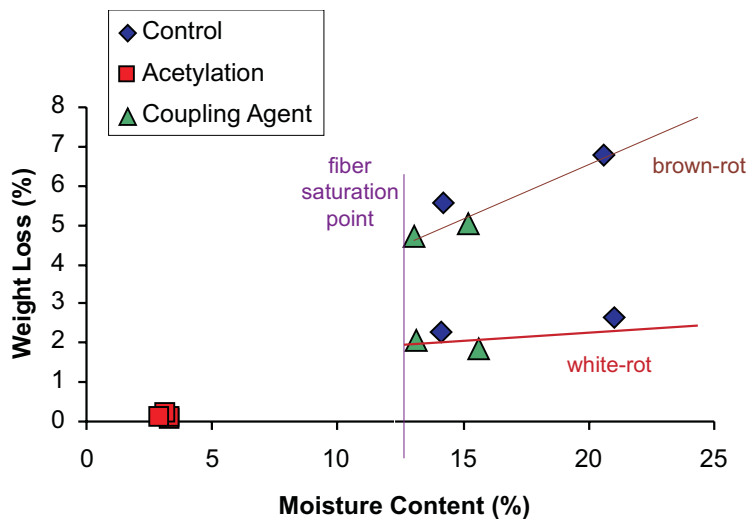


Figure 3. ~ Average MC vs. weight loss from decay of WPC specimens (both flour and fiber) after removal from a 12-week soil block test.

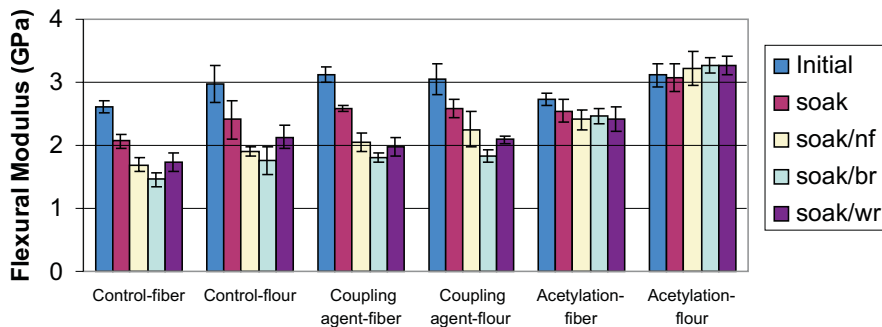


Figure 4. ~ Effects of moisture from water soaking and the soil block test on flexural modulus (nf = no fungus; br = brown-rot fungus, *G. trabeum*; wr = white-rot fungus, *C. versicolor*).

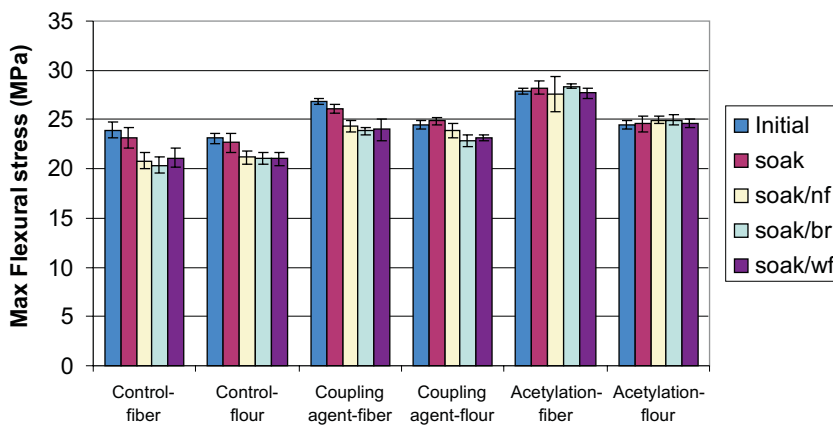


Figure 5. ~ Effects of moisture from water soaking and the soil block test on flexural strength (nf = no fungus; br = brown-rot fungus, *G. trabeum*; wr = white-rot fungus, *C. versicolor*).

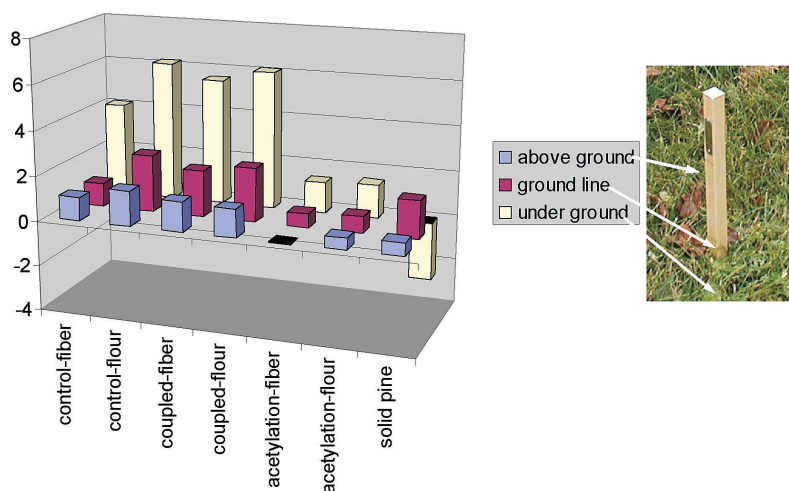
fects of further moisture sorption during the soil bottle test and to separate it from the effects of fungal attack.

The greatest reductions in flexural modulus were due to moisture sorption both during the water soak and the soil bottle tests. Since the flexural tests were performed on dried specimens, these reductions in modulus are irreversible. Exposure to white-rot fungus did not result in further reduction in modulus. This was in part due to weight losses found for the specimens that were not inoculated with fungi (Fig. 1)

which resulted in little weight loss due to white-rot decay (=1% weight loss.)

Adding a coupling agent mitigated these losses marginally at best, whereas acetylating the wood component resulted in barely detectable losses due to moisture sorption in woodfiber composites and no losses in woodflour composites. Flexural strength (Fig. 5) appeared to be considerably less sensitive to the damage from moisture sorption and fungal attack than was flexural moduli. Differences, where present,

Figure 6. ~ In-ground thickness change after 1 and 2 years at the Saucier, Mississippi site.



were less than with flexural modulus but followed similar trends.

Field Testing

Two years of field testing has been completed on composite field stakes of the same formulations as those in the laboratory tests described. Both in-ground and aboveground stakes in both Mississippi and Wisconsin are being evaluated. Untreated solid wood stakes were also included in the field tests for comparative purposes. Though these tests are long-term and further time is necessary for a more complete evaluation, some initial observations have been noted and are described below.

In-ground stakes

Figure 6 shows the thickness swell of in-ground stakes in Mississippi after 2 years. Up to a 6 percent increase in thickness was found for the composites. Not surprisingly, the largest increases were found below the ground line. The thickness decreases in the untreated solid wood stakes below ground were undoubtedly due to the severe decay that was found. The thickness increases for the composites are most likely due to moisture sorption and permanent damage. Differences between the woodfiber composites and the woodflour composites were not statistically significant nor were the differences between composites with and without coupling agent. Acetylating the wood component, however, led to significant reductions in thickness swell. This is consistent with laboratory findings where large reductions in MC were found when the wood component was acetylated (**Fig. 2**). Similar, though slightly smaller, thickness changes were found for in-ground composite stakes in Wisconsin.

In-ground stakes were visually rated from 0 to 10, with 10 having no decay. **Figure 7** summarizes the decay ratings for the first 2 years in Mississippi. After the first year, only the wood stakes had a rating below 10.

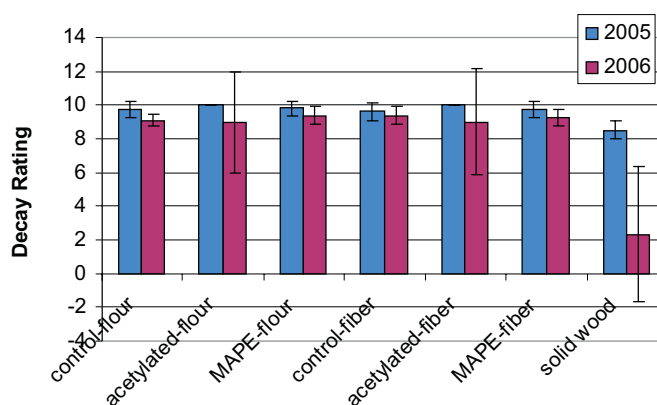


Figure 7. ~ In-ground decay ratings after 1 and 2 years of exposure at the Saucier, Mississippi site.

After the second year, some of the composite stakes are starting to decay but it will take several more years before trends become evident. In-ground composites in Wisconsin have not shown any signs of decay yet.

Though subterranean termites are present in Mississippi, only very minor attacks were found on the unmodified control WPCs, whereas major termite attacks (visual rating of 2.5 out of 10) were found for solid wood. No termites are present at the Wisconsin site.

Aboveground stakes

Aboveground stakes were mounted on racks and visually rated (0 to 10) for each of five criteria:

1. cracking and checking,
2. color change,
3. decay,
4. mold and stain, and
5. termite attack (Mississippi only).

Figures 8 and 9 summarize the 2-year ratings. No cracking/checking, decay, or termite damage has been found after 2 years at either site.

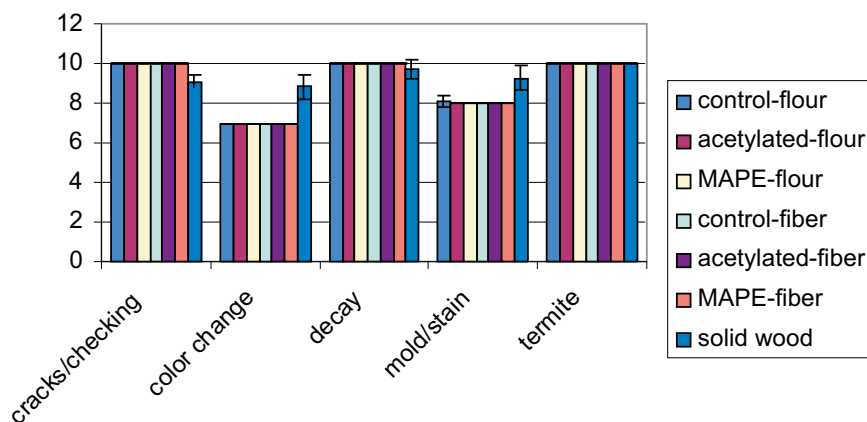


Figure 8. ~ Aboveground ratings after 2 years of outdoor exposure in Saucier, Mississippi.

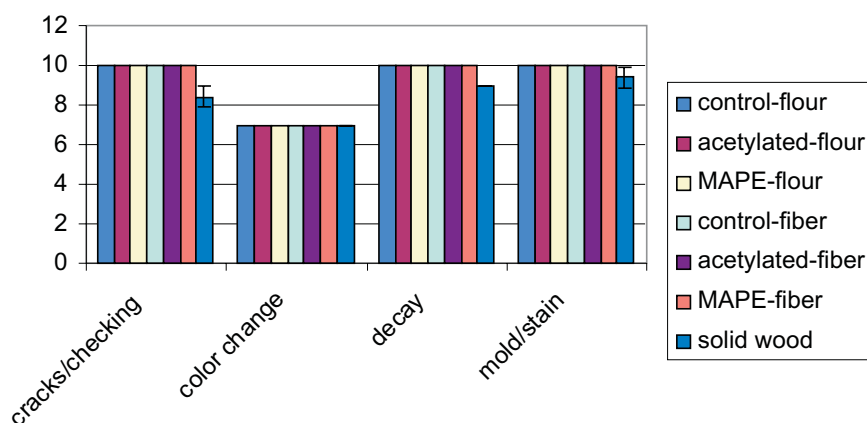


Figure 9. ~ Aboveground ratings after 2 years of outdoor exposure at the Valley View site in Madison, Wisconsin.

In both Mississippi and Wisconsin, color change was significant and considerably greater for the composites when compared with the solid wood. This is not surprising since colorant or light stabilizers were not added to the composites. All composites have faded similarly. After 2 years, mold and stain growth were found on the composites at the Mississippi site only, which typically gets twice the rainfall as the Wisconsin site. Despite the potential for moisture reduction, acetylating the wood component did not appear to inhibit the formation of mold on the surfaces of the composites.

Conclusions

Several methods of moisture reduction were investigated through both laboratory and field testing. Acetylating the wood component in WPCs reduced moisture sorption and related performance losses such as decay and flexural property loss in laboratory tests. Reduction in moisture sorption and decay with addition of coupling agent were significant but not as large as acetylation. Reductions in mechanical properties during soil block tests appeared to be primarily due to moisture sorption and not fungal attack.

Early findings in field tests were consistent with laboratory results but considerably more time is

needed to adequately assess the different formulations. Acetylating the wood component of WPCs significantly reduced belowground thickness increases due to moisture sorption. After 2 years, decay and termite attack on in-ground stakes on the composites were minimal despite heavy attack on untreated solid wood. In aboveground tests, similar color changes were found for composites at both sites. Mold grew on aboveground stakes only at the Mississippi site. Despite the potential for moisture reduction, acetylating the wood component did not appear to inhibit the formation of mold on the surfaces of the composites.

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