Mold Susceptibility of Rigid PVC/Wood-Flour Composites

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Rigid PVC/wood-flour composite lumber containing either hardwood (maple) or a softwood (southern pine) wood flour at different levels of wood-flour content was evaluated for susceptibility to fungal colonization and discoloration by using standard tests that mimicked exterior (ASTM G21) and interior (ASTM D3273) environments, respectively. In the exterior test protocol, although both types of PVC/wood-flour composite lumber exhibited fungal colonization and discoloration, the composites containing maple exhibited greater discoloration than those containing pine. Irrespective of wood species, fungal colonization and discoloration in the composite lumber were greater at the bottom faces where they were in constant contact with moisture. The wood content range (50–100 phr) used in this study showed no effect on extent of fungal colonization and discoloration. All composites showed no discoloration in the interior test protocol. Both optical microscopy and environmental scanning electron microscopy clearly demonstrated that wood flour particulates are not completely encapsulated by the PVC matrix, so that exposed wood flour in the surface crevices of the composite lumber may serve as points of moisture sorption and staging points for fungal colonization and discoloration. J. Vinyl Addit. Technol. 10:179–186, 2004.

INTRODUCTION

Wood-plastic composites (WPCs) are among the fastest growing construction materials today. A market study undertaken by Principia Partners in November 2002 indicated that demand for WPC products in North America and Western Europe combined was expected to reach nearly 1.3 billion pounds valued at roughly $900 million in 2003, which represents a growth of almost 20% from 2001 levels (1). Current estimates place the annual growing rate at double-digit rates, and market growth in the next decade is projected to triple. In addition, the value of additives such as lubricants, coupling agents, and UV stabilizers, which improve the processing, performance, and appearance of WPC building products, is expected to top $100 million (2).

WPCs are used in both exterior and interior applications. Exterior applications such as decking and fencing expose installed WPCs to periodic or continuous regimes of high moisture, relative humidity, and fungal propagules, making them susceptible to fungal colonization and discoloration. Notably, WPCs have been marketed as "green" materials because they are devoid of biocides, yet are resistant to microbial colonization and discoloration (2). The latter observation is ascribed to the complete encapsulation of the wood-fiber component by the polymer matrix.

However, service performances of installed WPCs have proven otherwise (3–6). Decay fungi have been reported growing on externally installed WPCs (4). Previous work with high-density polyethylene (HDPE)/wood-flour composite lumber clearly demonstrated that fungi colonize

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and discolor this product when it is exposed to elevated moisture and ambient temperature conditions (3). Wood-plastic composites have also been shown to fade when exposed to sunlight and moisture environments (7–10). Most studies, however, on the biodeterioration of WPCs have centered on the investigation of polyolefins as matrices (3–7). Currently, only a limited amount of information is available on the susceptibility of PVC/wood-flour composites, although PVC represents one of the largest portions of the wood-plastic composites market, accounting for almost 30% of plastics used in the manufacture of wood-plastic composites (11).

Since WPCs containing rigid PVC and wood flour have increasingly become available on the market, this study was initiated to evaluate the susceptibility of rigid PVC/wood-flour composite lumber to fungal colonization and discoloration under simulated exterior (consistent contact with moisture) and interior conditions (high relative humidity and fungal air spora). This study also examined the effects of wood species and wood-flour content on fungal colonization and discoloration of the composites. Particular emphasis was placed on understanding how well wood flour was encapsulated by the polyvinyl chloride matrix, using Environmental Scanning Electron Microscopy (ESEM).

Background on Biodeterioration of Wood and Synthetic Polymers

Wood, a biopolymer, is inherently biodegradable unless it is naturally durable owing to the presence of extractives. Examples of such wood species in North America include black locust, cedar, black cherry, and redwood (12). Notable agents that can degrade wood include microorganisms (fungi and bacteria), insects (termites, carpenter ants, post-powder beetles, etc.) and marine organisms (marine borers). Decay, molds, and stains are caused by fungi, and both their occurrence and development depend strongly on temperature and moisture conditions.

Three major types of wood decay fungi are recognized: white-rot, brown-rot, and soft-rot. While white-rot fungi generally attack the three major chemical components of wood cell wall (cellulose, lignin, and hemicelluloses), some exhibit selectivity in the relative amounts removed (13). A case in point is while *Polyporus berkeleyi* degrade lignin more than the carbohydrates (cellulose, hemicelluloses), *Trametes versicolor* attack simultaneously lignin, cellulose, and hemicelluloses. Brown-rot fungi preferentially degrade the carbohydrate components of wood cell wall with minor modification of lignin (13). Wood is attacked by soft-rot fungi under elevated moisture content and is degraded from the surface inwards. Carbohydrate components of the wood cell wall are the main target of degradation (14). Depending on the fungi type, decay fungi break down the structural wood cell wall components by enzymatic and non-enzymatic mechanisms, resulting in significant reduction of the structural properties of wood (e.g., toughness, static bending) (15).

Unlike wood decay fungi, stain and mold fungi utilize simple carbohydrates (sugars and starch), leaving the structural wood cell wall components unaffected. Consequently, the ultimate effect of wood deterioration by mold and stain is the degradation of the aesthetic properties of wood. Stain fungi are usually associated with sapwood and impart discoloration to the wood as the result of the dark hyphae of the fungi. In addition to surface growth, stain fungi can grow deep into the wood. Mold fungi generally grow on the surface of wood and produce masses of colored spores from shades of green to black. Thus while molds produce shallow discoloration, stain fungi produce both surface and deep discoloration (15). Typical mold fungi include species belonging to the genera *Penicillium, Alternaria, Aspergillus, Trichoderma,* and *Gliocladium*.

Biodeterioration and biodegradation of synthetic polymers are influenced by physical and chemical factors such as (i) the nature of chemical bonds in the polymer backbone, (ii) the surface area, and (iii) the crystallinity (16). Other important factors include the molecular weight and hydrophobicity of the polymer, as well as the types of additives present in the polymer. Commodity synthetic polymers are generally considered as biologically "inert" when their molecular weight exceeds 1000 (16). However, synthetic polymers such as polyesters, polyamides and polyurethanes, which contain hydrolysable groups, are susceptible to biodeterioration. Additionally, such polymers containing aliphatic groups (e.g., poly-ε-caprolactone) are more susceptible to biodeterioration than those containing aromatic groups (17). Biodegradability is also influenced by the chemistry of the functional groups present in the polymer backbone. Presence of amino acid groups enhances biodegradation. A case in point is that polyester-based polyurethane is more susceptible to biodeterioration than polyether-linked polyurethane. Overall, many commodity plastics are deemed "biologically" inert (18). Wood-plastic components comprising commodity plastics and wood have also been considered as "biologically" inert on the premise that the wood particulates of fibers are completely encapsulated by the commodity plastic component.

EXPERIMENTAL

Materials

Two different wood species were used as flour: (i) southern pine (*Pinus* sp.), a softwood (Gymnosperm) and (ii) hard maple (*Acer saccharum*), a hardwood (Angiosperm). The particle size was 425 micron (40 mesh size) for both species; the wood flour was supplied by American Wood Fibers (Schofield, WI). These flours were oven-dried at 103 ± 2°C for approximately 48 hr before processing to remove moisture. The PVC matrix and other additives used in the manufacture of the composites are listed in *Table 1*. Solid green (i.e., undried) southern pine and maple sapwood lumber were used as controls.

Fungal cultures obtained from Presque Cultures, Erie, PA, were used in this study. Two sets of fungal

Table 1. Formulations Used in Rigid PVC/Wood-Flour Composites.

Ingredients	Contents (phr)
PVC (K value = 66) [Oxyvinyls]	100
Tin stabilizer (PlastiStab 2808) [OMG Americas]	2
Calcium stearate (Synpro)	1.5
Paraffin wax (Gulf Wax)	2
Processing aid (Paraloid K-120) [Rohm & Haas Co.]	2
Processing aid (Paraloid K-175) [Rohm & Haas Co.]	2
Impact modifier (K-334) [Rohm & Haas Co.]	10
Wood flour (American Wood Fibers)	50, 75, 100

consortia were employed as inocula. For the exterior test protocol, a mixed fungal suspension comprising of four mold fungi (Deuteromycetes): *Aspergillus niger, Penicillium pinophilum, Gliocladium virens,* and *Aureobasidium pullulans* were used. Also included in the fungal inocula was the soft-rot fungus *Chaetomium globosum*. However, for the interior test protocol, the inocula used to inoculate the soil bed comprised propagules of the following fungi: *Aureobasidium pullulans, Aspergiluus niger* and *Penicillium* sp.

Compounding and Manufacture of Rigid PVC/ Wood-Flour Composite Lumber

PVC matrix, dried wood flour (softwood or hardwood) and other relevant additives (*Table 1*) were dry-blended in a 10-L high-intensity mixer (Papenmeier, Type TGAHK20) for 5 min. Three levels of wood-flour content were used for each wood species: 50 phr, 75 phr, and 100 phr. The concentrations of other additives were kept constant (*Table 1*).

Once mixed, the blended compounds were processed in a 32-mm conical counter-rotating twin-screw extruder (C.W. Brabender Instruments Inc.) with a length-to-diameter (L/D) ratio of 13:1. The extruder was driven by a 7.6 Intelli-Torque Plasti-Corder Torque Rheometer®. The rotational speed of the screws was maintained at 50 rpm and the temperature profile from the hopper to the horizontal rectangular die was 190°C/ 175°C/170°C/180°C. Dimensions of the samples produced were 2.54 cm (width, 1") by 0.95 cm (depth, 3/8"). Extrudate samples were cut to 6.23-cm lengths for decay testing.

Preparation of Fungal Inocula for Use in Exterior and Interior Test Protocols

To mimic exterior conditions where the composite lumber is in constant contact with moisture, the ASTM standard G21-96 test protocol (19) was used, and the ASTM standard D3273-94 (20) was used to simulate interior environments with severe mold conditions.

For either protocol, fungal cultures were initially inoculated on 2% (weight/volume, wt/v) potato dextrose agar in petri plates at 28°C and 85% relative humidity until 75% of the surface of the petri plates was covered with fungal hyphae. Inocula consisted of spore/hyphae suspension from either of the two sets of fungi. Each fungal inoculum was prepared by adding incremental amounts of sterile solution of a nontoxic wetting agent (0.05 g/L of Tween 20) to the surface of each fungal-colonized petri plate. The surface of each petri plate was rubbed with the tip of a sterile glass rod to liberate spores and fragment hyphae. A total of 10 ml of nontoxic wetting agent (0.05 g/L of Tween 20) was used for each plate. The resulting fungal spore/hyphae suspensions from either set of fungi (for either exterior or interior test) were added, respectively, to a 125-ml Erlenmeyer flask containing glass beads (5-mm diameter) and 45-ml deionized sterile water. The Erlenmeyer flask was stoppered and shaken vigorously to liberate spores and fragment fungal hyphae. This suspension was then transferred to a 500-ml spray bottle and used as the source of fungal inocula for each respective test. Viability of fungal inocula for the external and internal test protocols was tested as per the ASTM standards G21 and D3273, respectively.

For the exterior test protocol, the fungal inocula were applied to test WPC samples by spraying test specimens sitting on the surface of nutrient-agar. However, for the interior test protocol, the prepared fungal inocula were applied to the soil bed in the chamber (*Fig. 1*); turned over a couple of times; and incubated at 21°C and 97% RH for a week. Viability of the chamber was tested by placing about four malt-agar petri plates (2% wt/v, with the lids off) on the fungal-inoculated soil bed in the test chamber. In a biologically active chamber, fungal growth is observed on the malt-agar plates in a week (20).

Susceptibility of Rigid PVC/Wood-Flour Composite Lumber to Mold Fungi Colonization and Discoloration Under Simulated Exterior Environment

WPC samples were placed on plastic mesh squares (75 × 75 mm) sitting on nutrient salts agar media. Nutrient salts agar media were prepared by dissolving the ingredients listed in *Table 2* in 1 L of deionized water. The prepared-nutrient solution was sterilized by autoclaving at 121°C for 20 min. After sterilization, the pH

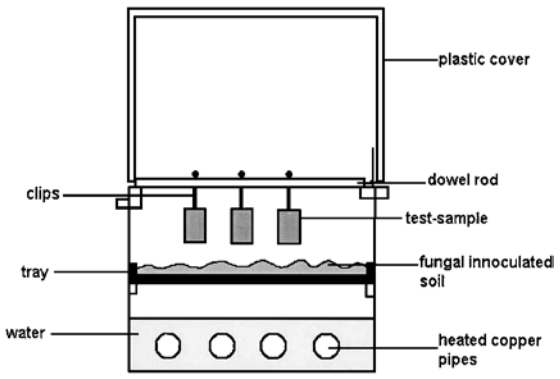

Fig. 1. Schematic representation of test chamber used for incubation under interior environments (ASTM Standard D3273).

Table 2. Formulation Used in the Preparation of Nutrient Salts Agar Media.

Ingredients	Amount (g)
Potassium dihydrogen orthophosphate	0.7
Potassium monohydrogen phosphate	0.7
Magnesium sulfate	0.7
Ammonium nitrate	1.0
Sodium chloride	0.005
Ferrous sulfate	0.0002
Zinc sulfate	0.002
Manganous sulfate	0.001
Agar	15

of the nutrient solution was adjusted to 6–6.5 using 10^{-2} mM sodium hydroxide solution. The nutrient solution was allowed to cool under aseptic conditions (Laminar Flow Hood); poured into sterile aluminum pans, and allowed to solidify.

In the absence of a specific standard test for evaluating the exterior susceptibility of WPCs to mold colonization and discoloration, our previous work (3) evaluated the susceptibility of HDPE/wood-flour composites to mold discoloration using the ASTM G21 and ASTM D4445 standards (21). The ASTM standard G21 test protocol produced greater mold colonization and discoloration with HDPE/wood-flour composites and was deemed a more sensitive and better test. Subsequently, this has been adopted in our lab as the preferred protocol for evaluating the susceptibility of WPCs to mold colonization and discoloration under external conditions.

As a consequence, the resistance of rigid PVC/wood-flour composites to mold discoloration under exterior environments was evaluated using the ASTM standard G21. A number of modifications were made to the protocol of the ASTM Standard G21 test: All test samples were evaluated under non-sterile conditions to better reflect service conditions. No test sample contained a moldicide. Aluminum baking pans measuring 30.5 cm by 30.5 cm (12" by 12") with plastic covers were used. The prescribed sterile nutrient-salts agar medium was poured into sterile aluminum pans under sterile conditions and allowed to solidify under aseptic conditions. Test samples were placed on sterile square inert plastic meshes sitting on the nutrient-salts agar media. Test samples and the nutrient-salts agar media were inoculated with the test inocula as per standard. Each aluminum pan containing inoculated test specimens was covered with plastic cover and incubated at 28°C and 85% relative humidity for 4 weeks. The WPC and solid wood lumber (controls) were evaluated for discoloration at the end of 2 and 4 weeks. Solid wood lumber (controls) was tested to verify fungal bio-activity.

Discoloration ratings of test specimens were on a 0–5 scale, where a rating of 0 indicated no visible fungal hyphae and a rating of 5 indicated 75%–100% fungal hyphae coverage. Top and bottom faces of each exposed sample were given separate rating scores. Selected test specimens were further examined both by light microscopy and environmental scanning microscopy.

The effect of wood-flour content on the susceptibility of rigid PVC/wood-flour composites to mold colonization and discoloration of the composites under simulated exterior environment was also investigated. The wood-flour contents in the composites made with both maple and southern pine ranged from 50 to 100 phr.

Susceptibility of Rigid PVC/Wood-Flour Composite Lumber to Mold Fungi Colonization and Discoloration Under Simulated Interior Environment

The resistance of rigid PVC/wood-flour composites to mold discoloration under interior environments was evaluated using the ASTM standard D3273-94 (20). This test protocol evaluates the resistance of material surface including coatings to surface mold growth in a severe interior environment (20). The chamber setup was as shown in *Fig. 1*.

Rigid PVC/wood-flour composites specimens containing 50 phr of wood flour were installed randomly in the test chamber by suspending them on wood rods from eye hooks screwed into one end of each panel. The lower ends of panels were approximately 75 mm from the surface of the fungal-inoculated soil bed. Solid southern pine and hard maple sapwood of the same dimensions as the WPCs were used as controls to verify fungal activity. The chamber cover was replaced and test specimens were allowed to run for 8 weeks. At 4-week and 8-week intervals, samples were removed from the chamber and examined for microbial colonization and discoloration. Microbial colonization and discoloration of both controls (solid wood) and rigid PVC/wood-flour composites were rated visually on a scale of 0 to 5. The scale of 0 and 5 represented no growth and 75%–100% microbial coverage of test panels. Ratings of 1, 2, 3 and 4 represented low microbial growth (< 5%), 5%–25%, 30%–50%, and 50%–75% microbial coverage, respectively.

Microscopy of Exposed Rigid PVC/Wood-Flour Composite Lumber

Optical microscopy was used to monitor the colonization and discoloration of the surface of rigid PVC/wood-flour composite lumber exposed to mold. Optical microscopy was performed using a Bausch and Lomb Stereozoom 7 microscope (magnification 20×).

The encapsulation of wood flour by the matrix and the growth of fungi spora on the surface of the exposed composites were studied with by means of environmental scanning electron microscopy (ESEM). For ESEM characterization, rigid PVC/wood-flour composite specimens cut from appropriate regions of exposed specimens were mounted on aluminum stubs using 3M double-sided tape and placed in an Electroscan 2020 ESEM chamber maintained at 3–5 Torr water vapor at room temperature as per the protocol of Dawson-Andoh and Kamdem (22). Each specimen was examined at different magnifications at different locations.

RESULTS AND DISCUSSION

Mold Susceptibility of the Composite Lumber Under Simulated Exterior Environment

Figure 2 illustrates the discoloration of both solid wood lumber (controls) and rigid PVC/wood-flour composites after 2 weeks (*Fig. 2a*) and 4 weeks (*Fig. 2b*) of incubation. Both maple and pine solid wood (controls) were colonized and discolored by the fungi, an indication of the susceptibility of green sapwood of the two wood species to microbial growth. Control maple lumber exhibited greater discoloration than pine, irrespective of the duration of the test (*Fig. 2*). This indicates greater inherent susceptibility of maple to microbial colonization and discoloration under test conditions. However, no discernible difference was observed in fungal colonization or discoloration between the top and bottom faces of either solid maple or solid southern pine lumber. Although the bottom face was more in constant contact with moisture, the top lumber faces maintained moisture content greater than the minimum (fiber saturation point) required for fungal colonization because of the high hygroscopicity of wood (12).

A different trend was noticed for the rigid PVC/wood-flour composites. Unlike the solid wood (controls), rigid PVC/wood-flour composites exhibited greater mold colonization and discoloration on the bottom faces than the top faces of the samples, regardless of wood species used in the composites and test duration (*Fig. 2*). Nevertheless, mold colonization and discoloration on the top face of the composites with maple flour was greater than for the composites with pine flour, in agreement with the higher fungal colonization and discoloration observed with the solid maple control. Moreover, the spores and hyphae of mold fungi growing on the surface of the composites are clearly shown in *Figs. 3* and *4*.

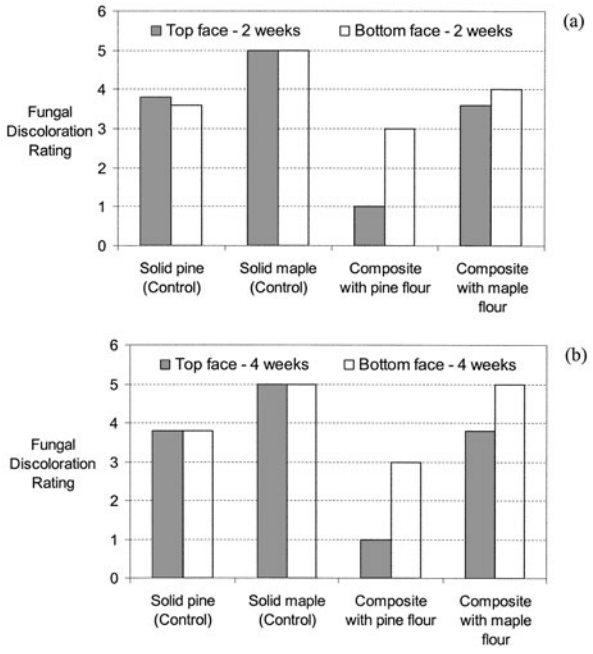

Fig. 2. Mold discoloration ratings of solid wood (controls) and rigid PVC/wood-flour composites after (a) 2 weeks and (b) 4 weeks of incubation under exterior environments (ASTM standard G21). The composites contained 50 phr of wood flour.

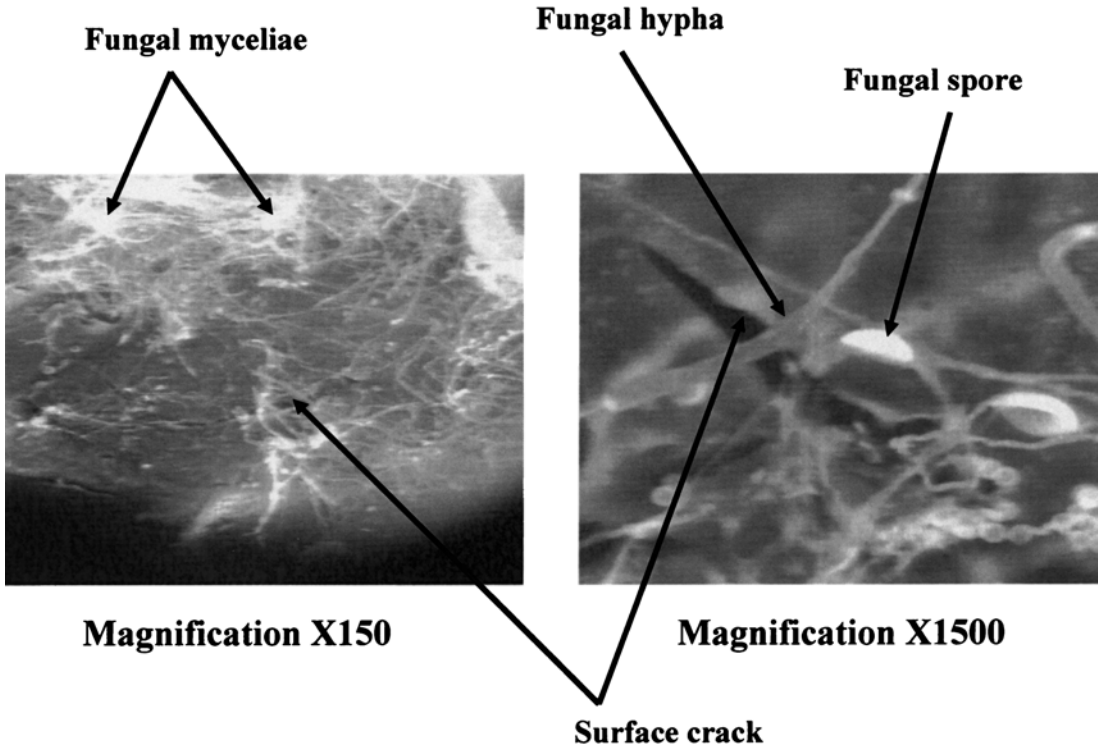

Fig. 3. ESEM micrographs of discolored rigid PVC/wood-flour composites showing surface breaks and spores/hyphae of mold fungi. The composites contained 50 phr of maple wood flour.

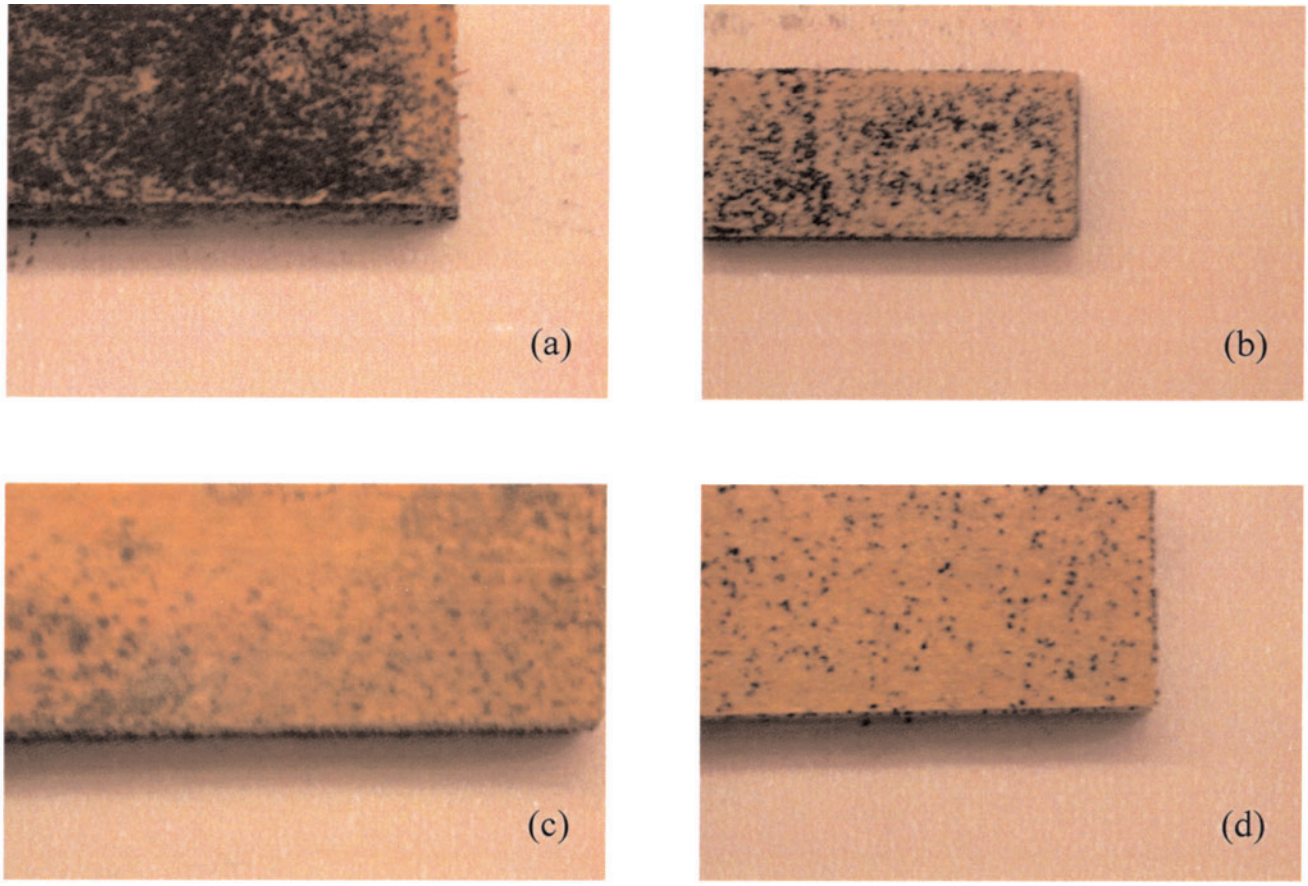

Fig. 4. Fungal colonization and discoloration of rigid PVC/wood-flour composite lumber after 4 weeks of incubation. The composites contained 50 phr of both maple flour (samples a and b) and pine flour (samples c and d). Specimens a and c represent the bottom faces; specimens b and d are for the top faces. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

The effect of wood content on the discoloration and mold colonization of rigid PVC/wood-flour composites is shown in *Fig. 5*. It is seen that wood-flour content exhibited no discernible effect on mold colonization and discoloration of the composites, regardless of the wood species. The results suggest that a threshold of wood-flour content is all that is necessary, and once this is met, microbial colonization and discoloration take place. Mold colonization is also a surface problem, and the presence and availability of exposed wood particulates on the surface of the WPCs is a prerequisite for microbial colonization. This was confirmed by the ESEM micrographs and is discussed in the following section.

Mold Susceptibility of the Composite Lumber Under Simulated Interior Environment

In the interior protocol, although both maple and pine lumber controls were colonized and discolored, none of the rigid PVC/wood-flour composite lumber (both maple and pine) exhibited fungal colonization or discoloration (data not shown). This is a reflection of the low hygroscopic nature of the WPCs. In the interior test protocol, moisture is available in the vapor form only in contrast to the exterior protocol where test samples are in constant contact with liquid water.

Hitherto, the general consensus has been that in WPCs, the polymer matrix completely encapsulates the wood particulates or fibers and minimizes its contact with water or sorption of moisture from the ambient environment. As a consequence, moisture required by fungi for germination, growth, and colonization is unavailable, and biodeterioration of WPCs does not occur. ESEM studies of discolored and non-discolored (not shown) rigid PVC/wood-flour composite lumber clearly showed that the synthetic polymer matrix does not completely encapsulate the wood flour (*Fig. 3*). Therefore, wood particulates are exposed to the ambient environment as a result of surface voids and cracks.

Deterioration of both wood and synthetic materials starts at the surface. Thus, the exposed wood particulates or fibers at the surface serve not only as points of moisture uptake, but also as a source of nutrient for fungal propagules that settle on these surfaces. Fungal colonization is initiated and anchored at these sites, and the resulting myceliae spread over the composite lumber surface. The authors have made similar observations in previous studies with HDPE/wood-flour composite lumber (3). In addition, sustained exposure to moisture is necessary for fungal colonization and discoloration of WPCs. This accounts for the difference in

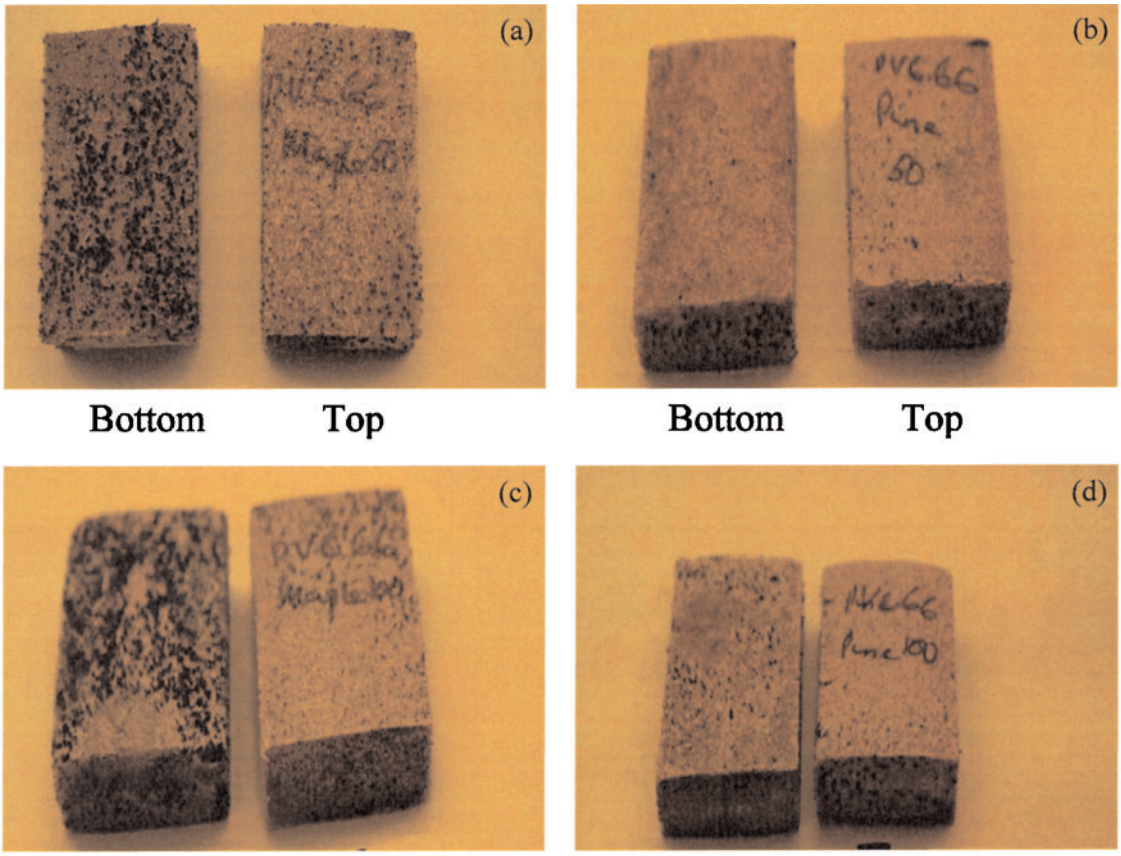

Fig. 5. Effect of wood-flour content on the mold colonization and discoloration of rigid PVC/wood-flour composites made with (a) 50 phr maple flour, (b) 50 phr pine flour, (c) 100 phr maple flour, and (d) 100 phr pine flour. The composites samples were incubated for 4 weeks. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

discoloration between top and bottom faces of rigid PVC/wood-flour composite lumber in the exterior protocol and the absence of discoloration in the interior test protocol.

CONCLUSIONS

Wood flour particulates in rigid PVC/wood-flour composite lumber are not completely encapsulated by the PVC matrix during processing, thus leaving some wood-flour particulates exposed and available for moisture sorption and subsequent fungal colonization. Rigid PVC/wood-flour composite lumber containing either maple or southern pine exhibited fungal colonization and discoloration when exposed to the exterior test protocol, which simulates exterior hazardous environments such as docks and decks. Regardless of the wood species, fungal colonization and discoloration in the exterior test protocol was greater at the bottom faces, where they are in constant contact with moisture. However, mold colonization and discoloration on the top face of the composites with maple flour was greater than for the composites with pine flour. The range of wood-flour content used in this study (50–100 phr) exhibited no effect on the degree of fungal colonization and discoloration on the composites.

In the absence of direct moisture contact, rigid PVC/wood-flour composite lumber did not show visible fungal colonization or discoloration in a severe mold environment as mimicked in the interior.

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