DURABILITY OF AN EXTRUDED HDPE/WOOD COMPOSITE

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ABSTRACT

As apart ofwood-plastic composite (WPC) material development, this research evaluated the resistance of WPC formulations to fungal decay and biocide leaching. In laboratory tests using small WPC samples exposed directly to brown-rot and white-rot fungi, there were no weight losses caused by decay in most formulations. Formulations with relatively high wood content and not protected by zinc borate exhibited moderate weight losses. Scanning electron microscopy of WPC specimens supporting fungal growth show mycelium concentrated in the interfacial gaps between the wood and thermoplastic component near the specimen surface. The relative influence of each material component on the WPC resistance to decay was analyzed by simplex analysis. Of the various component term effects, the most influential was that relating to composite wood content. Increases in talc concentration also resulted in greater weight losses. An increase in high-density polyethylene (HDPE) in the formulation reduced susceptibility to weight loss during the accelerated decay test. Zinc borate at 2 percent concentration, the lowest level tested, prevented any weight loss. Our conclusion is that either the inclusion of zinc borate or limiting the wood content of WPC can effectively prevent fungal decay. The very low leach rate as determined by laboratory testing appears to be controlled mainly by dissolution of the zinc borate and not by diffusion. The tests show that zinc borate will take at least 20 years to completely dissolve and leach from the material. Results indicate that careful material design can prevent fungal decay of WPC even under severe exposures.

It is widely recognized that unprotected wood is subject to attack by decay fungi in moist, terrestrial environments, and by marine borers in seawater (9). Preservative-treated wood used in shore facility applications such as timber piling can also experience rapid deterioration due to fungal decay and marine borer damage (13). This is not the case with synthetic polymeric materials, which, compared to wood, are largely impervious to marine borer attack (3) and fungal decay. Where fungal attack is present, additives (e.g., plasticizers, cellulosics, lubricants, stabilizers, and colorants) are generally responsible (2).

Laboratory protocol for evaluating the decay resistance of wood products is commonly used but remains somewhat controversial as a predictor of field performance because it is more severe than most aboveground field exposure tests (6). In addition, some researchers have found that laboratory-based procedures such as the soil block test are unsuitable for use as a predictor of composite panel performance (1,5). The concerns primarily address the unknown impact of differences between solid wood and composites with regard to decay susceptibility, moisture gradients, and the increased surface area to volume ratio of composite panel products. Others have suggested that solid wood test procedures can be used with minor modifications (10,15).

Wood fillers have been used in plastics for some time (7). More recently, high wood content (50%) of such materials has led to the development of wood-plas-

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tic composites (WPC). The commercial success of these hybrid composites has addressed the need for durable, exterior wood composites. However, scientific investigations of material design have largely focused on improving mechanical properties (8,16). Despite the claims of rot resistance, few scientific studies address the biodegradation of WPC. Morris and Cooper (12) present only anecdotal evidence suggesting that a commercial wood-plastic composite is susceptible to fungal decay.

Mankowski and Morrell (11) reported on the soil-block testing of three commercial samples with nominal material composition cited. In this study, little or no degradation was found in two of the samples. A third sample that was reportedly composed of 70 percent wood, did experience about a 20 percent weight loss caused by brown-rot fungi. After microscopic evaluation, the primary mode of degradation appeared to be fungal penetration of material voids. Neither of these studies evaluate materials with known components nor address the influence of material formulation or design on susceptibility to fungal attack.

Any WPC that does not employ a wood preservative must rely on barrier properties of the plastic matrix for protection of the wood. Presumably, the thermoplastic matrix in a WPC could act as a barrier layer to exclude moisture and fungal attack. Scheffer and Morrell (14) illustrated this concept by placing untreated wood stakes in polyethylene boots. Such a physical mechanism to avoid biodegradation would lead to systematic engineering through material design. In this case, susceptibility to fungal attack may be a controllable material property like strength and stiffness. Specifically, this research intended to:

1. Evaluate the resistance of WPC formulations to fungal decay and biocide leaching using laboratory procedures;

2. Discern the relative contributions of various components of WPC formulations toward propensity to fungal decay;

3. Elucidate possible mechanisms for fungal attack for use in material design.

EXPERIMENTAL

MATERIALS

The WPC materials were produced from multi-component formulations comprised of wood, thermoplastic, inorganics, and process additives. Maple 40 mesh wood flour (American Wood Fibers 4010) was obtained commercially for use in this study. A reactor grade high-density polyethylene (HDPE) powder (Equistar Chemical LBO 100 00) was used for the thermoplastic. Inorganic components were used to modify the mechanical and fire performance of the material. These materials included talc (Suzorite 2661, Chemical Distributors Inc.) and zinc borate (Firebrake ZB, US. Borax). The levels of these components were varied in the formulations as described in the statistical analysis methods. Process additives were maintained at 6 percent in all formulations and included ethylene bis-stearamide wax (General Electric Specialty Chemicals), zinc stearate (Chemical Distributors Inc. DLG20), phenolic resin (Plenco 12631), and methyl di-isocyanate resin (Bayer Corp. Mondur 541).

TABLE 1. — WPC formulations.

Formulation no.	Wood	HDPE	Zinc borate	Talc	Processing additives
			(%)		
1	45.00	39.00	10.00	0.00	6.00
2	45.00	39.00	10.00	0.00	6.00
3	49.00	45.00	0.00	0.00	6.00
4	49.00	45.00	0.00	0.00	6.00
5	53.00	33.00	8.00	0.00	6.00
6	51.00	36.00	7.00	0.00	6.00
7	70.00	24.00	0.00	0.00	6.00
8	58.00	32.00	4.00	0.00	6.00
9	58.00	32.00	4.00	0.00	6.00
10	49.00	41.00	4.00	0.00	6.00
11	47.00	45.00	2.00	0.00	6.00
12	64.00	28.00	2.00	0.00	6.00
13	53.00	33.00	0.00	8.00	6.00
14	49.00	41.00	0.00	4.00	6.00
15	65.00	29.00	0.00	0.00	6.00

TABLE 2. — Decay fungi.

Reference no.	Fungi species	Rot induced
M617	*Gloeophyllum trabeum*	Brown
M697	*Trametes versicolor*	White
M698	*Poria placenta*	Brown

COMPOUNDING AND PROCESSING

A 20-kg batch of each WPC formulation (Table 1) was mixed in a drum blender for 10 minutes. The mixture was then conveyed to the feed hopper of a counter-rotating conical twin-screw extruder (Cincinnati Milacron 55 mm). A slit die, measuring 15.24 cm by 1.27 cm, was attached to the extruder. During extrusion, a barrel/screw temperature of 163°C, die temperature of 171°C, and a screw rotational rate of 10 rpm. The extrudate was cooled with 20°C water after exiting the die.

ACCELERATED DECAY TESTS

Susceptibility to biodegradation was assessed using ASTM D 2017. Standard procedures were followed with two exceptions: 1) WPCs instead of solid wood specimens were evaluated; and 2) smaller specimens were used. Six WPC specimen cubes each measuring approximately 10 mm per side, from each WPC formulation, were conditioned to a constant weight at a standard environment of 27°C and 30 percent relative humidity then steam sterilized without pressure for 30 minutes. After cooling, the test blocks were placed in culture bottles containing one of three actively growing brown- or white-rot fungi (Table 2). After 12 weeks, the specimens were brushed free of mycelium and dried overnight in a forced draft oven at 60°C. Blocks were then reconditioned to a constant weight at the standard environment before the percentage of weight loss due to fungal decay was determined. The average

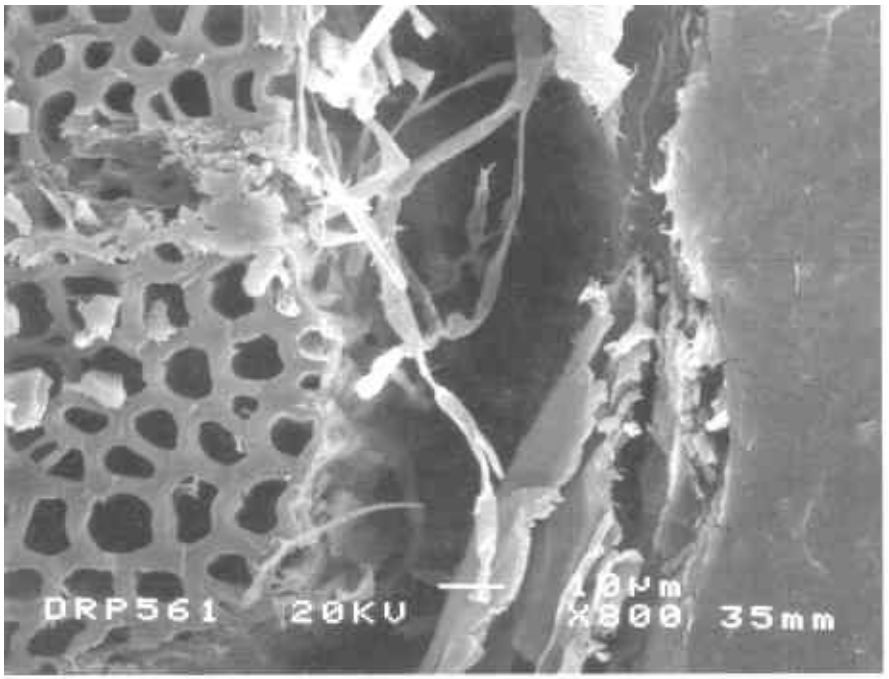

Figure 1.— A scanning electron micrograph of a WPC formulation supporting fungal decay. The mycelium is concentrated in an interfacial gap between the wood and thermoplastic.

weight loss per treatment group was calculated. Because the wood component of these composites is less than that of solid wood, the amount of potential weight loss is proportionally less.

EXPERIMENTAL DESIGN AND STATISTICAL ANALYSIS

Empirically analyzing the relative contribution of multiple components within a material formulation is complicated by the fact that changing one component inherently influences the relative proportion of the others (4). Mathematically, this unique case is denoted when the dependent variables always sum to equal one. Simplex analysis, a response surface methodology, is constructed to uniformly distribute formulations around a multivariate design space. The selected formulations for the experimental design are presented in **Table1.**

To analyze the influence of each material component on the WPC resistance to decay, the corresponding sample weight losses, h_I, produced from the specified formulations composed of fractional component amounts (x_i) were fit to a Scheffé equation:

$$\eta_I = \sum_{i=1}^{q} \beta_i x_i + \sum_{i<j}^{q} \beta_{ij} x_i x_j \qquad [1]$$

Only the terms deemed statistically significant ($p > 0.05$) were maintained in the final polynomial. For many problems, a variable's influence can be interpreted by the polynomial coefficient (b_i).

ACCELERATED LEACH TESTS

Accelerated leaching was conducted over 9 months on three formulations in accordance with ASTM C 1308 modified as described herein. Six specimens taken from each of three WPC formulations listed in **Table 1** (formulations 1,3, and 11) were placed in individual 1-L fluorinated polyethylene (PE) bottles with screw cap lids. Fluorinated PE is inert to most chemicals, preventing reaction with the leachant or leachate (zinc borate). The specimens were about 13 mm thick and cut to 48 by 52 mm (dimensions of diffusing faces). The cut edges were sealed using a chemically resistant epoxy coating to minimize diffusion from the cut surfaces. Each bottle was filled with deionized water as the leachant. During exposure, the containers with specimens were allowed to rest undisturbed in an environment of 23°C (ambient testing) or 50°C (elevated temperature testing in an oven) for the specified periods of time. Each sample rested on one epoxy-coated edge with only the corners of a diffusion face resting along the wall of the container, thus ensuring the exposure of both faces to the water.

The sampling intervals for drawing aliquots of leachate increased as the test progressed and it became apparent that leach rates were changing very slowly. The leachant in each container was changed after each sampling for the elevated temperature samples, and every other sampling for the ambient temperature samples.

The leachate was sampled using a digital pipette rinsed with deionized water between aliquots. Each aliquot of leachate (10 mL for the ambient samples and 11 mL for the accelerated samples) was drawn and placed in a PE sample tube. The aliquot samples were then acidified with 1.5 mL of three molar nitric acid for preservation and analysis. Zinc and boron concentrations were determined using an inductively coupled plasma optical emission spectrometer (ICP/OES). Results were corrected for the acid dilutions and aliquot volumes just described and converted from mg/L concentration values to mg/hr. leach rates. The data were plotted as leach rate versus exposure time, and as cumulative fraction leached (CFL) and incremental fraction leached per day (IFL) versus exposure time using the software companion to the ASTM C 1308 method.

RESULTS AND DISCUSSION

DECAY MECHANISM

In the WPC formulations that exhibited decay, the activity was concentrated on the exterior of the specimen. With progression, the decay was manifested in a form of surface erosion. The limited wood particles exposed on the surface would degrade when exposed to the fungi. If pathways existed into the specimen, mycelium would propagate further. This decay mechanism is similar to that observed for microbial degradation of polyethylene-starch composites (17).

Microscopic examination of WPC specimens supporting fungal growth show mycelium concentrated in the interfacial gaps between the wood and thermoplastic component near the specimen surface (Fig. 1). Differences in molecular polarity between wood and polyolefin thermoplastics do not allow the development of good bonding at their interface. Without chemical or physical bonding, interfacial gaps can develop from a variety of external forces. These gaps appear to provide a pathway

for fungi in some formulations. In formulations with a well-dispersed wood component, the thermoplastic appears to act as a barrier and prevents the development of a continuous pathway for penetration into the mterial.

ACCELERATED DECAY TEST

Only 3 of the 15 formulations tested indicated any clear evidence of susceptibility to weight loss **(Table 3).** Of those formulations, formulation 7 showed the greatest susceptibility to weight loss. The relatively low weight loss results are close to the detection limits of this procedure but it is revealing that formulation 7, containing the least amount of plastic, high wood content, and no zinc borate, was also the formulation with the greatest average weight loss. Formulations 13 and 15 also contained relatively low amounts of plastic, high wood content, and no zinc borate and were intermediate in terms of weight loss. The remaining 12 formulations tested containing either zinc borate or higher plastic levels showed no significant weight loss.

None of the formulations in control bottles where no fungi were present incurred weight loss. This confirms sterile conditions for both the WPC samples and test containers. It is interesting to note, however, that when the WPC were placed in a fungi-free environment, all formulations gained weight. Wood moisture absorption or hydration of the inorganic components may have caused this weight gain.

The coefficient of variation (COV) values for weight losses of the various formulation are higher than those commonly found with WPC mechanical properties. The high variability may result from approaching the test detection limits of the inherently variable biological testing. The relatively high variability, however, did not seriously hamper component analysis.

Scheffé polynomial equations incorporating the four varible composite components, namely wood, HDPE, magnesium silicate, and zinc borate, were the most statistically appropriate (95% confidence levels) for describing the average weight loss for various formulations. The adjusted r^2 values of the Scheffé equations describing weight loss after exposure to fungal strains M617, M697, and M698 were 58, 71 and 60 percent, respectively. Using the equation coefficients, component term effects and ranks were calculated **(Table 4).**

TABLE 3—Average WPC weight loss resulting from accelerated decay tests.

Formulation no.	Average weight loss			
	M617	M697	M698	Sterile controls
	(%)			
1	–3.00 (0.41)[a]	–2.79 (0.47)	–2.53 (0.59)	–3.34 (0.27)
2	–2.44 (028)	–2.67 (0.23	–2.86 (0.23)	–2.48 (0.31)
3	0.23 (0.68)	–1.23 (0.46)	0.58 (0.38)	–2.48 (1.09)
4	–0.24 (0.58)	–0.95 (0.43)	0.32 (0.39)	–2.79 (0.18)
5	–0.87 (0.37)	–0.93 (0.41)	–0.89 (0.26)	–0.99 (0.42)
6	–0.07 (1.62)	–1.10 (0.37)	–0.96 (0.43)	–1.40 (0.45)
7	**4.90 (0.84)**	**4.24 (1.48)**	**8.47 (0.80)**	**–1.56 (0.25)**
8	–0.44 (0.39)	–0.80 (0.18)	–0.66 (0.24)	–1.24 (0.18)
9	–0.36 (0.18)	–0.30 (0.37)	–0.58 (0.21)	–1.16 (0.23)
10	–1.80 (0.02)	–1.66 (0.24)	–1.50 (0.23)	–2.17 (0.45)
11	–1.34 (0.18)	–0.97 (0.29)	–0.78 (0.22)	–1.11 (1.77)
12	–1.20 (0.40)	0.10 (1.54)	–0.94 (0.34)	–1.23 (0.32)
13	**0.85 (1.42)**	**2.57 (3.50)**	**4.30 (1.97)**	**–1.59 (0.29)**
14	–0.22 (0.82)	–1.53 (0.57)	0.01 (0.41)	–2.63 (0.35)
15	**1.36 (0.80)**	**0.29 (0.93)**	**3.28 (0.39)**	**–1.64 (0.25)**

TABLE 4. — WPC component term effects.

WPC component	M617	M697	M698	No fungal activity
Wood	4.42 (1.00)[a]	4.68 (1.00)	6.72 (1.00)	0.88 (0.66)
HDPE	–1.77 (040)	–2.67 (0.57)	–2.87 (0.43)	–0.23 (0.17)
Zinc borate	–2.47 (0.56)	–1.67 (0.36)	–3.57 (0.53)	–0.64 0.48)
Magnesium silicate	0.30 (0.07)	1.85 (0.40)	2.28 (0.234)	0.14 0.10)
Wood × zinc borate	--	--	--	1.33(1.00)

[a] Values in parentheses represent standardized component term effects.

Of the various component term effects, the most influential was that relating to composite wood content. As the wood content increased, the level of weight lose increased. This finding would seem reasonable, as the WPC wood component is the sole target for fungal degradation. Regardless of the active fungi, the influence of wood was twice that of any other component. It must be noted that the wood component term effect was slightly positive and the zinc borate component term effect was slightly negative when no fungi were active **(Table 4).** Because weight losses during the test can occur from changes in several components, this may suggest there were other factors measured in the test. For example, the influence of zinc borate on moisture retention in terms of specimen weight change was not explicitly measured.

Talc also showed an ability to increase a fomulation's susceptibility to weight loss **(Table 4).** Although fungi cannot directly attack this inorganic substance, its role in decreasing decay resistance may be accomplished though its influence on composite structure. As with wood, unmodified interfaces between talc and HDPE are poor. In addition, talc amendments to HDPE tend to increase brittleness. Both of these attributes would tend to assist the formation of micro-cracks that could provide pathways for fungal penetration and decay.

Both HDPE and zinc borate minimized a formulation's susceptibility to weight loss during the accelerated decay test. The level of influence associated with each was relatively similar, with zinc borate having a stronger influence in decreasing weight losses in two of the three fungi tested **(Table 4).** The relative effectiveness of HDPE to zinc borates in resisting decay may be somewhat surprising given the proven effectiveness of zinc borate as a biocide. Interpretation of these results would suggest that resistant wood-plastic formulations could be produced by either elevating HDPE content or adding zinc borate.

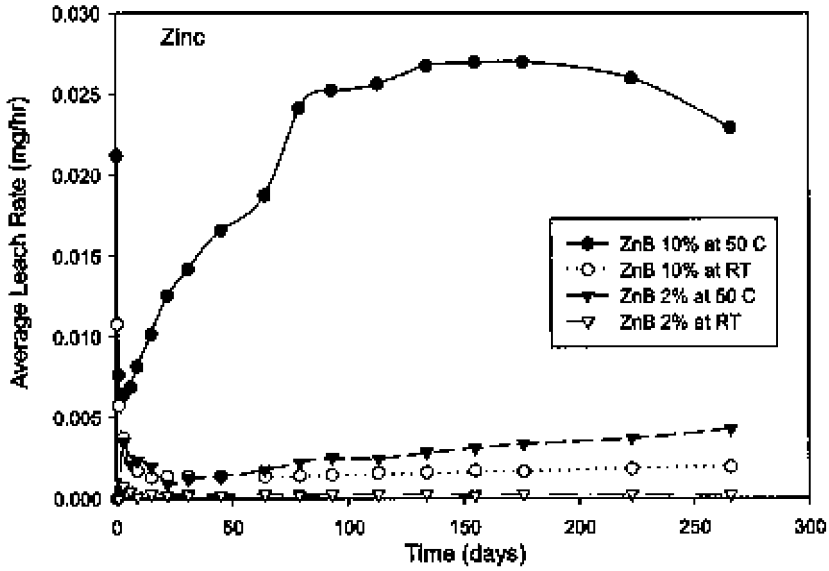

Figure 2. — Average leach rates of zinc from samples immersed in water over a 9-month period.

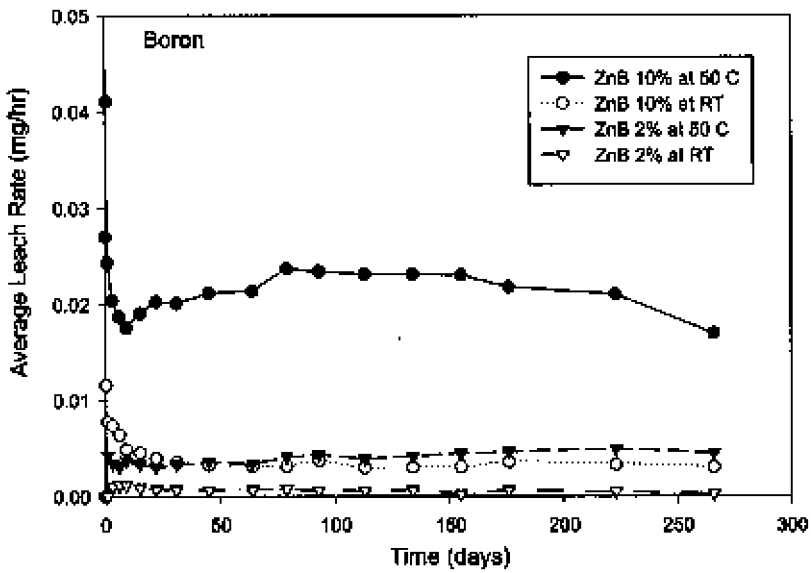

Figure 3. — Average leach rates of boron from samples Immersed In water over a 9-month period.

TABLE 5. — Zinc and boron maximum leach rates from WPC samples.

Treatment	Max. leach rate		Years to depletion at max. leach rate	
	Zn	B	Zn	B
	(mg/hr.)		(yr.)	
2% zinc borate at RT[a]	0.0002	0.0005	96.2	23.8
10% zinc borate at RT	0.0018	0.0033	66.7	18.2
2% zinc borate at 50°C	0.0037	0.0050	6.2	2.3
10% zinc borate at 50°C	0.0226	0.0210	4.6	2.8

[a] RT = room temperature.

The long- term effectiveness of either option, however, will require further study.

ACCELERATED LEACH TEST

ASTM C 1308 provides an accelerated means for assessing the leach rate of zinc borate from treated wood composites. In addition, an analysis method is provided to determine if diffusion is the primary leaching mechanism for the material. If diffusion is the dominant mechanism, test results can be modeled to estimate long-term releases of zinc borate from non-accelerated exposures, and aid in determining the fungicide's effective life in the wood composite. The ASTM computer program can also be used to indicate a solubility-controlled leaching mechanism.

Figures 2 and **3** provide graphical representation of the averaged leach rates for each interval. Interpretation of the ICP/OES analysis reveals that the average zinc leach rate in the 10 percent zinc borate/50°C leachate leveled off and decreased slightly after about 175 days of exposure, after increasing up to that time. The 2 percent/50°C leach rate appeared to be increasing slightly to the end of the 9 months, but is significantly lower than the rate for the 10 percent samples. Zinc leach rates for the room-temperature exposures remained relatively steady throughout the test period.

Boron leach rates appear to be relatively level for the room temperature samples at both concentrations. The 50°C samples have a slightly decreasing rate towards the end of the study. Similar to zinc, the boron rate for the 2 percent samples at 50°C is significantly lower than that of the 10 percent samples.

The total amount of zinc and boron leached from the test samples is extremely low in relation to the total amounts compounded in the samples. It may take a number of years for even the high-temperature samples to lose most of their zinc-borate content **(Table 5).** Eventually, the leach rates are expected to decrease significantly as the zinc borate nears depletion. This should occur in the high-temperature samples much sooner than in the room-temperature samples.

The companion software program from ASTM C 1308 was used to model the CFL of both zinc and boron in selected 10 percent zinc borate samples at room temperature and 50°C. **Figure 4**

and 5 show linear model fits and experimental data. For a log model fit, a value of E_r (relative error of fit) equal to or less than 0.5 percent indicates that diffusion accurately represents the leaching mechanism. All of the E_r values for these selected samples are well above the 0.5 percent limit, indicating that diffusion is not The driving force of the observed leaching behavior.

Zinc borate is known to have very low solubility in water, thus dissolution is a likely candidate as the rate controlling mechanism. The plot of the CFL versus time data will have a linear shape if the dissolution rate of a species is small (low solubility) compared to the diffusion rate. **Figure 6** shows plots of the incremental fractions leached per day (IFL/ day) for 10 percent zinc borate at both room temperature and 50°C. A COV of 10 percent or lower indicates that the data fit the linear model and that solubility limits the release of species of interest. The first three data points of each sample are excluded from the statistical calculation of the COV because the leaching behavior had not stabilized during that early sampling period. Additionally, the COV of leached zinc from the 50°C sample was calculated using only the last eight data points. All except one of the COVs in **Figure 6** are below 10 percent, which indicates that solubility is likely the key limiting factor. But that conclusion is not certain since the data appear to follow a somewhat nonlinear pattern. Several other mechanisms may be taking place within and on the surfaces of the composite samples and playing at least a minor role in leach rates and thus affect CFL values. These include physical adsorption of ions onto the sample surfaces, as well as species being present in two or more chemical forms and having different solubility. In addition, because the material is heterogeneous, position of the particles will affect both dissolution and diffusion rates. Those zinc borate particles next to the wood and not imbedded in plastic will experience more rapid dissolution and diffusion of ions. Conversely, those particles imbedded in the plastic will experience relatively slower dissolution and diffusion of ions,

Since the diffusion model does not fit the deta, applying mathematical expressions for diffusion phenomena will not adequately predict long-term leaching behavior of the composite materials in the field. After a period of stabilization, the leaching behavior appears to be driven mainly by solubility rather than diffusion. This linear behavior can be used to estimate depletion times for both accelerated and non-accelerated exposures if leach rates are assumed to remain relatively constant. **Table 5** presents projected depletion time for the zinc and boron, assuming constant leach rates (taken as the leach rates at 223 days), and solubility as the driving force.

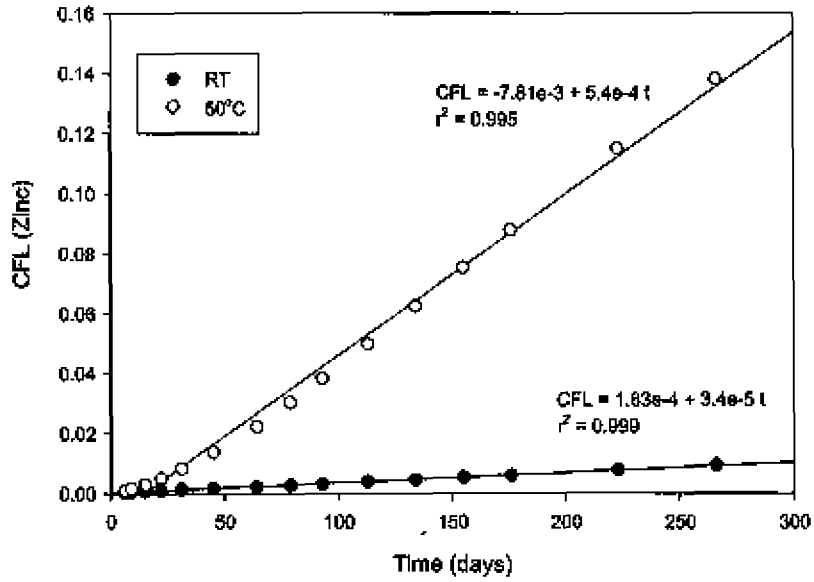

Figure 4. — Cumulative fraction (CFL) of zinc leached from samples at room temperature and 50°C.

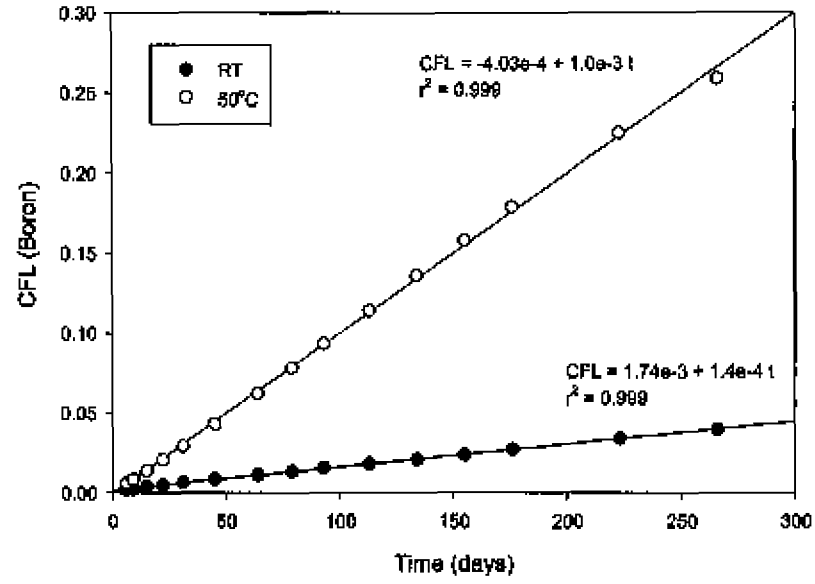

Figure 5. — Cumulative fraction (CFL) of boron leached from samples at room temperature and 50°C.

Conclusion

A modified ASTM D 2017 accelerated decay test can be used to indicate extruded WPC formulations that may be susceptible or resistant to fungal decay. Results of this research show a clear difference in weight loss of formulations with relatively high wood content and

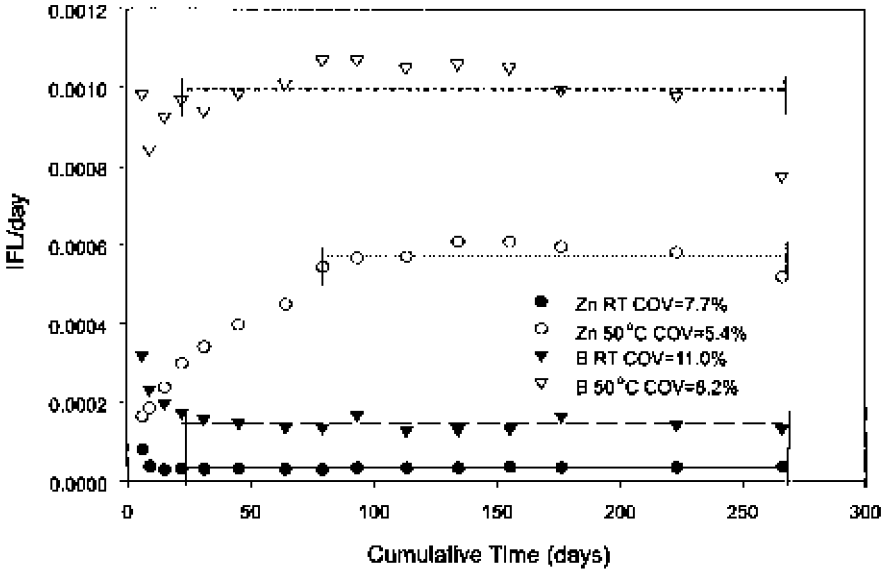

Figure 6. — Incremental fraction (IFL) of zinc and boron leached per day at room temperature and 50°C. Data range used for calculating sample statistics is indicated by vertical lines.

no preservative compared to formulations incorporating increased HDPE content or protected by zinc borate. Despite the fact that all WPC formulations evaluated in this research display relatively low weight losses compared to unprotected wood, any weight loss was considered to indicate some level of decay susceptibility. A simplex analysis of mixtures indicates that both wood and talc content increases the weight loss of formulations evaluated with the accelerated decay tests, whereas both HDPE and zinc borate appear similarly effective at decreasing weight loss. Although all the formulations evaluated with sterile soil media experienced small weight gains, significant component term effects for formulation components may indicate that factors other than fungal decay contribute to weight changes in WPC formulations. Accelerated leach tests were used to clearly indicate that zinc borate, an effective fungicide, will last for up to 20 years or longer in WPC formulations. The very low leach rate appears to be controlled mainly by dissolution of the zinc borate and not by diffusion.

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