

Effects of processing method and moisture history on laboratory fungal resistance of wood-HDPE composites

Craig M. Clemons
Rebecca E. Ibach

Abstract

The purpose of this study was to clarify the effects of composite processing and moisture sorption on laboratory fungal resistance of wood-plastic composites. A 2-week water soaking or cyclic boiling-drying procedure was used to infuse moisture into composites made from high-density polyethylene filled with 50 percent wood flour and processed by extrusion, compression molding, or injection molding. Extruded composites absorbed the most moisture; compression-molded composites absorbed less than did extruded composites, and injection-molded composites absorbed the least. Although more moisture was absorbed during water soaking, the cyclic boiling procedure caused more damage to the composites, especially the extruded ones. In neither procedure for infusing water did the composites reach equilibrium. A standard method for determining the durability of structural wood was modified for testing the fungal resistance of composites. Moisture content, flexural properties, and weight loss were measured over a 12-week exposure to the brown-rot fungus *Gloeophyllum trabeum*. Significant weight losses were found once the composite moisture content reached roughly 12 to 15 percent. This corresponds to an average wood flour moisture content near the fiber saturation point. The greatest weight losses were found for extruded composites that had been preconditioned by boiling. Damage to the composite from moisture sorption complicated the use of flexural performance as a measure of fungal attack. Mechanical performance appeared to be a less sensitive measure of fungal attack than was weight loss. However, flexural modulus loss and strength loss due to fungal attack correlated well with weight loss for individual specimens when the data were plotted together.

Traditionally, inorganic materials such as fiberglass, calcium carbonate, and talc have been combined with plastics to make inexpensive composites for a wide range of applications. More recently, wood and other lignocellulosic fibers have been combined with thermoplastics, such as polyethylene, polypropylene, and polyvinyl chloride, to make wood-plastic composites (WPCs).

Building applications are the largest and fastest growing market for WPCs (Leaversuch 2000, Mapleston 2001). Besides decking, products such as fencing, industrial flooring, landscape timbers, railings, and moldings are also produced. Additionally, new building products using WPCs, such as roof shingles, siding, and waterfront applications, have

recently been commercialized or are being developed (Clemons 2002). Using plastics in wood composites is seen as a low maintenance way to improve the durability of wood by at least partially encapsulating it in plastic.

The exact durability of WPCs is to a great extent unknown, and questions remain as to how resistant these materials

are to environmental factors. Little consistent information on fungal resistance is available in the literature. Morris and Cooper (1998) reported fungal growth on WPC decking in-service in Florida after 4 years. Mankowski and Morrell (2000) evaluated several proprietary WPCs by laboratory soil block tests. Weight loss varied from 0.4 to 20.4 per-

The authors are, respectively, Research General Engineer; and Research Chemist, USDA Forest Serv., Forest Prod. Lab., One Gifford Pinchot Drive, Madison, WI 53726-2398. This research was funded in part by a grant from the Partnership for Advancing Technology in Housing (PATH). The authors gratefully acknowledge Rebecca Schumann, Brian Destree, Andrew Isham, Sandy Lange, and Beom-Goo Lee of the Forest Products Laboratory for their assistance with fungal resistance testing and flexural testing. The wood flour was supplied by American Wood Fibers, Schofield, WI. This paper was received for publication in November 2002. Article No. 9579.

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cent depending on the fungi used and type of composite. Researchers have also investigated the fungal resistance of model composites, but the literature to date is far from conclusive. Khavkine et al. (2001) found little weight loss caused by fungal attack for polyethylene composites containing 40 to 70 percent wood, despite good fungal colonization on the composite surfaces and a conditioning procedure that included oven-drying at 105°C for 24 hours, a 2-hour boil, and a 24-hour water soak. However, using a modified soil block procedure, Verhey et al. (2001a) found significant weight loss in composites containing 60 percent or greater wood content. Pendleton et al. (2002) evaluated more complex formulations. Of the formulations not containing zinc borate, weight loss occurred if wood content was 53 percent or greater.

The confusing results are not surprising considering the widely varying formulations evaluated (i.e., different types and quantities of fiber, plastic, and additives), as well as different processing and test methodologies. Further complicating matters is the fact that WPCs absorb moisture slowly and can take months to equilibrate (Stark 2001). This slow moisture sorption makes some short-term testing problematic and the use of standard short-term fungal resistance tests questionable. In addition, moisture sorption can result in irreversible damage to the composite as a result of differential swelling strains between the wood and plastic components (Klason et al. 1984, Peyer and Wolcott 2000, Rangaraj and Smith 2000). This irreversible damage makes it more difficult to separate the moisture and fungal effects when mechanical performance is investigated because moisture is absorbed during fungal resistance testing (Verhey et al. 2001b).

This investigation was undertaken to clarify the effects of composite processing and moisture sorption history on laboratory fungal resistance of WPCs.

Methods and materials

Materials

The plastic was a reprocessed high-density polyethylene (HDPE) from milk bottles with a melt flow index of approximately 0.7 g/10 minutes at 190°C and 2.16 kg. The filler was nominal 40-mesh western pine wood flour from American Wood Fibers (Schofield, Wisconsin).

Specimen preparation

HDPE composites containing 50 percent wood flour were produced by three methods: 1) injection molding; 2) compression molding; and 3) profile extrusion.

Injection molding. — Wood flour was dried and then compounded with HDPE in a 32-mm compounding twin-screw extruder (Davis Standard, Pawcatuck, Connecticut). The plastic was melted in the first section of the compounding extruder. The wood flour was added downstream and blended (compounded) into the molten plastic. The molten material was then forced through a strand die cooled in a water slide and pelletized. The compounded pellets were dried at 105°C for at least 4 hours prior to injection molding into flexural specimens of standard size (3 by 13 by 127 mm, per ASTM D 790 [ASTM 1990a]) using a 33-ton reciprocating-screw injection molder (Cincinnati Milacron, Batavia, Ohio). In the injection molding process, the pellets were melted and forced into a cold mold. After solidification, the parts were ejected from the mold. The end opposite the gate was cut from the specimens prior to testing for fungal resistance so that the specimens could fit in a soil bottle placed on its side. Final specimen length was 89 mm.

Compression molding. — Wood flour was compounded with HDPE in a 1-L high-intensity thermokinetic mixer (K Mixer, Synergistics, Inc., St. Remi de Napierville, Quebec). The thermokinetic mixer was a simple batch mixer in which several high-speed blades supply the energy to melt the polymer and blend the material. An infrared sensor monitored the material temperature. Batches of 60 g each of HDPE and wood flour were processed at 5,500 rpm (rotor tip speed of about 30 m/sec.) with a discharge temperature of 170°C and a batch time of about 150 seconds. After the blended material was discharged, it was hot-pressed at 180°C to a 3-mm thickness for 1 minute and then cooled in the press until solidified. The desired board thickness was achieved by pressing the material to a metal 3-mm-thick ring with sufficient inner diameter to accommodate the batch volume. Teflon-coated cauls were used to aid in separating the boards after pressing. Test specimens (13 by 89 mm) were cut from the pressed boards.

Profile extrusion. — The 32-mm twin-screw extruder was reconfigured for profile extrusion. Wood flour and HDPE were added into the main feed throat. The plastic was melted and the wood flour blended into the molten plastic in the first stage of the extruder. The molten material was then shaped to a 19-by 19-mm square cross section by forcing it through a die at the end of the extruder. A sizing die, water spray tank, and puller were used to solidify the plastic in the desired shape and continuously remove it from the extruder. An addition of 3 percent lubricant (TR251, Struktol Company of America, Stow, Ohio) was necessary to prevent tearing of the melt as it exited the die. The lubricant was added downstream to minimize migration to the wood-plastic interface. Test specimens (3 by 13 by 89 mm) were cut from the extruded profile for fungal testing; each specimen had six cut sides.

Injection-molded and compression-molded composites contained HDPE with 50 percent wood flour by weight. Extruded composites contained HDPE with 50 percent wood flour and 3 percent lubricant. Specimens cut from southern pine sapwood were also prepared for comparison with wood-HDPE composites and to evaluate fungal activity.

Fungal resistance testing

To investigate fungal resistance of WPCs, ASTM D 1413 (ASTM 1990b) was modified by: 1) changing specimen size; 2) measuring weight loss after 4, 8, and 12 weeks of exposure; 3) adding flexural tests; and 4) measuring moisture content (MC).

Specimen size was changed from 19 by 19 by 19 mm to 3 by 13 by 89 mm to conform to span and depth requirements of flexural test method ASTM D 790-84 (ASTM 1990a). The 89-mm length allowed the specimen to fit into a standard soil bottle turned on its side. Longer feeder strips and several fungal inoculations along the specimen length were also necessary. The decrease in specimen thickness increased the surface-to-volume ratio, facilitating moisture sorption and increasing fungal exposure area.

Two preconditioning procedures were investigated to accelerate the moisture sorption of the composite samples: 1) 2 weeks of ambient water soak according to the leaching method in ASTM D 1413 (ASTM 1990b); and 2)

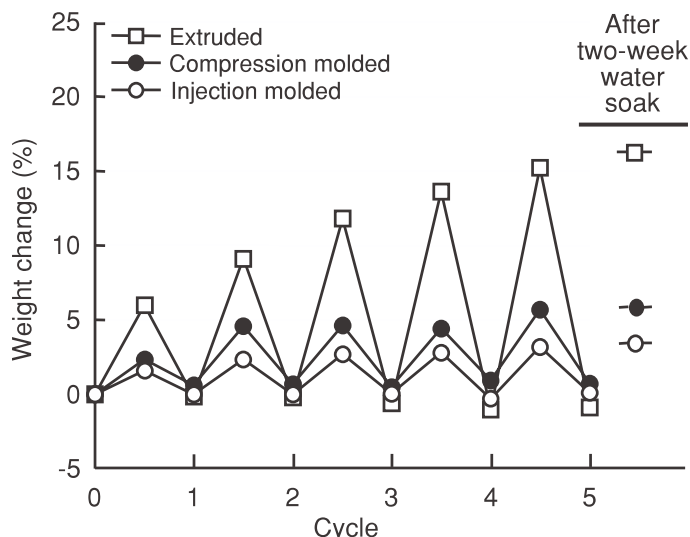


Figure 1. — Moisture sorption of HDPE filled with 50 percent wood flour during five cycles of 2 hours boiling and 22 hours oven drying. Error bars removed for visual clarity.

cyclic boiling and drying consisting of 5 cycles of a 2-hour boil followed by 22 hours of oven drying at 105°C. After preconditioning, all specimens, except for the water-soaked injection-molded specimens, were oven dried and then placed in a humidity room at 65 percent relative humidity and 27°C for 4 weeks. The injection-molded specimens that were preconditioned by the water soaking procedure were not oven dried but placed directly into the 65 percent relative humidity room immediately after water soaking.

Soil bottles were inoculated with the brown-rot fungus *Gloeophyllum trabeum* (Madison 617) following the ASTM procedure (ASTM 1990b). At 4, 8, and 12 weeks of fungal exposure, specimens were removed from the bottles, and the fungal hyphae were carefully brushed from the samples. The specimens were then weighed, oven dried at 105 °C, and then reweighed. MC was determined from the wet and oven dried weights after exposure. The weight loss was determined from the oven dry weight after fungal exposure and the oven dry weight prior to fungal exposure but after preconditioning. Untreated solid wood (southern pine) was also tested in soil block tests as a check for fungal activity.

Flexural testing

Flexural tests were performed on specimens to determine the effect of fungal attack on mechanical properties. Four-point flexural tests were per-

formed on oven dried specimens according to ASTM D 790-84 (ASTM 1990a). Initial tangent modulus and maximum stress were determined before preconditioning and on specimens that were removed after 0, 4, 8, and 12 weeks of exposure and oven dried at 105°C. In all cases, failure occurred between the load points in the center third of the specimen.

Results and discussion

The method by which thermoplastic composites are processed can have a great influence on their performance. Different temperatures, pressures, and flows are found in different processing methods and produce composites with differing performance. For example, a thin, polymer-rich surface layer forms in injection-molded composites. The surface layer of extruded and compression-molded composites contains less polymer than injection molded ones. Additionally, it was necessary to cut our extruded samples from larger specimens, removing this surface layer. It would be expected that water would be less readily absorbed by the injection-molded composite and that this kind of composite would be more resistant to fungal attack compared with the extruded composite. Higher pressures are used in injection molding compared to those in compression molding or extrusion, and composite density is affected. In injection-molded composites, the wood fiber bundles are nearly completely collapsed, which results in a higher density composite compared

with extruded composites. Densities of 1.11, 1.04, and 1.02 g/cm³ were found for injection-molded, compression-molded, and extruded composites, respectively. These significant effects of processing methods and specimen preparation are often overlooked in laboratory fungal resistance testing of WPCs.

Preconditioning

A water soaking or cyclic boiling procedure was used to “precondition” the sample. In service, WPCs are typically used in exterior applications where periodic exposure to water is common. Boiling has been used to accelerate moisture sorption in WPCs (Raj and Kokta 1989, Naghipour 1996) as well as to condition samples prior to fungal resistance testing (Khavkine et al. 2001). We investigated both water soaking and cyclic boiling to determine their effect on fungal resistance of the composites.

During both the water soaking and cyclic boiling procedures, the extruded samples absorbed the most water and the injection-molded composites the least. After 2 weeks of water soaking, the injection-molded, compression-molded, and extruded composites had absorbed 4, 7, and 17 percent moisture, respectively, but had not reached equilibrium. The water-soaked samples absorbed about the same amount of water as did the boiled ones after five cycles (Fig. 1).

During the boiling/drying procedure, the maximum moisture absorbed during each cycle continued to increase even after five cycles. This increase was especially evident in the extruded composites, which had permanent thickness swell (Fig. 2).

These results suggest that composite damage accumulates with each cycle, allowing more moisture sorption in each successive cycle. At first, it may seem surprising that the extruded composite swelled the most despite having the lowest density. This is opposite of the trend for more conventional composites such as fiberboard, where higher density yields greater swelling. However, recall that higher density causes composites to absorb less moisture (Fig. 1) as a result of the polymer-rich surface layers and lower void contents. No permanent thickness swell was found for the water-soaked composites even though these composites and boiled composites absorbed similar amounts of moisture during preconditioning. However, the

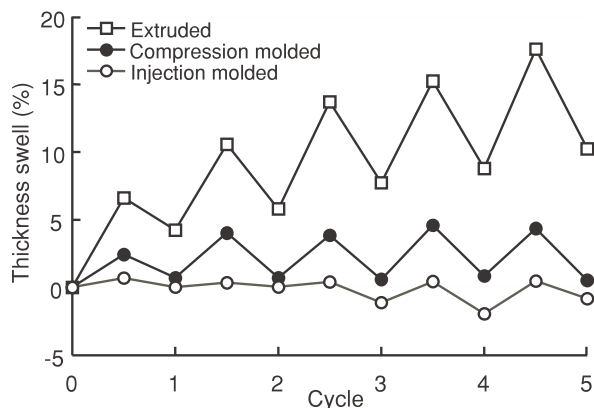


Figure 2. — Thickness swell of HDPE filled with 50 percent wood flour during five cycles of 2 hours boiling and 22 hours oven drying. Error bars removed for visual clarity.

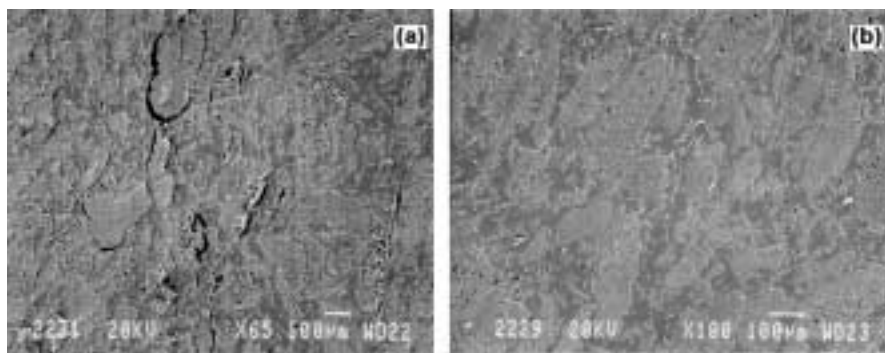


Figure 3. — Effect of five cycles of 2 hours boiling and 22 hours oven drying on composite microstructure: a) microtomed surface of composite after cyclic boiling; b) control sample.

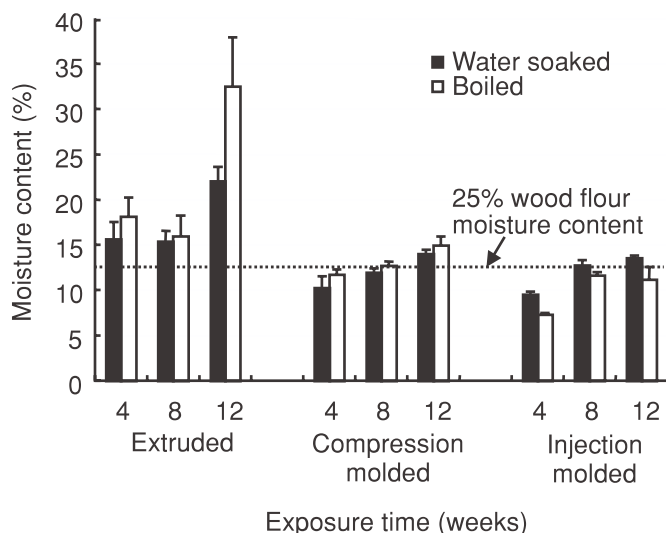


Figure 4. — Moisture sorption of HDPE filled with 50 percent wood flour during soil block test. Error bars are one standard deviation.

distribution of the absorbed moisture through the thickness of the samples may have differed.

Cutting soil-block specimens from larger boards and extruded profiles exposes the wood-rich interior of the composite and may reduce the moisture and fungal resistance provided by the resin-

rich outer layer. For example, Verhey et al. (2001a) found that removing the resin-rich layer in compression-molded composites resulted in greater fungal attack by white-rot fungi in composites containing 60 or 70 percent wood flour. This research also showed that fungal resistance of composites containing 50 percent wood flour or less was not affected by removal of the surface layer. In our tests, increased swelling was not found near the cut edges of the composites. However, it is difficult to determine the exact effect of cutting on fungal resistance since cutting was necessary to obtain adequate sample size. New tooling is being made to minimize the cutting necessary to obtain targeted dimensions, which may provide insight into these effects. Nevertheless, WPCs usually need to be cut to length during production and installation, and this represents a practical consideration.

Figure 3 shows the effect of cyclic boiling on the morphology of an extruded composite examined by scanning electron microscopy (SEM). Gaps are evident between the fiber and HDPE matrix, resulting from shrinking and swelling of the wood flour particles. This interfacial damage can inhibit fiber-matrix stress transfer, which lowers composite strength and provides fungal hyphae with access to the interior of the composite. Rangaraj and Smith (2000) found similar interfacial damage caused by moisture sorption. Klason et al. (1984) found matrix cracking in WPCs as a result of moisture sorption.

The interfacial damage was more obvious in boiled composites than in water-soaked ones and usually occurred near the surface. Because the boiling time was only 2 hours, the moisture may not have had time to penetrate far into the sample and the moisture of the wood flour at the surface may have been higher than that at the center. Although the water-soaked samples had a similar overall MC, this moisture may have been distributed more evenly than the moisture in the boiled samples and consequently caused less damage.

Soil block tests

The MC and weight loss data for the soil block tests are summarized in Table 1. Not surprisingly, the extruded composites once again absorbed more moisture than did the other composites (Fig. 4). The composites did not appear to reach equilibrium, even after 12 weeks

Table 1. — Results of soil block tests on wood–plastic composites exposed to *G. trabeum*.^a

Processing method	Fungal exposure time (wk.)	Pre-conditioning	Final MC	Weight loss
			----- (%) -----	
Extrusion	4	Water soaking	15.6 (1.9)	1.5 (0.2)
		Boiling	18.1 (2.1)	2.5 (0.5)
	8	Water soaking	15.4 (1.2)	4.4 (0.4)
		Boiling	16.0 (2.5)	9.6 (1.9)
	12	Water soaking	22.0 (1.6)	6.0 (1.0)
		Boiling	32.0 (5.5)	22.8 (10.2)
Compression molding	4	Water soaking	10.2 (1.4)	0.3 (0.3)
		Boiling	11.7 (0.6)	0.6 (0.7)
	8	Water soaking	12.0 (0.5)	1.2 (0.5)
		Boiling	12.7 (0.5)	1.4 (0.6)
	12	Water soaking	14.0 (0.5)	1.2 (0.2)
		Boiling	15.0 (1.0)	2.4 (0.9)
Injection molding	4	Water soaking	9.5 (0.3)	0 (0.2)
		Boiling	7.3 (0.2)	-0.4 (0.1)
	8	Water soaking	12.7 (0.6)	0.9 (0.4)
		Boiling	11.6 (0.4)	0.3 (0.2)
	12	Water soaking	13.5 (0.3)	3.2 (1.0)
		Boiling	11.1 (1.5)	0.4 (0.6)

^aValues in parentheses are one standard deviation.

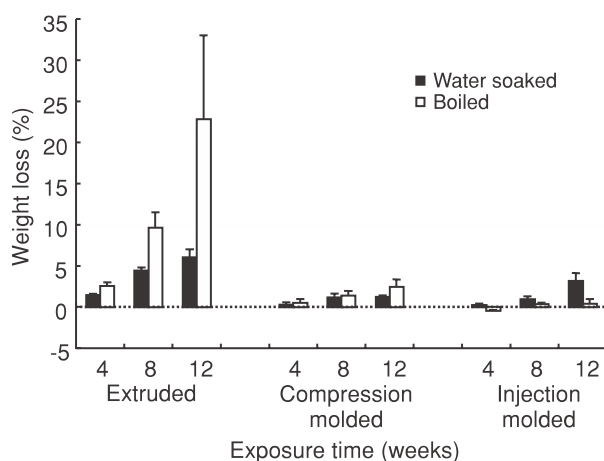


Figure 5. — Weight loss of HDPE filled with 50 percent wood flour during soil block test. Error bars are one standard deviation.

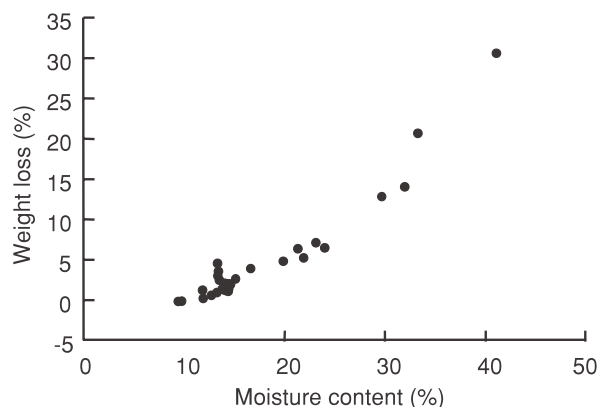


Figure 6. — Correlation between weight loss (dry basis) and MC in soil block test of HDPE containing 50 percent wood flour.

in wet soil. The extruded and compression-molded composites that were boiled generally absorbed more moisture than did the water-soaked ones. However, the opposite trend was found for the injection-molded composites. This undoubtedly resulted from the fact that unlike the other composites, the injection-molded composites were not dried after preconditioning.

For solid wood, it is necessary to keep the MC below the fiber saturation point (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 4**. Assuming that there is no moisture gradient through the thickness of the samples, keeping the MC below about 12.5 percent of the total composite weight (or about 25% wood flour MC) should prevent fungal attack. Only some injection-molded and compression-molded composites exceeded this threshold, and then only after considerable time in the soil bottles. These composites represent a borderline case for fungal attack. Our results reinforce the difficulty in assessing even thin WPCs by the soil block test because of their slow moisture sorption. Solid wood samples quickly approach equilibrium in a soil bottle, but WPCs do not. Thus, it is evident that a 3-month soil bottle test is insufficient to fully evaluate WPC durability. To use the 3-month soil block test to fully evaluate the fungal durability of WPCs, samples should be preconditioned to an MC near the fiber saturation point, the minimum required MC for fungal attack.

Figure 5 shows weight loss found in soil block tests. A large weight loss was found for specimens cut from extruded composites, especially boiled composites. At most, 3 percent weight loss was found for injection-molded and compression-molded composites; composites with more than 25 percent wood flour MC sustained the greatest weight loss. Weight loss as a function of MC for all the samples is plotted in **Figure 6**. A good correlation appears likely but more samples need to be tested, especially at higher MC levels, before the relationship can be confidently quantified.

Figure 7 shows optical micrographs of the surfaces of extruded composites after the soil block test, with and without fungus. The fungus aggressively at-

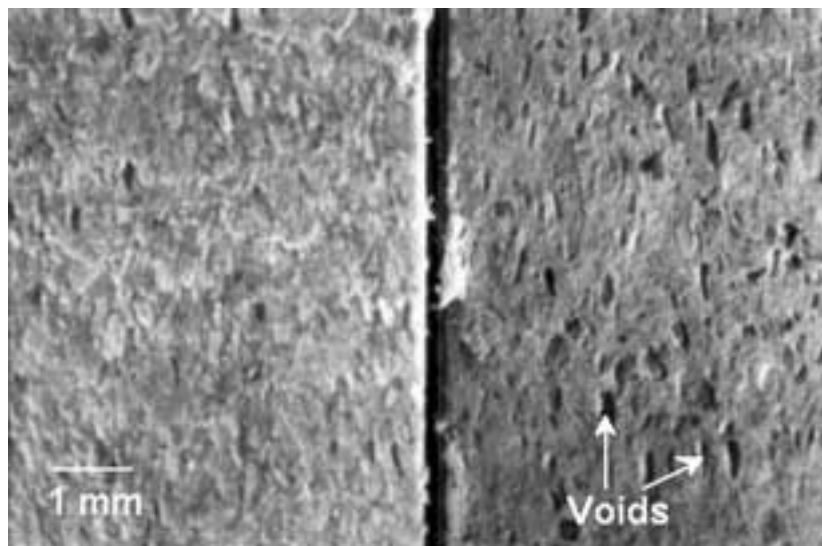


Figure 7. — Optical micrograph of surface of extruded HDPE-wood flour composite from soil bottle tests, without fungus (left) and with fungus (right). A cyclic boiling preconditioning procedure was used.

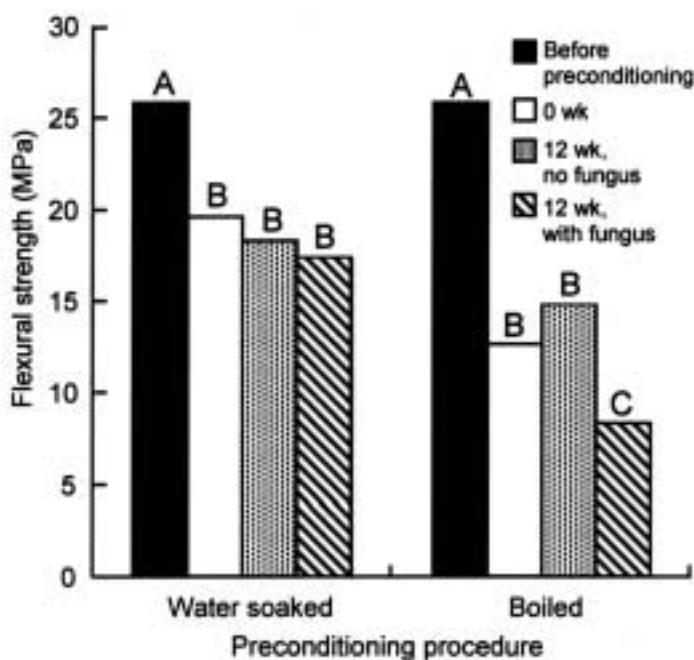


Figure 8. — Flexural tests on dry extruded composites. Bars with the same letter are statistically equivalent at 95 percent confidence.

tacked the wood flour particles at the surface during the soil block test. Partially degraded wood flour particles are evident, as well as holes where wood particles were fully degraded. The wood flour particles of the specimen tested without fungus remained intact (Fig. 7, left), even though the moisture history of both specimens shown in Figure 7 was similar.

Flexural testing

Researchers have used loss in mechanical properties of wood such as strength and impact as a sensitive measure of incipient fungal attack (Wilcox 1978, Curling et al. 2002). Since wood flour is used as a filler in many WPCs, it doesn't contribute greatly to mechanical performance measures such as strength.

Therefore, the sensitivity of the strength of these composites to fungal attack is probably not as great as that of solid wood. Nevertheless, loss in mechanical performance of composites filled with wood flour could help corroborate weight loss results.

The negative effects of moisture sorption on flexural performance complicate the use of mechanical performance to assess fungal resistance of WPCs. During soil bottle tests, the composite is not only attacked by fungus but may also incur damage due to moisture sorption. Therefore, a second set of soil bottles was prepared but not inoculated with fungus to separate the effects of moisture and fungal attack on flexural performance.

Figure 8 summarizes the flexural strength results for the extruded composites. Both water soaking and cyclic boiling resulted in large losses in strength (compare solid black and white bars in Fig. 8). Boiling resulted in greater strength losses than did water soaking, consistent with the greater damage found by SEM. Moisture sorption during soil bottle testing resulted in no further significant reductions in strength for either boiled or water-soaked composites (compare white and stippled bars in Fig. 8). However, strength losses caused by fungal attack were found for boiled composites but not water-soaked ones (compare stippled and striped bars in Fig. 8). This differs from the weight loss results (Fig. 5), where both water-soaked and boiled composites sustained weight loss. Similar findings were found for the other composites. For example, no reductions in flexural strength due to fungal attack were found for compression-molded composites despite statistically significant (albeit small) weight losses.

Weight loss may be a more sensitive measure of fungal attack than is flexural strength, since the fungus attacks the wood flour, which has already debonded from the matrix and consequently is no longer efficiently bearing the applied stress. However, flexural strength may prove more sensitive to fungal attack if wood flour (as filler) is replaced with wood fiber, which can act as a reinforcing element if well bonded to the plastic matrix. This distinction between the relative durability of wood-flour-filled and wood-fiber-reinforced composites may become an important

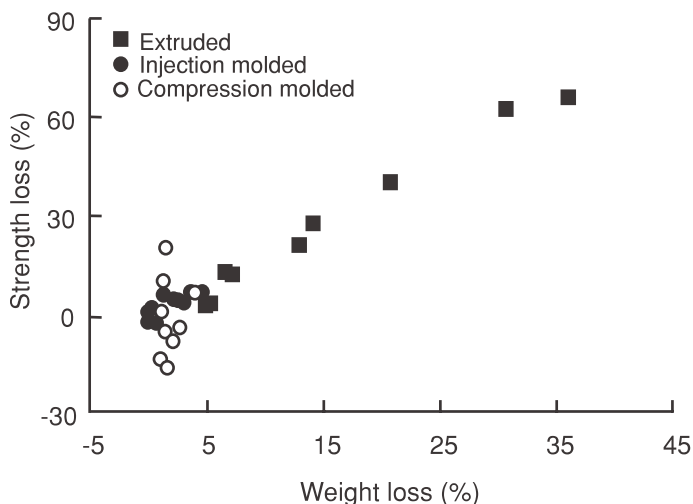


Figure 9. — Relationship between weight loss and flexural strength loss for HDPE composites containing 50 percent wood flour.

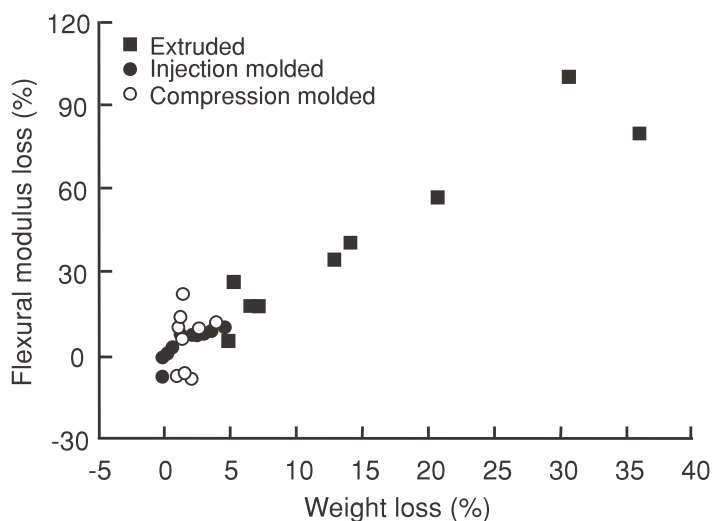


Figure 10. — Relationship between weight loss and flexural modulus loss for HDPE composites containing 50 percent wood flour.

issue if more wood flour is replaced by wood fiber.

Despite careful preparation, large bottle-to-bottle variability caused by differences in fungal activity resulted in large coefficients of variation, especially at high MC levels. Although this is typical of soil block tests, it makes comparisons between average weight and strength losses difficult since similar specimens may undergo different levels of fungal attack in different bottles. However, for each individual specimen, weight and strength losses should correlate well since both are a measure of fungal attack on the same specimen. Strength loss re-

sulting from fungal attack was determined for each specimen exposed to fungi by comparing its flexural strength with the average strength of similar samples in soil bottles that were not inoculated. The weight and strength losses for all specimens exposed to fungi for 12 weeks are plotted in **Figure 9**. Strength- and weight-loss data for individual specimens correlate reasonably well. **Figure 10** shows similar findings for the tangent modulus of elasticity data.

Concluding remarks

A 2-week water-soaking or cyclic-boiling procedure was used to infuse

moisture into WPCs. Extruded composites absorbed the most moisture and injection-molded composites the least. Although similar amounts of moisture were absorbed during water soaking, the cyclic-boiling procedure resulted in more damage to the composites, especially extruded ones. In neither procedure did the composites reach equilibrium. Significant weight losses were found with a modified soil bottle test once the composite MC reached roughly 12 to 15 percent. This corresponds to an average wood flour MC near the fiber saturation point. The greatest weight losses were found for extruded composites that had been preconditioned by boiling. Damage to the composite from moisture sorption complicated the use of flexural performance as a measure of fungal attack. Mechanical performance appeared to be a less sensitive measure of fungal attack compared to weight loss. However, flexural modulus loss and strength loss due to fungal attack correlated well with weight loss for individual specimens when plotted together. Flexural strength may prove more sensitive to fungal attack if wood flour is replaced with wood fiber, which can act as a reinforcement if well bonded to the plastic matrix.

These results suggest that WPCs may show some susceptibility to fungal attack if MC in the wood component nears fiber saturation and no fungicide is present. Even for a single formulation, the moisture sorption in laboratory tests is still a function of specimen preparation method, specimen conditioning, and fungal exposure conditions. Wood-plastic composites perform differently from wood and more traditional wood composites, and these procedures must be carefully considered when using laboratory testing to evaluate fungal resistance.

Although laboratory fungal resistance testing is useful in determining the potential degradability of a composite, field exposures are necessary. This investigation is part of a larger project on the durability of WPCs in exterior applications. Field tests on extruded composites, both in ground contact and above the ground, are in progress in Mississippi and Wisconsin. Although these tests take considerable time, they represent an exposure more typical of in-service conditions, with attack from a variety of fungi and stresses from other environmental exposures (e.g., ultraviolet

let radiation, freeze-thaw cycles). These combined exposures may provide a harsher environment as a result of synergism and more rapid degradation than that caused by a single type of exposure.

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