Wood and Bamboo-PP Composites: Fungal and Termite Resistance, Water Absorption, and FT-IR Analyses

S. Nami Kartal,[a,*] Sema Aysal,[a] Evren Terzi,[a] Nural Yılgör,[a] Tsuyoshi Yoshimura,[b] and Kunio Tsunoda [b,†]

This study evaluated biological resistance of composites produced from polypropylene and either wood or bamboo by using two different levels of particle content and three different particle sizes. Composite specimens containing higher particle content and smaller particle size resulted in increased mass losses in decay resistance tests against *Tyromyces palustris*, a standardized test fungus, *Schizophyllum commune*, and *Pycnoporus coccineus*. As particle content increased, mass losses in laboratory termite resistance tests increased; however, decreased particle size caused slightly decreased mass losses. Higher mass losses in bamboo-composites were obtained compared to mass losses in wood-composites in biological resistance tests. There is no significant effect of particle size on water absorption and thickness swell. The IR spectrums of composite specimens showed that significant changes were seen in the wood components following the application of heat during the manufacturing process. While the IR spectrum of WPC specimens with 70% wood was similar to the wood, the composite specimen with 50% wood displayed similarities to polypropylene.

Keywords: Wood plastic composites; Biological resistance; Schizophyllum commune; Pycnoporus coccineus; Coptotermes formosanus

*Contact information: a: Forestry Faculty, Istanbul University, P. O. Box 34473, Istanbul, Turkey; b: RISH, Kyoto University, P.O. 611-0011; *Corresponding author: snkartal@istanbul.edu.tr*

[†] Deceased on September 5th, 2011

INTRODUCTION

Biological performance of wood-plastic composites (WPCs) in field and laboratory tests has become a major interest as the demand for WPCs increases and they are increasingly used as alternative materials to treated and untreated wood. Even though WPCs are generally considered to be more resistant to biodegradation than wood due to encapsulation of wood by the plastic, decay rates in general are much slower than those in solid wood (Schmidt 1993; Naghipour 1996; Clemons 2002; Wang and Morrell 2004; Lomelí-Ramírez *et al*. 2009; Fabiyi *et al*. 2011), and the wood in WPCs still remains susceptible to decay (Morris and Cooper 1998; Mankowski and Morrell 2000; Verhey *et al*. 2001, 2002; Ibach and Clemons 2002; Pendleton *et al*. 2002; Silva *et al*. 2002; Simonsen *et al*. 2002; Ibach *et al*. 2003; Clemons and Ibach 2004). Since there are currently no standards for assessing the biological performance of WPC's in either laboratory tests or in field exposure, the durability of WPCs had been long assessed by standard tests such as soil block tests or agar tests developed for solid wood. American Wood Protection Association (AWPA), however, has recently suggested water immersion at either room temperature or 70ºC before decay testing of WPCs in the

standard test AWPA E10-12 to increase the moisture content of the specimens (AWPA 2012). High moisture levels are generally needed by microorganisms to attack WPCs; however, test methods without water immersion of WPC specimens do not produce enough mass loss on specimens due to the slow rate of water absorption.

Various characteristics such as density of the material, particle size of wood fibers, moisture content, biocides, and additives are important for biological durability of WPCs (Chow *et al.* 2002; Verhey and Laks 2002; Silva Guzman 2003; Klyosov 2007; McDonald *et al.* 2009; Morrell *et al*. 2010). Silva Guzman (2003) has reported that since the plastic is basically resistant to fungi, wood/plastic ratio in WPCs affects the decay resistance of WPCs. High wood content in WPCs generally results in faster water absorption since more hydrophilic material is present (Clemons 2002; Verhey *et al*. 2002). On the other hand, in general, WPCs produced with smaller wood particles show increased water resistance (Tatakani 2000) since such particles generally improve the interface between the wood fibers and the plastic and reduce voids in the interface area as pathways for moisture flow and colonization by fungi (Stark and Berger 1997; Mankowski and Morrell 2000; Verhey *et al.* 2002).

Mankowski *et al.* (2005) showed that WPCs in aboveground applications could also be susceptible to decay by fungi. Morris and Cooper (1998) observed the brown rot fungus *Gloeophyllum striatum* and the white rot fungus *Pycnoporus sanguineus* growing on WPC deck boards after 4 years in Florida. Mankowski *et al*. (2005) and Manning *et al.* (2006) also reported the presence of *Schizophyllum commune* and *Pycnoporus sanguineus* fruiting bodies on the surface of WPCs exposed for 18 and 30 months, respectively, in Hawaii. In the recent study, we have modified the Japanese standard test method JIS K 1571 (JIS 2004) to evaluate biological performance of the WPCs manufactured using soil substrate instead of quartz sand and adding wood chips as feeder in order to increase water absorption of the specimens during incubation and thus, increase mass losses in the specimens. In addition to the standardized brown rot fungus, *Tyromyces palustris*, three different strains of *Schizophyllum commune* and one strain of *Pycnoporus coccineus* that was observed on WPCs in previous studies stated above were used. Termite and mold resistance of the WPC specimens were also evaluated in laboratory tests. Water absorption and thickness swell tests were performed to observe the effects of particle content and size on water uptake of WPC specimens. FT-IR analyses were also run on some WPC specimens before and after decay tests.

EXPERIMENTAL

Production of Wood- and Bamboo-Polypropylene Composites

Composite samples were manufactured by MISAWA Homes Co. Ltd., Japan. The samples were prepared from either wood (hardwood/softwood mixture) or bamboo flour using two different levels of particle content (50 and 70%), three different particle sizes (30, 60, and 100 meshes), and commercialized polypropylene (PP) as a thermoplastic resin. Bamboo particles were selected as a natural fiber source which is abundant in Asia and South America. The PP was commercial homo-polymer (E-200GP) with a melting point of 160°C and a melt flow rate (MFR) of 2.0 g 10 min^{-1}. A blend of wood or bamboo flour with PP was compounded in a closed mixing blender for 10 min at a constant temperature of 180°C with a constant blender revolution of 30 rpm. Test samples were made using a 100 by 100 by 5 mm mold. The mold containing wood or bamboo flour,

PP, zinc borate (Zn borate) (1% by weight), and other additives stated in Table 1 as a footnote was heated to 180°C and pressed 45 sec at 2 MPa, then cooled at room temperature. The resulting WPC samples were cut to 20 by 20 by 5 mm specimens. WPC sample groups are shown in Table 1 along with the amounts of wood and bamboo flour and additives.

Decay Resistance Tests

A monoculture decay test was conducted according to the Japanese Industrial Standards JIS K 1571 (JIS 2004) with some modifications using the brown rot fungus, *Tyromyces (Fomitopsis) palustris* (Berkeley et Curtis) Murrill (FFPRI 0507), three different strains of *Schizophyllum commune* Fries (NBRC 4929, NBRC 30749, and NBRC 6504), and one strain of *Pycnoporus coccineus* (NBRC 9768) (Fries) Bondartzev & Singer (Syn: *Polystictus sanguineus*, *Trametes sanguinea*). The modifications were usage of screened garden soil instead of white sea sand in decay tests and thinner specimen size for both decay and termite tests (in the JIS standard, the specimen size is 20 x 20 x 10 mm; however, the test specimens were used in their original board thickness which is 5 mm due to fabrication process). The culture of *T. palustris* was obtained from RISH, Kyoto University, Kyoto, Japan, whilst the cultures of *S. commune* and *P. coccineus* were purchased from Biological Resource Center (NBRC), National Institute of Technology and Evaluation, Chiba, Japan. All cultures were maintained on dextrose-potato-agar medium at 27 ± 2ºC.

Liquid fungal cultures were prepared by inoculating 1000 mL of liquid medium, which contained 40 g glucose, 3 g peptone, 15 g malt extract, and 1000 mL distilled water with the fungi. The medium was shaken at 26 ± 2ºC for 10 days at 100 rpm.

Glass test bottles were filled with screened garden soil (8 to 20 mesh) and wetted with distilled water to bring the moisture content of the soil to 130% water holding capacity (WHC), as suggested by the American Wood Protection Association (AWPA) AWPA E10-12 standard method (AWPA 2010).

The soil was obtained from Takii & Co. Ltd. Japan and had the following properties: N: 330 mg/L; P: 570 mg/L; K: 480 mg/L; Cd: 0.004 mg/L; Hg: 0.0005 mg/L; As: 0.005 mg/L; Cr^{+6}: 0.02 mg/L; pH: weak acidic; water holding capacity (WHC): 200%. Five to six wooden sticks (2 to 3 cm in length and 2 to 3 mm in thickness) were placed on the top of the soil as feeders. The jars were then steam-sterilized at 103.4 kPa (15 psig) for 30 minutes and then inoculated with 3 mL of individual liquid fungal cultures and incubated at 27 ± 2ºC and 70 ± 2% relative humidity (RH) until the fungi completely colonized the feeders and topsoil.

After the oven-dried weights at 60ºC were determined, the test specimens (20 by 20 by 5 mm) were sterilized with gaseous ethylene oxide. Three specimens per composite group were placed in a pre-inoculated decay test jar on the surface of soil. Nine replicates were tested for each decay fungus. The test jars were then incubated at 27 ± 2ºC and 70 ± 2% RH for 12 weeks.

Following incubation, surface mycelium was brushed from each specimen before the specimens were oven-dried at 60ºC for 3 days. The extent of the fungal attack was expressed as the percentage of mass loss. In addition, moisture content of the WPC specimens was measured following incubation for 12 weeks.

Table 1. Formulations of Composite Groups Produced

Composite groups	Wood or bamboo content (%)	Particle size (mesh)	Wood or Bamboo content (g)	PP (g)	PE wax (g)	Ca-stearate (g)	Pigment I (g)	Pigment II (g)	Zn borate (g)	Total weight (g)
Wood composites										
1	50	60	30.69	25.12	0.56	0.56	0.28	2.79	-	60
2	50	30	30.69	25.12	0.56	0.56	0.28	2.79	-	60
3	70	60	41.86	13.95	0.56	0.56	0.28	2.79	-	60
4	70	30	41.86	13.95	0.56	0.56	0.28	2.79	-	60
5	70	60	41.26	13.95	0.56	0.56	0.28	2.79	0.6	60
6	70	30	41.26	13.95	0.56	0.56	0.28	2.79	0.6	60
7	50	100	30.69	25.12	0.56	0.56	0.28	2.79	-	60
8	70	100	41.86	13.95	0.56	0.56	0.28	2.79	-	60
9	70	100	41.26	13.95	0.56	0.56	0.28	2.79	0.6	60
Bamboo composites										
10	50	40	30.69	25.12	0.56	0.56	0.28	2.79	-	60
11	70	40	41.86	13.95	0.56	0.56	0.28	2.79	-	60
12	70	40	41.26	13.95	0.56	0.56	0.28	2.79	0.6	60

PP: Polypropylene; PE: Polyethylene; Pigment I: Purple oxide; Pigment II: Br pigment

Termite Resistance Tests

Test specimens (20 by 20 by 5 mm) were exposed to the subterranean termites, *Coptotermes formosanus* Shiraki, according to the JIS K 1571 standard method (JIS 2004). An acrylic cylinder (80 mm in diameter, 60 mm in height) whose lower end was sealed with a 5 mm thick hard plaster (GC New Plastone, Dental Stone, GC Dental Industrial Corp., Tokyo, Japan) was used as a container. A test specimen was placed at the center of the plaster bottom of the test container. A total of 150 worker termites collected from a laboratory colony of Research Institute for Sustainable Humanosphere (RISH), Kyoto University, Japan were introduced into each test container together with 15 termite soldiers. Three specimens per composite group were assayed against the termites. The assembled containers were set on damp cotton pads to supply water to the specimens and kept at 28 ± 2°C and >85 ± 2% RH in darkness for three weeks. The mass losses of the specimens due to termite attack were calculated based on the differences in the initial and final oven-dry (60°C, 3 days) weights of the specimens. Termite mortality and material consumption rates were also determined.

Mold Resistance Tests

The specimens (10 by 5 by 100 mm long) were evaluated for resistance to mold fungi according to the American Society for Testing and Material (ASTM) D4445-10 (ASTM 2010). Three mold fungi, *Aspergillus niger* 2.242, *Penicillium chrysogenum* PH02, and *Trichoderma viride* ATCC 20476 were grown and maintained on 2% malt agar (Difco, Detroit, MI, USA) at 27 ± 2°C, and 80% RH. A mixed spore suspension of the three test fungi was prepared by washing the surface of individual 2-week-old Petri plate cultures with 10 to 15 mL of sterile DI water. Washings were combined in a spray bottle and diluted to approximately 100 mL with DI water to yield approximately $3x10^7$ spores mL^{-1}. The spray bottle was adjusted to deliver 1 mL inoculum per spray. Specimens (five specimens per composite group) were sprayed with 1 mL of mixed mold spore suspension and incubated at 27 ± 2°C and 80% RH for 4 weeks. Following incubation, specimens were visually rated on a scale of 0 to 5 with 0 indicating that the specimen was completely free of mold growth and 5 indicating that the specimen was completely covered with mold growth.

Water Absorption and Thickness Swell Tests

Water absorption (WA) and thickness swell (TS) tests were determined by using five replicate specimens (20 by 20 by 5 mm) from each composite group, immersing them in water at 23°C for 30 days, and weighing them periodically. Weight gain and thickness swell were measured on a total composite basis for determination of WA and TS, respectively.

Water absorption (WA) was calculated according to the following formula,

$$WA\ (\%) = (M_c - M_o) / M_o \times 100, \quad (1)$$

where M_c is the mass of the specimen after immersion (g); M_o is the mass of the specimen before immersion (g).

Thickness swelling (TS) was calculated as follows,

$$TS\ (\%) = (t_c - t_o) / t_o \times 100, \quad (2)$$

where t_c is the thickness of the specimen after immersion (mm), and t_o is the thickness of the specimen before immersion (mm).

FT-IR Analyses

The FTIR absorption data were obtained using a Perkin Elmer 100 FT-IR Spectrometer combined with an ATR unit (Universal ATR Diamond Zn/Se) at a resolution of 4 cm^{-1} for 32 scans in the spectral range 600 to 4000 cm^{-1}. Measurements at three randomly chosen spots on the specimen surfaces were taken. IR spectra were also obtained directly from dried and milled wood powder used in WPC production. The spectra were baseline corrected and normalized to the highest peak. The analyses were performed on undecayed WPC specimens (50% wood / 50% PP and 70% wood / 30% PP with 60 mesh particle size only) and the same blend specimens exposed to *S. commune* (NBRC4929) and *T. palustris*.

Statistical Analysis

Statistical analysis was conducted using the SPSS program in conjunction with analysis of variance (ANOVA). Duncan's multiple range test (DMRT) was used to test statistical significance at α= 0.05 level.

RESULTS AND DISCUSSION

Density

As particle content in WPC specimens increased, air-dry density values increased; however, particle size had slight influences on air-dry densities (Table 2). As particle size decreased from 30 to 100 meshes, slight increases were seen in air-dry density values. The same trend was also observed in bamboo composite specimens when particle content was considered. In the study, air-dry densities in both WPC and bamboo composite specimens with Zn borate were slightly higher than those without Zn borate.

Decay Resistance

Mass losses in WPCs, bamboo composites, and sugi solid wood specimens in fungal decay resistance tests are summarized in Table 3. In general, the strains of *S. commune* resulted in similar mass losses in the specimens. As particle content increased from 50 to 70% in the specimens, mass losses increased. Particle size, however, had a mixed effect on the mass losses. Mass losses in the specimens with smaller particle size were generally higher than in those with bigger particle size with some exceptions. In general, the fungus *P. coccineus* caused more mass losses in WPC specimens compared to the strains of *S. commune*.

Particle content in the test specimens exposed to *P. coccineus* had a slight effect on mass loss. A mixed effect of particle size was also observed when *P. coccineus* was employed. The brown rot fungus, *T. palustris*, accounted for the highest mass losses in the WPC specimens. In the test specimens exposed to *T. palustris*, the effect of particle content was more distinct than those exposed to the other fungi tested; however, particle size had no significant effect on mass losses in the specimens.

Table 2. Densities of Composite Specimens

Composite groups	Wood or Bamboo content (%)	Particle size (mesh)	Air-dry density
Wood composites			
1	50	60	1.08 (0.03)AB
2	50	30	1.07 (0.01)AB
3	70	60	1.17 (0.01)A
4	70	30	1.15 (0.01)A
5	70	60	1.19 (0.02)A
6	70	30	1.17 (0.01)A
7	50	100	1.08 (0.02)AB
8	70	100	1.19 (0.02)A
9	70	100	1.20 (0.05)A
Bamboo composites			
10	50	40	1.04 (0.02)AB
11	70	40	1.13 (0.02)A
12	70	40	1.14 (0.02)A

Each value is the average of 30 specimens per composite group. Values in parentheses are standard deviations. The same letters in each column indicate that there is no statistical difference between the specimens according to Duncan's multiple range test ($p<0.05$).

Zn borate incorporated in WPC specimens resulted in lower mass losses in fungal resistance tests compared to untreated WPC specimens. As particle size in Zn borate incorporated-WPC specimens decreased from 30 to 100 mesh, mass losses in those specimens increased. A similar trend was also seen in untreated specimens with some exceptions or slight changes in mass losses.

As in WPC specimens, Zn borate decreased mass losses during fungal resistance tests in bamboo composite specimens. In case of the strains of *S. commune*, mass losses in the specimens with 70% particle content and Zn borate were higher than in those with 50% particle content and without Zn borate suggesting that particle content is an important key in degradation of bamboo composites by fungi and higher Zn borate content is needed to protect such products with particle content of 70%.

To summarize, as particle content increased, mass losses in the specimens increased decay resistance. The above findings are quite typical and it is well known that the more wood in the composite, the higher the potential for microbial degradation. However, in contrast to some previous studies, particle size in our study had a mixed effect on mass losses. Mass losses in the specimens with smaller particle size were generally higher than in those with bigger particle size with some exceptions.

Table 3. Fungal Decay Resistance Test Results

			S. commune NBRC4929		*S. commune* NBRC30749		*S. commune* NBRC6504		*P. coccineus* NBRC9768		*T. palustris*	
Composite groups	Wood or bamboo content (%)	Particle size (mesh)	Mass loss (%) Average	*Std Dev*	Mass loss (%) Average	*Std Dev*	Mass loss (%) Average	*Std Dev*	Mass loss (%) Average	*Std Dev*	Mass loss (%) Average	*Std Dev*
Wood composites												
1	50	60	1.71D	*0.27*	2.04CD	*0.15*	1.85E	*0.28*	3.63CD	*0.24*	4.21D	*0.34*
2	50	30	1.55DE	*0.61*	1.47DE	*0.23*	1.31EF	*0.18*	3.16CD	*0.27*	4.19D	*0.29*
3	70	60	4.7BC	*1.22*	3.04C	*0.25*	2.87D	*0.37*	4.44C	*0.68*	6.52C	*0.60*
4	70	30	2.64CD	*1.08*	1.99D	*0.24*	2.07DE	*0.33*	3.91C	*0.47*	6.47C	*0.84*
5 (Zn borate)	70	60	2.22CD	*0.29*	2.04CD	*0.12*	1.86E	*0.30*	1.74E	*0.30*	1.78EF	*0.12*
6 (Zn borate)	70	30	0.85E	*0.13*	1.37DE	*0.12*	1.25E	*0.24*	1.00EF	*0.14*	1.02F	*0.11*
7	50	100	1.93D	*0.57*	1.80D	*0.22*	1.73	*0.30*	2.90D	*0.28*	4.11D	*1.13*
8	70	100	3.43C	*0.21*	3.31C	*0.36*	3.54C	*0.40*	3.57CD	*0.36*	6.71C	*1.07*
9 (Zn borate)	70	100	2.49CD	*0.23*	2.42CD	*0.16*	2.35D	*0.23*	1.89DE	*0.22*	2.01E	*0.21*
Bamboo composites												
10	50	40	2.89CD	*0.21*	3.08C	*0.33*	3.21C	*0.29*	4.73C	*0.41*	5.17CD	*0.38*
11	70	40	5.29B	*0.21*	5.15B	*0.25*	6.39B	*1.82*	9.65B	*0.45*	8.03B	*0.81*
12 (Zn borate)	70	40	3.99C	*0.30*	3.80C	*0.33*	3.89C	*0.19*	3.59CD	*0.24*	3.15DE	*1.06*
Sugi sapwood	-	-	18.34A	*3.42*	17.21A	*4.01*	18.34A	*2.46*	21.13A	*3.89*	46.86A	*7.74*

Each value is the average of 9 specimens per composite group. Mass losses are presented on a total composite basis. The same letters in each column indicate that there is no statistical difference between the specimens according to Duncan's multiple range test ($p<0.05$).

Mankowski and Morrell (2000) found considerably more mass losses in pine/polyethylene composites with wood particle content of 70% when compared to composite specimens with wood content of 50%. Verhey *et al.* (2001) showed that the composites with higher wood content generally had more mass loss in decay tests. Stark and Berger (1997) and Pendleton *et al.* (2002) state that wood content rather than particle size may have a greater effect on the decay resistance of WPCs. Verhey *et al.* (2002) and (Verhey and Laks 2002) also show that WPCs with large particles are more susceptible to decay. This effect is attributed to better encapsulation of smaller wood particles by the plastic matrix and a uniform distribution in the matrix (Lomelí-Ramírez *et al.* 2009).

Moisture Content in Specimens after Decay Resistance Tests

Table 4 shows moisture content achieved by the specimens exposed to the fungi tested during 12 weeks of incubation in soil-block tests. Moisture content in solid wood specimens prepared from sugi sapwood varied from 85 to 154% during the decay process. The composite specimens, however, showed less moisture absorption changing from 25 to 42%. As expected, the specimens with higher particle content showed higher water absorption than those with lower particle content. Particle size, on the other hand, had an insignificant effect on moisture content in the specimens. Degree of fungal attack is closely related to moisture content of test specimen and increased mass losses are in general accompanied by increases in moisture content since water is crucial for colonization and decay of lignocellulosics by fungi (Lomelí-Ramírez *et al.* 2009; Silva Guzman 2003). Lomelí-Ramírez *et al.* (2009) states that proper moisture content to start decay by fungi is about 20%, and ideally this value is 30%; however, Silva Guzman (2003) suggests moisture levels need to be higher than 30%. Zabel and Morrell (1992) also state wood moisture content of 25 to 30% for fungal decay. Even though the plastic in WPCs encapsulates particles, the wood component generally reaches moisture levels that are proper for fungal decay due to un-encapsulated fibers near the surface (Silva Guzman 2003; Wang and Morrell 2004). Thus, it is highly possible to observe severe fungal attack near the surfaces of WPCs. Wang and Morrell (2004) and Silva *et al*. (2001) state that degradation by fungi is likely to be limited to the surfaces of WPCs, and the inner parts of the composites remain generally unaffected until sufficient moisture levels are achieved. Lomelí-Ramírez *et al*. (2009) also explains that the source of increased water content in specimens during decay process can be achieved by fungal respiration, which metabolizes wood components and produces water. Additionally, hyphae transport water from high humidity media into drier specimens (Ammer 1964; Muller *et al*. 2001).

Termite Resistance

Table 5 shows mass losses in the composite specimens following laboratory termite resistance tests with sugi (*Cryptomeria japonica*) solid wood specimens as controls. Termite mortalities and daily consumptions of the specimens by the termites during the 3-week-exposure are also shown in Table 5. As observed in decay resistance tests, mass losses increased when higher wood content (70%) was employed in the specimens. However, in termite resistance tests, as particle size decreased from 30 to 100 mesh, mass losses generally decreased. Termite mortalities in WPC specimens without Zn borate were higher than those in solid wood specimens. More particle content (70%) decreased termite mortalities when compared to particle content of 50% except for the specimens with a particle size of 100 meshes.

Table 4. Moisture Content in Composite Specimens after Decay Resistance Tests

			S. commune NBRC4929		*S. commune* NBRC30749		*S. commune* NBRC6504		*P. coccineus* NBRC9768		*T. palustris*	
Composite groups	Wood or bamboo content (%)	Particle size (mesh)	Moisture content (%) Average	*Std Dev*	Moisture content (%) Average	*Std Dev*	Moisture content (%) Average	*Std Dev*	Moisture content (%) Average	*Std Dev*	Moisture content (%) Average	*Std Dev*
Wood composites												
1	50	60	27.42C	*0.96*	26.85C	*0.76*	27.23C	*0.84*	28.66C	*0.78*	25.84C	*3.54*
2	50	30	27.61C	*1.90*	28.16C	*1.15*	27.09C	*0.72*	28.19C	*1.21*	27.03C	*1.10*
3	70	60	36.35B	*1.23*	37.17B	*1.52*	35.25B	*1.34*	39.31B	*1.24*	33.29B	*0.56*
4	70	30	39.26B	*1.41*	38.76B	*0.88*	38.04B	*1.24*	41.53B	*1.43*	39.19B	*0.67*
5 (Zn borate)	70	60	33.84B	*0.90*	32.71B	*0.64*	33.78B	*0.88*	33.17B	*0.76*	33.31B	*0.53*
6 (Zn borate)	70	30	34.17B	*0.70*	35.03B	*0.68*	33.52B	*0.96*	33.10B	*0.47*	33.68B	*0.64*
7	50	100	26.35C	*0.89*	26.56C	*1.17*	26.67C	*0.68*	26.84C	*0.55*	24.77C	*1.55*
8	70	100	36.08B	*1.32*	36.63B	*1.50*	36.35B	*1.04*	39.99B	*1.14*	36.59B	*1.50*
9 (Zn borate)	70	100	33.70B	*0.60*	33.67B	*0.47*	33.54B	*0.85*	32.49B	*0.48*	33.63B	*0.54*
Bamboo composites												
10	50	40	28.80C	*1.96*	27.94C	*2.69*	37.67B	*1.73*	28.06C	*1.63*	25.74C	*2.10*
11	70	40	33.89B	*0.90*	33.69B	*0.83*	33.42B	*1.48*	31.04B	*1.13*	30.53B	*2.09*
12 (Zn borate)	70	40	32.86B	*1.85*	31.83B	*0.67*	32.79B	*1.11*	31.22B	*0.74*	32.07B	*1.48*
Sugi sapwood	-	-	154.43A	*22.50*	88.69A	*17.63*	85.32A	*14.72*	86.20A	*10.95*	132.18A	*27.73*

Each value is the average of 9 specimens per composite group. Moisture contents are presented on a total composite basis. The same letters in each column indicate that there is no statistical difference between the specimens according to Duncan's multiple range test ($p<0.05$).

Table 5. Termite Resistance Test Results

Composite groups	Wood or bamboo content (%)	Particle size (mesh)	Mass loss (g) Average	*Std Dev*	Mass loss (%) Average	*Std Dev*	Termite mortality (%) Average	*Std Dev*	Consumption rate (ug/termite/day) Average	*Std Dev*
Wood composites										
1	50	60	0.06	*0.018*	2.75CD	*0.88*	27.9D	*0.61*	16.16CD	*5.19*
2	50	30	0.06	*0.005*	3.13C	*0.24*	25.8D	*0.31*	18.33C	*1.30*
3	70	60	0.17	*0.011*	7.50B	*0.40*	11.8E	*0.30*	50.36B	*3.03*
4	70	30	0.17	*0.003*	7.47B	*0.10*	13.0E	*0.30*	50.22B	*0.87*
5 (Zn borate)	70	60	0.05	*0.001*	2.16CD	*0.02*	87.6A	*0.91*	14.72D	*0.29*
6 (Zn borate)	70	30	0.04	*0.002*	1.54D	*0.09*	89.4A	*0.31*	10.68E	*0.58*
7	50	100	0.04	*0.002*	2.11CD	*0.08*	27.9D	*1.21*	12.55DE	*0.43*
8	70	100	0.16	*0.005*	6.58BC	*0.25*	27.0D	*2.12*	45.17BC	*1.59*
9 (Zn borate)	70	100	0.06	*0.002*	2.78CD	*0.14*	77.9B	*0.30*	18.47C	*0.58*
Bamboo composites										
10	50	40	0.07	*0.003*	3.34C	*0.21*	29.4D	*2.13*	19.34C	*0.87*
11	70	40	0.18	*0.017*	8.09B	*0.82*	17.9E	*0.91*	51.80B	*4.76*
12 (Zn borate)	70	40	0.07	*0.008*	3.26C	*0.29*	67.3C	*0.60*	21.21C	*2.16*
Sugi sapwood	-	-	0.23	*0.012*	18.33A	*1.61*	8.8F	*0.31*	65.37A	*3.32*

Each value is the average of 3 specimens per composite group. Mass losses are presented on a total composite basis. The same letters in each column indicate that there is no statistical difference between the specimens according to Duncan's multiple range test ($p<0.05$)

Bamboo composites also showed the same trends in termite mortalities as seen in WPC specimens. Zn borate incorporated-WPC specimens had considerably lower mass losses than untreated specimens, as expected, and termite mortalities were considerably higher in those specimens when compared to untreated specimens. As particle size decreased from 30 to 100 mesh, mass losses increased and termite mortalities decreased gradually in Zn borate incorporated-WPC specimens. In general, the mass losses in the specimens were compatible with the termite mortalities in the tests.

As particle content increased from 50 to 70%, mass losses in the specimens increased. Even though incorporation on Zn borate into specimens with particle size of 70% decreased mass losses, mass losses in those specimens were as high as in the specimens with particle size of 50% and without Zn borate, suggesting that particle content is an important factor in degradation of such composites by termites.

Kylosov (2007) states that WPC materials are commonly resistant to termites. Termites cannot get into the plastic matrix easily and can only slightly trim wood fibers at the surface of WPCs. This was also shown in our study, and as particle content increased from 50 to 70% in WPC specimens, mass losses due to termite attack generally increased by more than twice.

Mold Resistance

Mold resistance test results revealed that all WPC specimens and specimens produced by bamboo particles were covered completely by the three common mold fungi tested. Specimens were rated after just a 4-week-incubation, since complete fungal coverage was reached and rating of the specimens was discontinued. Control wood specimens from southern pine were also covered by mold growth at week 4. No inhibitory effect for added Zn borate was seen for any of the specimens. Since all specimens were rated 5 (100% coverage by the mold fungi), no relation between mold resistance and particle content/particle size was established in the study.

Laks *et al*. (2005) found that an increase in wood content in WPC specimens increased mold growth. Klyosov (2007) stated that Zn borate in amounts of 0.5, 1, and 2% in the WPCs practically stopped the microbial degradation in the latter in the AWPA laboratory soil block tests. However, the use of Zn borate did not completely control surface mold on the WPC decks exposed in outdoor field tests. One percent of Zn borate practically did not affect mold development on a 60-mesh-maple wood (particle content of 70%)-filled polyethylene, and only 3 and 5% of Zn borate were effective. Schirp *et al*. (2008) reported that mold and staining fungi may develop more rapidly on the WPC surfaces, and fungi belonging to such genera as *Aureobasidium*, *Aspergillus*, and *Penicillium* are important to evaluate organisms for both plastic and wood.

Water Absorption and Thickness Swell after 30-Day-Immersion

Water absorption of all composite specimens tended to be much lower than that of sugi solid wood specimens; however, thickness swellings in the composites were considerably higher than solid wood specimens, which is typical of composites (Table 6). Overall moisture content of the composite specimens varied from 16 to 25% during a 30-day-immersion period; however, sugi solid wood specimens had a moisture content of 75%, which is considerably higher when compared to the composite specimens. In WPCs, in general, smaller particles are more likely to be evenly encapsulated by the plastic material presenting a less continuous pathway for water uptake (Wang and Morrell 2004).

Table 6. Water Absorption and Thickness Swelling in Composite Specimens after 30-Day Immersion in Water

Composite groups	Wood or bamboo content (%)	Particle size (mesh)	Water absorption (%) Average	*Std Dev*	Thickness swelling (%) Average	*Std Dev*
Wood composites						
1	50	60	16.43C	*0.69*	9.73B	*0.79*
2	50	30	16.56C	*0.93*	9.46B	*0.99*
3	70	60	23.16B	*1.01*	15.13A	*0.79*
4	70	30	24.53B	*0.87*	15.73A	*1.02*
5 (Zn borate)	70	60	21.96B	*1.71*	14.62A	*0.79*
6 (Zn borate)	70	30	22.72B	*0.68*	14.46A	*0.96*
7	50	100	15.49	*1.59*	7.80C	*1.43*
8	70	100	22.38B	*0.80*	15.91A	*0.92*
9 (Zn borate)	70	100	21.36B	*1.12*	14.84A	*1.11*
Bamboo composites						
10	50	40	17.14C	1.15	6.00CD	0.99
11	70	40	21.20B	0.72	11.16B	0.81
12 (Zn borate)	70	40	20.93B	1.84	10.44B	0.62
Sugi sapwood	-	-	74.56A	2.45	1.25D	0.59

Each value is the average of 9 specimens per composite group on a total composite basis. The same letters in each column indicate that there is no statistical difference between the specimens according to Duncan's multiple range test ($p<0.05$).

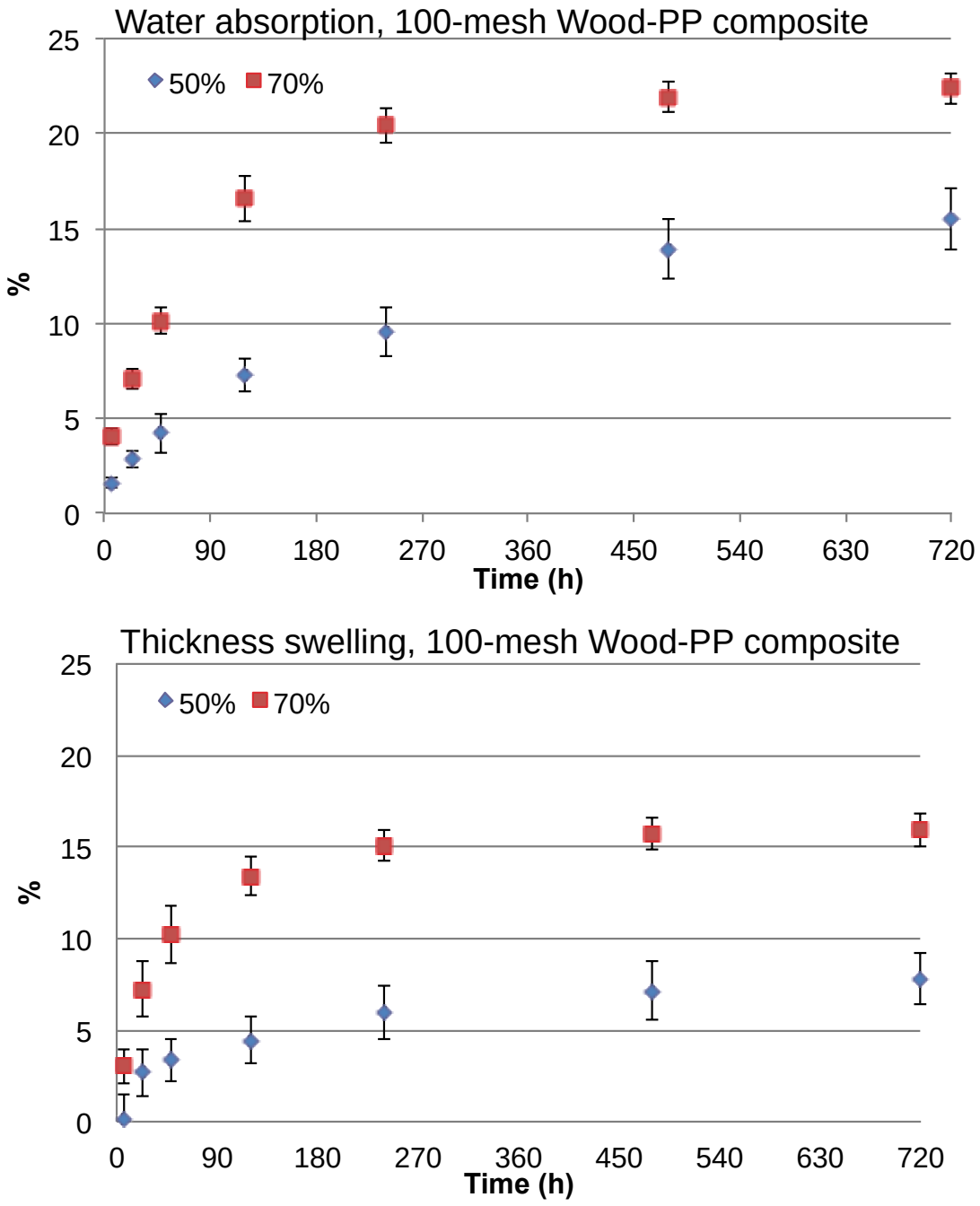

Fig. 1. Water absorption and thickness swelling of wood plastic composites

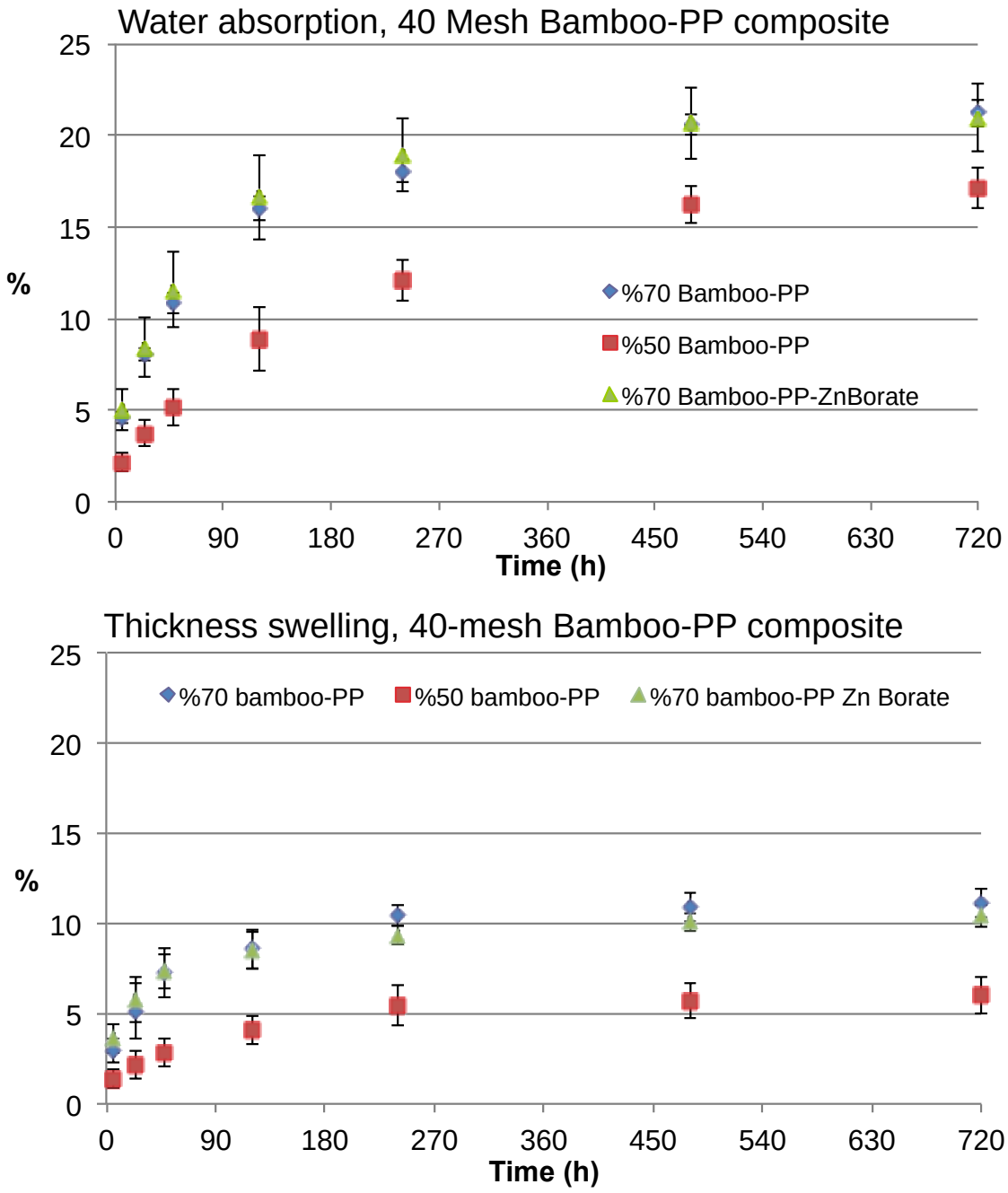

Fig. 2. Water absorption and thickness swelling of bamboo plastic composites

Fabiyi *et al.* (2011) state that compatibility between wood particles and plastic and shape of the particles can affect water absorption and dimensional change. In our study, even though we observed slight decreases in moisture content among the specimens as particle size decreased, they were not statistically significant. However, effect of particle content was predominant in water absorption and thickness swelling of the specimens. This is absolutely expected since the PP as a plastic material used in the composites is hydrophobic and the wood and bamboo particles are hydrophilic. With the

increase of the amount of wood and bamboo particles in the composites, their polar character increases, resulting in higher water content. Plotting water absorption versus time showed that water absorption curves increased linearly until reaching a pseudo-equilibrium level (Figs. 1 and 2). The time taken to reach this equilibrium level was identical for both wood and bamboo plastic composites tested.

FT-IR Analyses of WPC Specimens

Control WPC specimens, wood in WPC and PP

PP displayed a strong absorption band at 2916 (asymmetric CH_2 stretch), 1453, 1375, 996, and 971 cm^{-1}, as seen in Fig. 3. The wood species used in the WPCs is not known; however, IR bands give an idea that it might be a mixture of hardwood and softwood, as seen in Table 7. While the bands observed at 1505 and 1261 cm^{-1} demonstrated the presence of guaiacyl type of lignin (softwood lignin) in the wood mixture, the bands observed at 1318 and 1231 cm^{-1} indicate the presence of hardwood (Pandey and Pitman 2003).

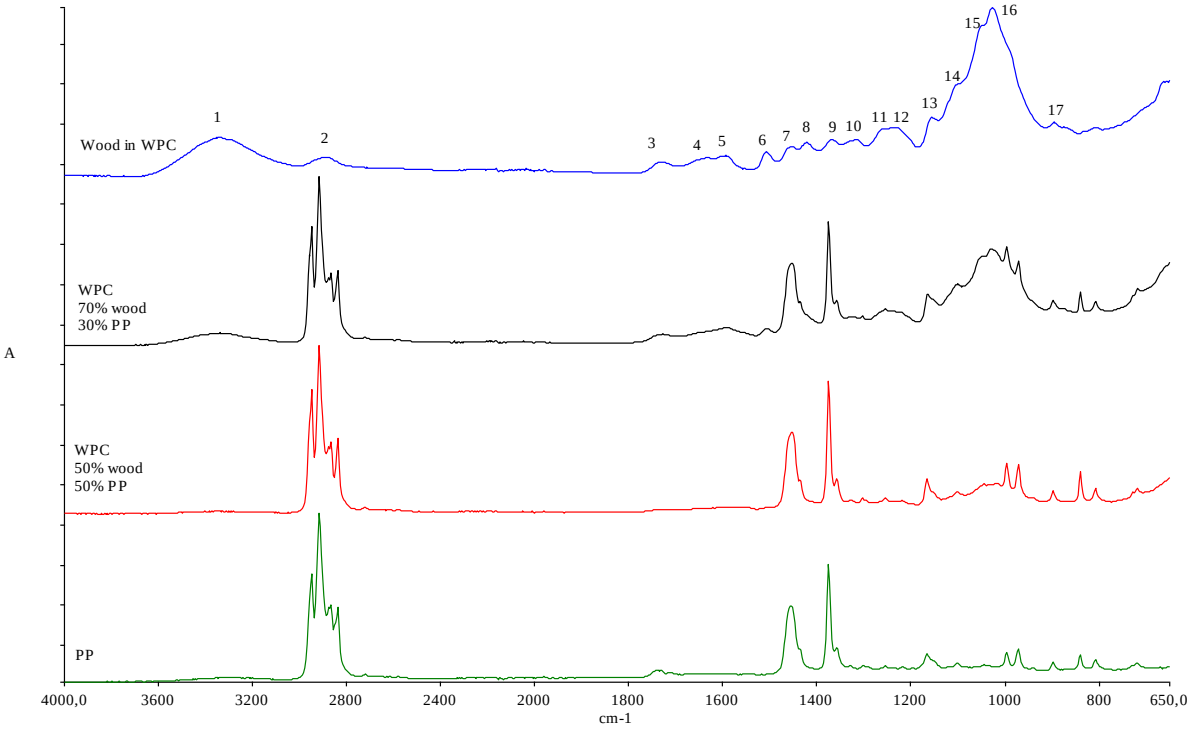

Fig. 3. FT-IR spectra of PP, wood used in WPC, and WPC specimens (70% wood / 30% PP and 50% wood / 50% PP)

Remarkable changes were seen in the wood components by the application of heat during the WPC production process (Müller *et al.* 2009). Even though the process temperature was about 180°C, the melting temperature of PP, it might have caused some important changes in the functional groups of the wood components. Figure 3 shows the differences between the spectra for wood and WPC specimens with 70% wood / 30% PP and 50% wood / 50% PP. The peak observed at 1732 cm^{-1} in wood spectrum (band number 3), as seen in Table 7, originates from holocellulose and indicates C=O stretch in unconjugated ketones, carbonyls, and ester groups from carbohydrate origin (Owen and Thomas 1989; Bodirlau *et al.* 2008) nearly disappeared in the spectrum for WPC

specimen with 50% wood / 50% PP. The band detected for wood at 3336 cm^{-1} (band number 1) indicated the intensity of O-H absorption. While the intensity of this band decreased in WPC, the specimen with 70% wood, it disappeared nearly in the WPC specimen with 50% wood. The hydroxyl group contents in wood are thought to be affected by heat treatment and showed a decrease due to the increased amount of wood content in the WPC specimen. The bands originating from holocellulose and cellulose at 1103, 1046, and 897 cm^{-1} were also absorption bands observed in both WPC specimens and the wood sample. While some of the bands in WPC specimens were shifted to lower bands, some of the bands shifted to higher bands compared to the wood sample due to the effect of high temperature and addition of PP and the other additives. Some of the spectral bands present in wood sample such as band numbers 2, 7, 8, and 9 (Table 7), were dominated in WPC specimens by the spectral bands in PP.

WPC specimen with 70% wood was more similar to the wood sample used to produce the WPCs compared to the other WPC specimen with 50% wood as seen in Fig. 3. As expected, WPC specimens with more PP (50%) displayed similar spectra to PP used in the manufacturing of the WPCs.

WPC specimens exposed to S. commune and T. palustris degradation

(i) comparison of WPC (undecayed) specimen (70% wood / 30% PP) to the same blend WPC specimens exposed to *S. commune* and *T. palustris*: The most remarkable differences in the spectra of WPC specimens exposed to either the white rot or the brown rot fungus were observed in the spectrum bands at 3336 (band number 1) and 1635 cm^{-1} (4) (Fig. 4). Since brown rot fungi degrade preferentially carbohydrates, the spectrum band at 1732 cm^{-1} (3) originated from holocellulose was lost, then the intensity of the band at 1635 cm^{-1} (4) increased. On the other hand, the intensity of the same spectral band in the sample exposed to the white fungus was lower than those exposed to the brown rot fungus due to degradation occurred in carbohydrates and lignin simultaneously. The WPC specimen exposed to *S. commune* displayed a more similar IR spectrum in the fingerprint region (1400 - 896 cm^{-1}) compared to undecayed WPC specimen; however, the WPC specimen exposed to *T. palustris* showed significant differences in this region. The differences might be arising from decomposition by *T. palustris* in mainly attacking carbohydrates. Besides decomposition of cellulose and the hemicelluloses, *T. palustris* also made some modifications in the lignin structure (Eriksson *et al.* 1990). Lignin modified by brown rot fungi is more reactive than native lignin due to the increased content of phenolic hydroxyl groups. It is also suggested that the methoxyl groups of lignin decrease but the carbonyl and carboxyl groups of lignin increase after the attack of brown rot fungi (Fengel and Wegener 1984). This could be an explanation for the strong absorption band at 1638 cm^{-1} observed in the WPC specimen exposed to *T. palustris.* The significant increase and the shifted band at 1635 cm^{-1} after the brown rot exposure may be due to the contribution of chitin, which is made of N-acetylglucosamine units (Nilsson and Bjurman, 1998). On the other hand, the spectrum bands at 1504 - 1505 cm^{-1} originating from lignin could be seen in both WPC specimens (50 and 70% wood) exposed to the fungi.

(ii) comparison of control WPC (undecayed) specimen (50% wood / 50% PP) to the same blend WPC specimens exposed to *S. commune* and *T. palustris*: The IR spectrum of WPC specimen with 50% wood / 50% PP exposed to *T. palustris* represents a similar spectrum to undecayed WPC specimen as seen in Fig. 5.

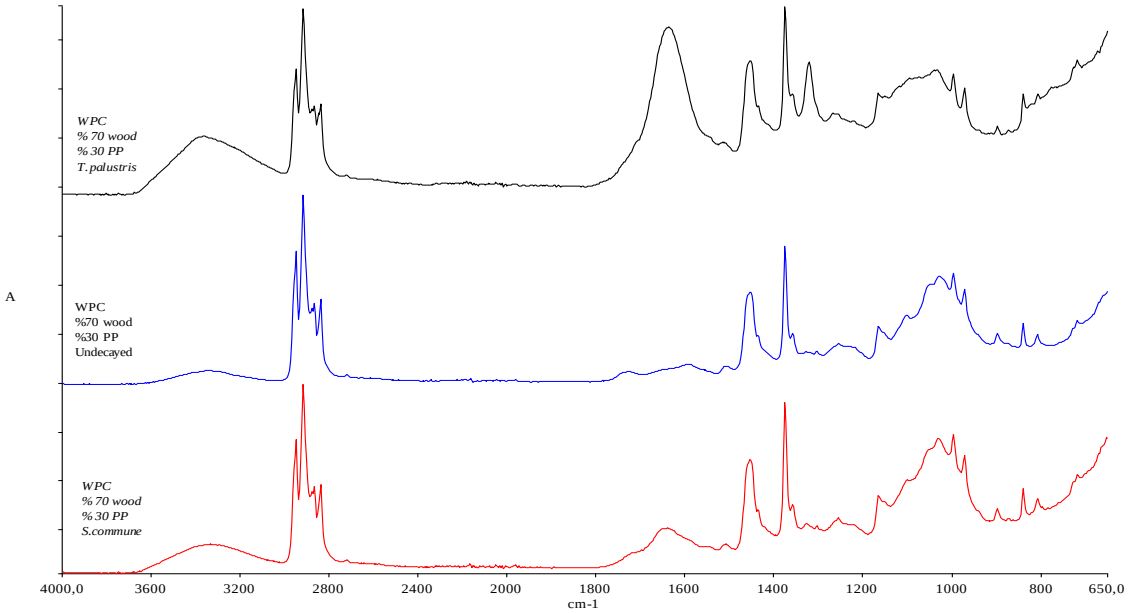

Fig. 4. Comparison of WPC specimen with 70% wood / 30% PP and the same blend WPC specimen exposed to *S. commune* and *T. palustris*

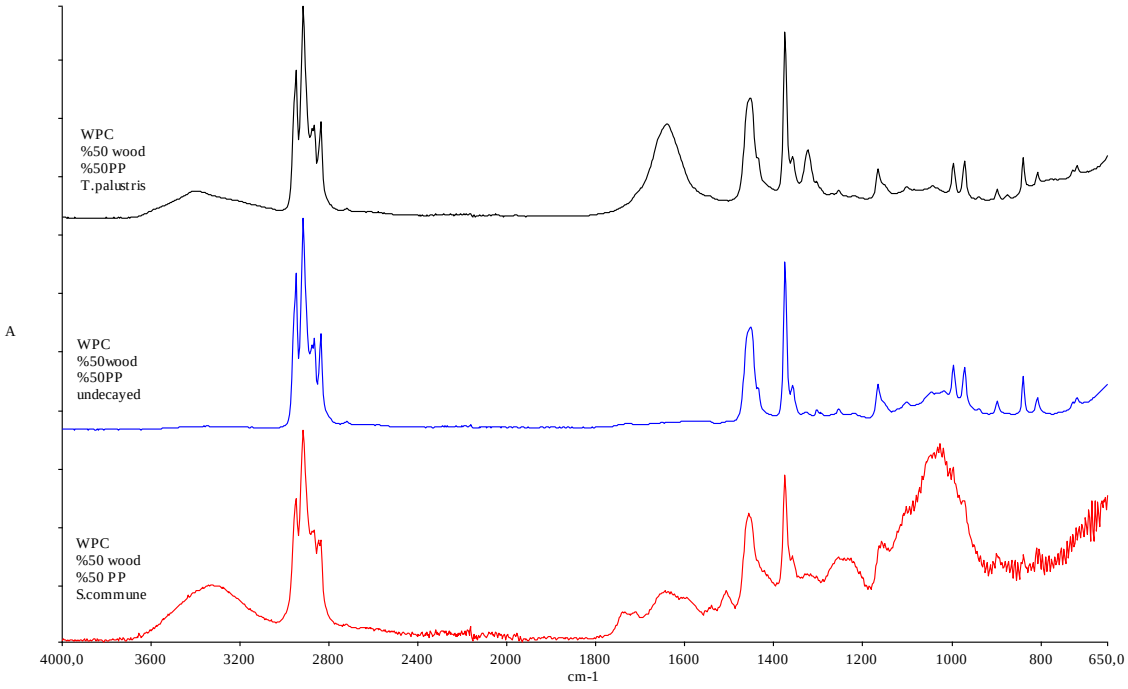

Fig. 5. Comparison of WPC specimen with 50% wood / 50% PP and the same blend WPC specimen exposed to *S. commune* and *T. palustris*

There were some significant differences in the spectra of WPC specimens exposed to the white rot and brown rot fungi such as the strong absorption band at 3336 (1) and 1635 cm^{-1} (4). As expected, the absorption band at 3336 cm^{-1} in this blend was not as strong as that in the WPC specimen with 70% wood due to its low wood content. The band at 1635 cm^{-1} in WPC specimen with 50% wood exposed to *S. commune* did not

display a strong intensity as well as the same blend WPC exposed to *T. palustris*, which is probably due to different decay processes of these two fungi. While most white rot fungi decompose lignin selectively depending on the extent of the exposure period, some brown rot fungi decompose carbohydrates and lignin simultaneously and they modify lignin structure (Fengel and Wegener 1984; Eriksson 1990). On the contrary, the IR spectrum of WPC specimen exposed to *S. commune* displayed more differences than the IR spectrum of WPC specimen exposed to *T. palustris* when compared to undecayed WPC specimen.

When the IR absorbance bands of both WPC specimens with 50 and 70% wood subjected to *S. commune* were compared, significant differences were observed in the spectral bands at 1260 and 1231 cm^{-1}. The band at 1260 cm^{-1} became more intense and significant in the WPC specimen including 70% of wood and exposed to *S. commune* compared to the specimen with 50% wood. This absorbance band represents the presence of guiacyl aromatic methoxyl groups (Pandey and Pitman 2003), and guiacyl units are more resistant to white rot fungi degradation (Hatakka 2001). In this study, FT-IR analyses showed that lignin still kept its original structure in the WPC specimen with 70% wood after *S. commune* attack. On the other hand, the decrease of the intensity of absorption band at 1231 cm^{-1} in the sample with 70% wood exposed to *S. commune* compared to the wood sample showed that xylan decomposition occurred after fungal attack. However, no change was observed in the relevant spectrum of WPC specimen with 50% wood exposed to the same fungus.

Table 7. Wave Number Characterization (Pandey and Pitman 2003; Müller *et al.* 2009)

Band number	Wave number (cm^{-1})	Assignments and remarks
1	3336	O-H stretch
2	2886	C-H stretch in methyl and methylene groups
3	1740-1730	C=O stretch in xylans (carbohydrate origin)
4	1635-1645	Absorbed O-H and conjugated C-O
5	1592	Aromatic skeletal in lignin
6	1505	Aromatic skeletal in lignin
7	1460-1455	C-H deformation in lignin and carbohydrates
8	1425-1421	C-H deformation in lignin and carbohydrates
9	1367	C- H deformation in cellulose and hemicellulose
10	1315	C- H vibration in cellulose and C-O vibration in syringyl derivatives
11	1262	Guaiacyl ring breathing, C-O stretch in lignin, C-O linkage in guaiacyl aromatic methoxyl groups
12	1228	Syringyl ring and C-O stretch in lignin and xylan
13	1153	C-O-C vibration in cellulose and hemicellulose
14	1102	O-H association band in cellulose and hemicellulose
15	1046	C-O stretch in cellulose and hemicellulose
16	1026	C-O stretch in cellulose and hemicellulose
17	897	C-H deformation in cellulose

Numbers in the table refer to the numbers assigned to the bands in wood (Fig. 3).

CONCLUSIONS

1. The composite specimens produced with higher particle content and smaller particle size experienced increased mass losses in decay resistance tests in most cases. Even though mass losses in the specimens in termite resistance tests increased as particle content increased, decreased particle size caused slightly decreased mass losses.
2. All composite specimens were colonized by the mold fungi in a short period of 4 weeks. In order to increase biological resistance of the specimens, higher loadings of Zn borate might be beneficial for providing more resistance against decay fungi and preventing mold growth in WPCs. The modified soil-block decay test can be used to specify WPCs that may be resistant or non-resistant to decay by fungi. Results of this study show a clear difference in mass losses of the composites with higher wood content without Zn borate preservative compared to the specimens with lower wood content amended with Zn borate.
3. As expected, the IR spectrum of WPC specimen including 70% wood is more similar to the wood sample than those of the specimen with 50% wood. The changes that occurred in the specimen with 70% wood exposed to the brown rot fungus are more significant than the same blended specimen exposed to the white rot fungus.
4. It was observed that lignin kept its original structure in WPC specimen with 70% wood and exposed to *S. commune*. Agar plate and soil block tests using either air-dried, water-soaked, or aged WPC specimens are in progress to evaluate the effect of initial moisture content of the specimens on susceptibility to decay fungi to compare the AWPA E10-12 standard method updated for decay tests for WPCs.

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