Combined Ultraviolet and Water Exposure as a Preconditioning Method in Laboratory Fungal Durability Testing

Rebecca E. Ibach, Craig M. Clemons, and Nicole M. Stark

Abstract

During outdoor exposure, woodfiber-plastic composites (WPC) are subject to biological, moisture, and ultraviolet (UV) degradation. The purpose of laboratory evaluations is to simulate outdoor conditions and accelerate the testing for quicker results. Traditionally, biological, moisture, and W laboratory tests are done separately, and only combined in outdoor field exposure. This research evaluates WPCs exposed to a combination of UV, moisture, and fungal degradations. Due to the slow moisture sorption of WPCs, the ASTM D 1413 standard laboratory soil block test was modified to include preconditioning by W and water.

WPCs consisting of 50 percent western pine wood flour and 50 percent high-density polyethylene (HDPE) were extruded into 30 by 140 mm (1.2 by 5.5 in.) radius edge deck boards. Four blends were extruded containing the following additives by weight percentages:

1. control;
2. 1 percent zinc borate;
3. 1.5 percent W stabilizer package; and
4. 1 percent zinc borate + 1.5 percent UV package.

Specimens (3 by 13 by 89 mm) were cut from the boards and exposed to one of two preconditioning methods:

1. 2-week water soak or
2. 1,000 hours in a weatherometer plus a 2-week water soak.

After the 2-week water soak the autoclaved "wet" specimens were placed in a modified soil block test against the brown-rot fungus *Gloeophyllum trabeum* for 12 weeks. Moisture content and weight loss of the specimens were determined after 12 weeks. Matched field stakes were placed above and in-ground in Madison, WI, and Saucier, MS, for ultimate comparison.

Ibach:
Research Chemist
Clemons:
Research General Engineer
Stark:
Chemical Engineer, USDA Forest Service, Forest Products Laboratory, Madison, Wisconsin, USA

Introduction

Woodfiber-plastic composite (WPC) products for exterior applications are growing not only for decking materials, but railings, windows, siding, and roofing as well. The introduction of WPCs into the outdoor environment has led to concerns about durability, and some failures have been found in the field (1).

The use of plastics in WPCs is seen as a low maintenance way to improve the durability of wood by at least partially encapsulating the wood. However, the actual lifetime of WPC lumber is uncertain. As composites are considered for applications that need structural performance, and as more fiber is added to lower raw material cost and increase stiffness, durability information is critical (2).

If wood is kept dry it will not decay, but with the right conditions of moisture, temperature, and fungal spores, nonresistant species will decay (3). Therefore, if the wood component in WPCs reaches the same conditions, untreated WPCs will also decay. Currently, manufacturers rely on the moisture resistance provided by the plastic component or fungicides such as zinc borate to prevent decay in their products.

Since WPCs are often used in applications dominated by wood, they are often evaluated using existing wood standards, which may not be appropriate. For example, when testing additives for improving fungal durability, the fact that WPCs absorb moisture slowly and can take months to equilibrate makes laboratory decay testing difficult using existing wood standards (4).

Modification of the ASTM D1413 standard laboratory soil block test has previously been presented (5-8). Two preconditioning procedures were investigated to accelerate the moisture sorption of the composite specimens: 2-week water soak or cyclic boiling and drying. Weight loss, moisture content, and flexural tests were performed. The compression molded and injection molded specimens had little weight loss (0 to 3%), whereas the extruded specimens had higher weight losses: 6 percent for the water soaked and 22.8 percent for the boiled specimens.

To determine the long-term outdoor durability of WPCs, it is critical to understand the combined effects of biological agents, ultraviolet (UV) energy, and moisture on the composites. The work presented in this paper moves research in this direction. UV exposure in a weatherometer was performed as a preconditioning method as well as water soaking, and then placement of all specimens into the soil block test "wet" to determine the fungal durability of WPCs manufactured on a commercial-scale extruder. The influences of a fungicide and UV stabilizer package were investigated to determine their effects alone and in combination.

Materials and Methods

The following materials/chemicals were used to produce the WPC deck boards. All percentages are on a weight basis.

- Plastic (HDPE): High-density polyethylene (BP Solvay Polyethylene, grade A60-70-162; melt flow index (MFI) approximately 0.72).
- Wood flour (WF): Mostly ponderosa pine flour, -40, +80 mesh (American Wood Fibers grade 4020).
- Lubricant (Lub): 8 percent of a blend of calcium stearate, ethylene bistearamide, and a proprietary amide (Struktol Company of America, Struktol TR 016).
- Fungicide (ZnB): 1 percent zinc borate (U.S. Borax, Boragard ZB).
- Light stabilizer package (UV): 0.5 percent of a hydroxyphenylbenzotriazole light absorber (Ciba Specialty Chemicals, Tinuvin 328) and 1 percent of a zinc ferrite colorant (Holland Colors Americas, Holcobatch Yellow L25153)

A simple 2^2 factorial design was used to investigate the effects of several additives on fungal resistance. All blends used HDPE as the matrix material and contained 50 percent WF and 8 percent lubricant. Additionally, three of the four blends contained additives. The blends are as follows:

1. no additives
2. ZnB only
3. W stabilizer package only
4. ZnB and W stabilizer package

Processing

Commercial-scale extrusion trials were run at the University of Maine's Advanced Engineered Wood Composite Center. Processing parameters were determined and 30 by 140 mm (1.2 by 5.5 in.) radius edge deck boards were produced using an in-line twin-screw extruder profiling system on a 94-mm profiling extruder (Davis Standard, Woodtruder). Due to the low density of the ZnB and W absorber, concentrates containing 10 percent by weight active ingredient in HDPE were compounded at the USDA Forest Products Laboratory on a 32-mm compounding extruder (Davis Standard, D-TEX) to facilitate feeding of the additives during the trials.

Specimen Preconditioning

Specimens (3 by 13 by 89 mm) were cut from the boards, ovendried at 105°C for 24 hours, cooled 1 hour in a desiccator, weighed, and then exposed to one of two preconditioning methods:

1. Water soak (S):
   Specimens were vacuum impregnated with reversed osmosis water. This consisted of a 30-minute vacuum, a 5-minute hold, then soaking in water for 2 weeks; the water was changed daily. The 2-week water soak was started exactly 2 weeks before placement into the soil bottles. The specimens were weighed after 2-weeks of water soaking and the moisture content was calculated. Five specimens of each blend were ovendried to determine weight loss from water soaking or weathering and water soaking.
2. Weatherometer and then water soak (WS):
   Specimens were exposed in an Atlas 6500 XEII Weatherometer for 1,000 hours (42 days). This instrument uses a borosilicate inner and outer filter, with an irradiance of 202 KJ/m^2 (52.0 W/m^2) of energy on the specimens. ASTM D2565 standard was followed. The exposure cycle consisted of 102 minutes of W light exposure (20.4 hours/day) and 18 minutes of simultaneous water spray and W light exposure (3.6 hours/day). Specimens were rotated every 200 hours from the top to the middle to the bottom to maintain UV exposure for all of the specimens. After 1,000 hours exposure, all specimens were examined for damage, color, and weight loss. They were then water soaked as described above.

Biological Evaluations

After conditioning, the specimens were autoclaved "wet" and then placed in a modified soil block test against the brown-rot fungus *Gloeophyllum trabeum* for 12 weeks. The modification included flipping the bottles horizontally and having a larger feeder strip to accommodate the tensile size specimen *(6)*. Five specimens were run for each blend and preconditioning combination. Soil bottles not inoculated with fungi were also prepared and tested to determine the moisture uptake during the tests. Untreated southern pine sapwood specimens were run to determine fungus viability.

After the 12-week soil block test, specimens were removed, cleaned of fungus, and weighed wet. Specimens were airdried overnight, ovendried at 105°C for 24 hours, cooled in a desiccator for 1 hour, and then weighed again. Moisture content of specimens after the soil block test and the ovendried weight loss were calculated. The weight loss due to fungal decay was determined by subtracting the weight losses (ovendry basis) due to preconditioning (S and WS) and weight losses, if any, found during tests in uninnoculated soil bottles.

Table 1. – *The actual chemical composition (weight percentage) of commercial scale extrusion trials made at the University of Maine.*

	Blend #			
	1	2	3	4
Wood flour	48.9	48.2	51.9	50.5
HDPE	43.1	43	38.3	39
Lubricant	8	7.8	8.4	8.2
ZnB	--	1.1	--	0.9
Light absorber	--	--	0.5	0.5
Colorant	--	--	0.9	0.9

Microscopy Analysis

Scanning electron microscopy (SEM) was performed on selected specimens with a Joel 840 instrument. The surface and cross sections were gold coated and representative pictures taken.

Results and Discussion

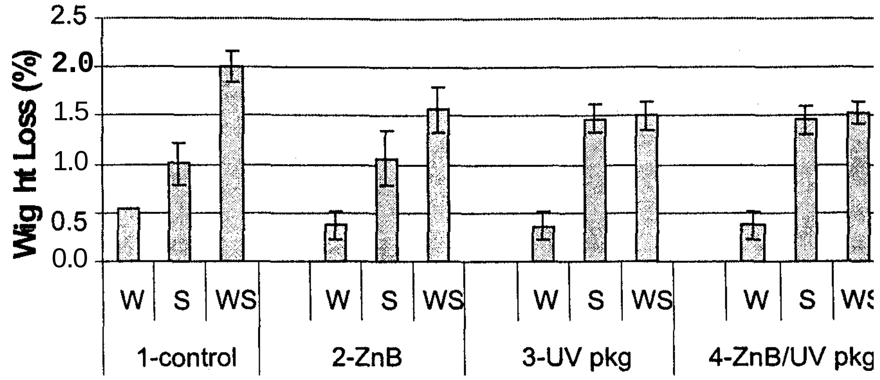
Processing

The actual weight percentages of the composition of the deck boards made at the University of Maine are in **Table 1.** During commercial-scale extrusion trials, it is difficult to weigh precise amounts of materials. Therefore, the wood flour varies from 48.2 percent in blend 2 to 51.9 percent with blend **3.** These slight differences can influence the moisture content and weight losses to a small degree.

Specimen Preconditioning

The average weight losses and standard deviations of the specimens due to preconditioning by weathering (W), soaking (S), and weathering then soaking (WS) are shown in **Figure 1.** The weight loss due to W was less than 0.5 percent for all four blends indicating that after **1,000** hours of UV/water spray the additives had little effect on weight loss. For the untreated control (blend 1), the S had 1 percent and the WS had 2 percent weight loss. Without any additives the WS control specimens had the most weight loss from preconditioning.

Figure 1. – *The average weight loss of specimens after preconditioning. W = weathering, S = watersoaking, and WS = weathering then water soaking.*



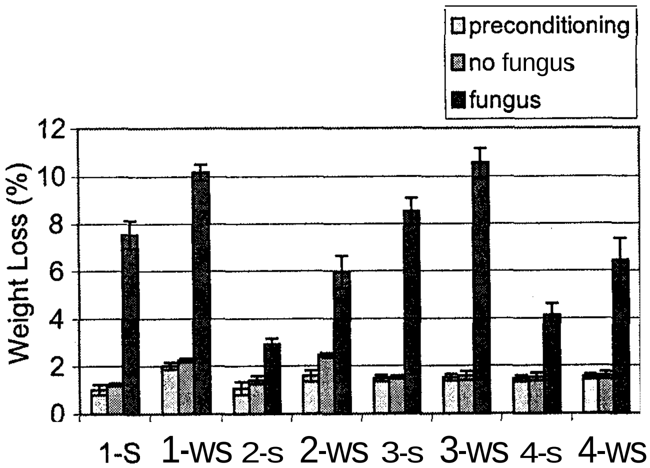


Figure 2. – *The average weight loss after preconditioning or fungal decay testing.*

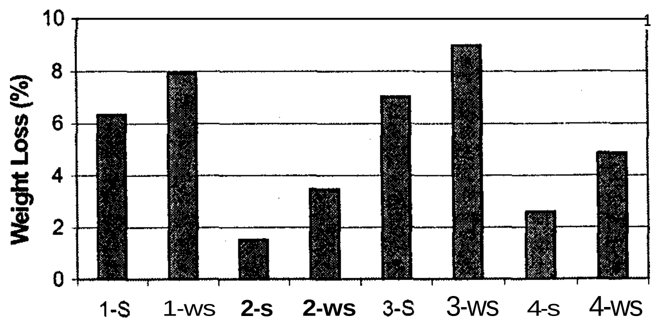


Figure 3. – *The average weight loss after a 12-week modified soil block test. (No fungus correction.)*

The ZnB (blend 2) had weight losses of 1 percent with S and 1.5 percent with WS, but with the standard deviations, they are similar. Both blend 3 (UV) and 4 (ZnB/UV) had 1.5 percent weight loss from either S or WS. The additive blends (2-4) all had similar weight losses from preconditioning with either S or WS. These small weight losses from preconditioning may be due to the degradation from weathering and/or the water soluble components of the wood flour and/or additives.

Biological Evaluations

Weight loss

Southern pine solid wood was run in the decay test to monitor fungal activity. The solid wood untreated controls had 41 percent weight loss after the 12-week exposure to G. ***trabeum.*** This showed that the fungus was viable.

Figure 2 shows the percentage weight loss of the specimens from preconditioning, soil block testing with uninoculated bottles (no fungus), and decay testing with the brown-rot fungus G. ***trabeum*** (fungus). No weight losses were found for the uninoculated specimens except for a small weight loss in blend **2-WS.** The reason for the small weight loss is not clear.

Figure 3 shows the weight loss due just to decay by G. ***trabeum*** found by comparing the weight losses in the inoculated and uninoculated tests (no fungus correction.) Weight losses are given for the total composite. If one only takes into account the wood portion, then the weight losses would be approximately double **(Table 1).** During the soil block tests, the WS specimens lost more weight than the S specimens did in all four blends. This is probably due to the added effect of the 1,000 hours of UV/water weathering exposure, providing additional pathways for moisture sorption and fungal hyphae. Blends **2** and 4 (both with ZnB) had lower weight losses than blends 1 (no additives) and **3** (with W package only). The W package did not

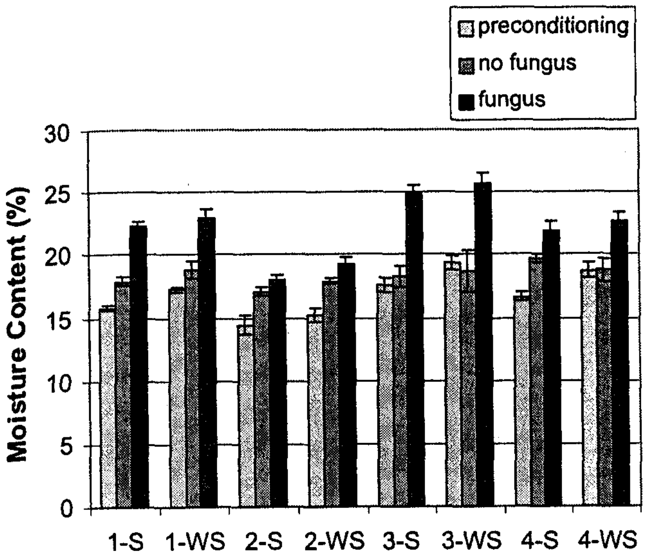


Figure 4. ~ *The average moisture content ofthe specimens after preconditioning and then fungal decay testing with G. trabeum.*

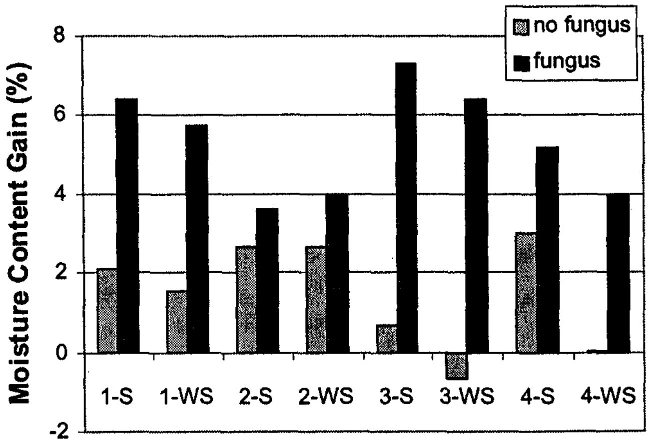


Figure 5. ~ *The average moisture content gained during the 12-week soil block test.*

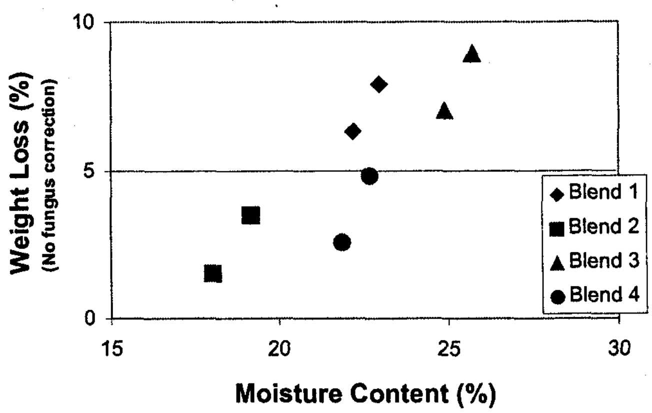


Figure 6. ~ *The correlation between weight loss and moisture content after exposure to G. trabeum in the modified soil block testfor 12 weeks.*

provide any fungal resistance. Normally in a soil block test with solid wood, specimens with weight losses of less than 5 percent are considered resistant to fungal attack. Using this criterion, the blends containing ZnB (blends 2 and 4) would be considered resistant to fungal attack. The blends containing ZnB had less fungal weight loss, and the W package alone had greater fungal weight loss than the controls containing no additives. There appeared to be no interaction between ZnB and the W package indicating that the two additives did not interfere with each other.

Moisture content

Figure 4 shows the average moisture content of the specimens after preconditioning and then fungal decay with *G. trabeum.* The average moisture content of each blend was greater than 14 percent after preconditioning alone. The WS specimens had a slightly higher moisture content compared with the S. This was expected because additional moisture exposure as well as UV degradation on the specimens leads to more moisture sorption. The specimens exposed to fungus have higher moisture contents especially in those blends not containing ZnB (blends 1 and 3). This was also expected since the fungus will transport water into the specimens, and they had the highest weight losses due to decay. The preconditioning of the ZnB specimens resulted in less moisture sorption, and the W package alone had increased moisture sorption compared to the controls.

Figure 5 shows the average moisture content gained during the 12-week soil block test. All four blends show higher moisture gains compared with the no fungus controls. Blends 1 and 3 had the highest moisture gains after exposure to fungus, followed by blend 4 and then blend 2. This is consistent with the weight loss data. The greater the weight loss due to decay, the higher the moisture content. The moisture contents at the end of soil block testing were less with the ZnB and more with the W package than the controls.

Figure 6 is a plot of the correlation between weight loss (from **Fig. 3** with no fungus correction) versus moisture content of specimens after expo-

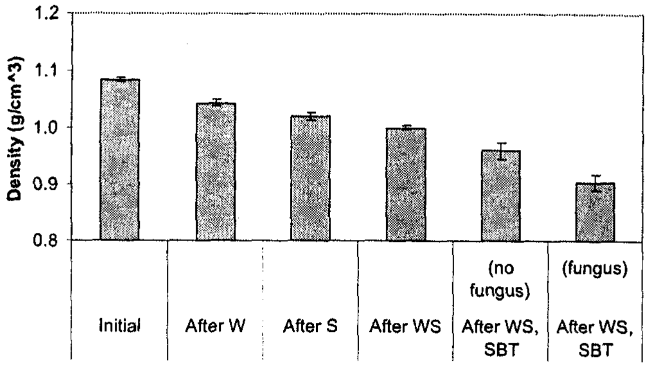


Figure 7. ~ *Density reduction after various exposures with blend 1. SBT = soil block test.*

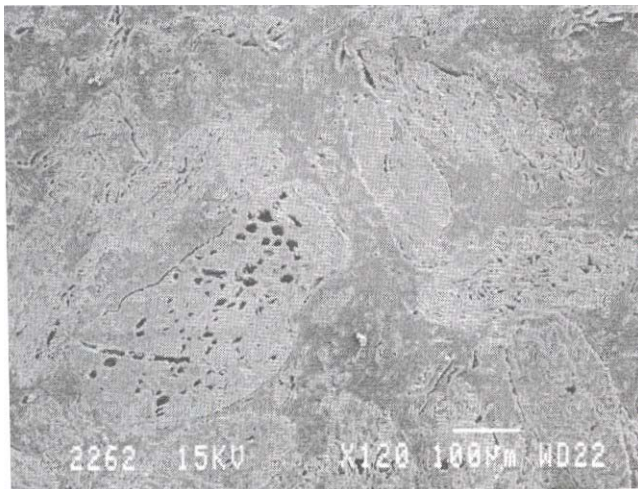


Figure 8. ~ *SEM micrograph ofthe core ofa cross section ofblend 3 (UV package) with no preconditioning or decay (120x).*

sure to *G. trabeum* in the modified soil block test for 12-weeks. A wood moisture content of 25 to 30 percent is necessary for fungal decay (9). Since all of these specimens had moisture contents above 12.5 percent (25% wood flour moisture content assuming all moisture is absorbed by the wood flour) conditions for decay were attained. Adding the fungicide ZnB reduced the weight loss during the decay tests (compare blends 2 and 4 with blends 1 and 3). Additionally, blend 4 with ZnB had lower weight losses than blend 1 at equivalent moisture contents.

Figure 7 shows the densities of the specimens at each stage of exposure for blend 1. For each blend there was a decrease in density with preconditioning and soil block exposure. Each blend showed a similar trend. There was less of a decrease in the

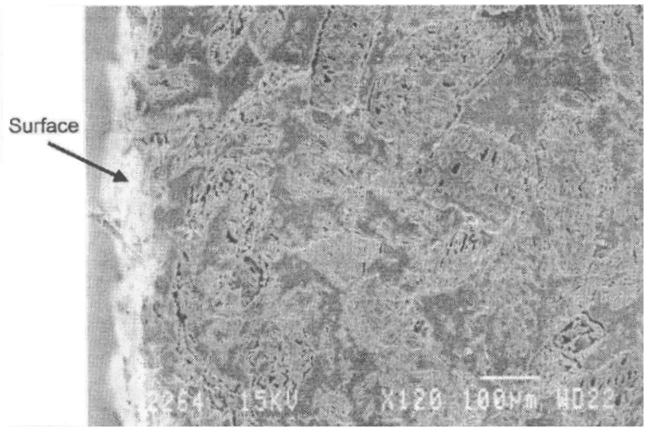


Figure 9. ~ *SEM micrograph of the surface of a cross section ofblend 3 WS after exposure to G. trabeum. (UV package, weathered, soaked, decayed, 120x.)*

density of the ZnB (S and WS preconditioned) and the weathered W blend specimens. The greatest density decrease was with the fungal decayed specimens of blend 3-WS (0.888 g/cm^3 ± 0.012). This is consistent with the weight loss due to decay data. There appears to be no interaction between the ZnB and W chemicals for either the preconditioning or soil block testing.

Microscopy Analysis

Assessing damage to the surface of the specimens due to preconditioning or decay with SEM proved difficult since the surfaces were rough due to the planing used when cutting the soil block specimens from the deck boards. Therefore, cross sections were prepared for evaluation. **Figure 8** is an SEM micrograph (120x) of the cross section of blend 3 (containing UV package only) that has not been preconditioned or decayed. Some of the wood flour lumens are unfilled, filled, or collapsed. In comparison, **Figure 9** is a cross section of blend 3 that has been weathered, water soaked, and then decayed. This specimen had similar features such as unfilled, filled, and collapsed lumens in the center of the specimen, but looking closer at the surface on the left there was disruption or break down of the wood flour. This was expected due to the 1,000 hours of weathering, 2-week water soaking, and then 11 percent weight loss from decay. **Figure 10** shows a picture of a decayed area at a magnification of 1,800x. Fungal hyphae can be seen. **Figure 11** is the surface of blend 3 showing the effect of W degradation from weathering producing a chalky appearance.

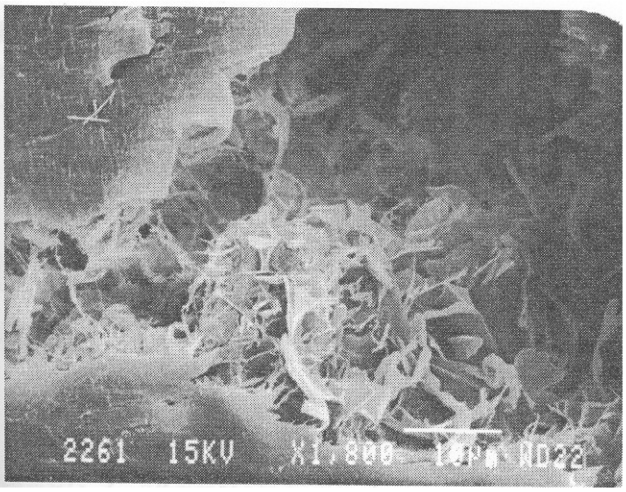


Figure 10. ~ *SEM micrograph of fungal degradation ofa cross section of blend 3 taken near the surface ofthe specimen (1,800x).*

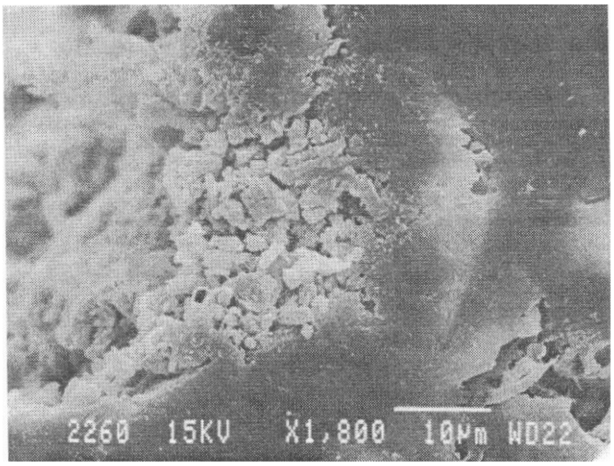


Figure 11. ~ *SEM micrograph of UV degradation of a cross section ofblend 3 taken at the surface ofthe specimen (1,800x).*

Conclusions

All total specimen moisture contents were at least 14 percent or more after preconditioning by water soaking. Placement of specimens directly into the 12-week soil block test at this moisture content accelerated the decay process. Preconditioning WPC specimens by weathering and then water soaking gave decay weight losses greater than conditioning by just water soaking. When exposed to *G. trabeum*, blends containing ZnB had lower weight losses than those with no additives or with W package only after exposure to decay fungi. There was no significant interaction between ZnB and the W package. The density decreased and moisture content increased during preconditioning and soil block testing, especially upon fungal exposure.

Color assessment and flexural tests on these specimens are in progress. The goal is to determine the effects of several additives on the susceptibility to fungal attack of WPCs in a laboratory, but long-term field studies are needed to corroborate these results. Hence, field-testing is in progress.

Acknowledgments

The authors gratefully acknowledge Sandra Lange, Rebecca Schumann, Ben Henderson, and Tom Kuster of the Forest Products Laboratory for their assistance with this research. Thanks also to the following companies for supplying materials: American Wood Fibers, Holland Colors Americas, Struktol Company of America, and U.S. Borax.

Literature Cited

1. Morris, P.I. and P. Cooper. 1998. Recycled plastic/wood composite lumber attacked by fungi. Forest Prod. J. 48(1):86-88.
2. Verhey, S. and P. Laks. 2002. Wood particle size affects the decay resistance of woodfiber/thermoplastic composites. Forest Prod. J. 52(11/12):78-81.
3. Wood Handbook: Wood as an Engineering Material. 1999. FPL-GTR-113. Madison, WI: USDA Forest Service, Forest Products Laboratory. p. 3-15.
4. Stark, N. 2001. Influence of moisture absorption on mechanical properties of wood flour-polypropylene composites. Journal of Thermoplastic Composite Materials. 14(5):421-432.
5. Ibach, R.E., R.M. Rowell, S.E. Lange, and R.L. Schumann. 2002. Effect of wet-dry cycling on the decay properties of aspen fiber high-density polypropylene composites. In: 6th International Conf. on Woodfiber-Plastic Composites. Forest Products Society. Madison, WI, pp. 267-270.
6. Clemons, C.M. and R.E. Ibach. 2002. Laboratory tests on fungal resistance of wood filled polyethylene composites. *In*: ANTEC Proceedings. San Francisco, CA. Society of Plastics Engineers. Vol. 11. pp. 2219-2222.
7. Ibach, R.E. and C.M. Clemons. 2002. Biological resistance of polyethylene composites made with chemically modified fiber or flour. *In*: 6th Pacific Rim Bio-Based Composites Symposium. Portland, OR: Oregon State University. Vol. II. pp. 574-583.
8. Clemons, C.M. and R.E. Ibach. 2002. Fungal exposure of wood filled polyethylene composites: laboratory evaluation. *In*: Progress in Woodfibre-Plastic Composites Conference. Toronto, Canada.
9. Carll, C.G. and T.L. Highley. 1999. Decay of wood and wood-based products above ground in buildings. Journal of Testing and Evaluation. 27(2):150-158.

Seventh International Conference on Woodfiber-Plastic Composites
(and other natural fibers)

May 19–20, 2003
Monona Terrace Community & Convention Center
Madison, Wisconsin, USA

Sponsored by the USDA Forest Service in cooperation with the
American Chemical Society's Cellulose and Renewable Materials Division,
University of Wisconsin's Polymer Engineering Center,
University of Toronto, Materials & Manufacturing Ontario,
and the Forest Products Society.

Hosted by the USDA Forest Service, Forest Products Laboratory.

Forest Products Society
2801 Marshall Court
Madison, WI 53705-2295
phone: 608-231-1361
fax: 608-231-2152
www.forestprod.org