Degradation Behavior and Accelerated Weathering of Composite Boards Produced from Waste Tetra Pak® Packaging Materials

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Manufacturing panels from Tetra Pak® (TP) packaging material might be an alternative to conventional wood-based panels. This study evaluated some chemical and physical properties as well as biological, weathering, and fire performance of panels with and without zinc borate (ZnB) by using shredded TP packaging cartons. Such packaging material, a worldwide well-known multilayer beverage packaging system, is composed of cellulose, low-density polyethylene (LDPE), and aluminum (Al). Panels produced from waste TP packaging material were also examined by FT-IR to understand the fungal deterioration and extent of degradation after accelerated weathering. Before FT-IR investigations, panel specimens were ground under nitrogen atmosphere due to non-uniformity of the composite material. The FT-IR results showed that fungal degradation occurred in the natural polymer of the panel matrix. Although the natural polymer is mostly composed of cellulose, there were also small amounts of polyoses and lignin. It was seen that especially polyose and lignin bands in FT-IR spectra were affected more than cellulose bands by fungal attack. No changes were observed by the fungi in the plastic component (LDPE) of the matrix; however, LDPE seemed more sensitive to weathering than cellulose. Incorporation of ZnB at loading level of 1% (w/w) did not contribute fire performance of the panels when compared to control panel specimens, while a loading level of 10% improved fire performance considering test parameters such as mass loss, ignition time and peak heat release rate.

INTRODUCTION

Increasing the use of plastic based beverage and food containers causes decreased space in landfills and a serious problem for the environment. Since recycling of postconsumer packaging is of both environmental and economic concern, it might be important to find new solutions to recycle and reuse any polymers in waste composites that can be economically and environmentally viable (Howard 2002). Tetra Pak® (TP) is

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the best-known and largest producer of beverage packaging systems in the world. Such packages provide beverage and liquid foods to be easily transported and kept without the need to refrigerate; however, they eventually become a major component of the solid waste stream due to their short time of usage.

Beverage packaging systems are considered to be composite materials since they are composed of three different materials organized in six layers: duplex paper (~75% - cellulose fibers), low-density polyethylene (LDPE) (~20%), and aluminum Al, (~5%). So, TP packages can be identified as composites, which are defined as a material with two or more constituents, not mixed in anatomic scale (Weeton *et al.* 1987). At the same time, TP packages can also be accepted as wood plastic composite, referring to any composites that contain wood and thermoplastics (Clemons 2002). According to Stark and Matuana (2007), wood polymer composites are composed of wood or other natural lignocellulosic fibers in a thermoplastic matrix (Stark and Matuana 2007). Natural fibers primarily consist of cellulose, hemicellulose, and lignin. The primary element of TP packages is cellulosic fibers (average 75%), and it is being produced by the kraft (sulfate) and CTMP -(chemi-thermo-mechanical process) pulping methods in order to meet the high requirements on stiffness of the boards. Recycled fibers are never used because of the possibility of food-contact (Abreu 2000).

There are a few ways to recycle and reuse waste packaging cartons. Re-pulping is a common method to separate cellulose fibers from polyethylene and aluminum layers in a pulper and recycled fibers are used to produce cardboards, corrugated paper, tissue paper, egg carton, and new paper products (Zuben 1996; Lopes and Felisberti 2006). Aluminum and LDPE parts in the system are recovered for generation of energy through incineration, or Al is recovered in pyrolysis ovens and LDPE is used to produce high-end plastic products (Lopes and Felisberti 2006; Hidalgo 2011). The main disadvantages of re-pulping TP waste packages are that plugging of screeners and defibrators by polyethylene result in frequent pauses in the process for cleaning up and considerable fiber losses as well. In addition, nearly 25% of such material is still considered as waste for the pulp mills.

A possible usage of such waste packaging material might be panel production to be used in the furniture industry as an alternative raw material. The potential advantages of such waste for panel manufacturing can be considered as low energy demand, cost-effectiveness, easiness in recovery, and usage of whole material without leaving further waste when compared to conventional methods for wood-based panel production (Yilgor *et al.* 2012). Previous studies suggested that panel products from waste cartons could be an alternative raw material in the laminate industry by using suitable adhesive due to its improved mechanical properties, increased water resistance, and high biological performance against decay fungi and insects (Murathan *et al.* 2007; Ayrilmis *et al.* 2008; Sen *et al.* 2010; Hidalgo 2011). Thermal degradation characteristics and pyrolysis characteristics of TP waste were also studied (Kanturk Figen *et al.* 2013; Wu and Chang 2001; Korkmaz *et al.* 2009).

The deterioration of the material can be affected by many different factors such as biological organisms, light, thermal, chemical, and mechanical forces. In wood, cellulose, hemicellulose, and lignin exist as natural polymers, and they all provide a food source for fungi and other degrading organisms in case of sufficient moisture and oxygen (Stark and Gardner 2008). Different types of biological organisms prefer different wood polymers in wood polymer composites so it is not easy to understand the mechanism of biological degradation in such composites (Stark and Gardner 2008).

The interaction between hydrophilic natured cellulose and hydrophobic natured LDPE leads to weak bands causing micro-cracks in the composite matrix and water absorption occurs through the micro-cracks besides the fibers. However, the encapsulation of the fibers by molten thermoplastics retards the moisture absorption; thus wood polymer composites or cellulose polymer composites such as TP wastes seem more resistant to moisture compared to wood or laminated materials. Regarding outdoor applications of polymer composites that contain cellulosic materials, it is important to understand the water absorption behavior of these materials to improve of both dimensional stability and fungal resistance. Especially weathering causes remarkable degradation on the surface layers of the material exposed to UV light, and also affects durability and dimensional stability of the material (Segerholm *et al.* 2012). Both natural and artificial polymers are sensitive to the weathering effects of UV irradiation and oxidation (Stark and Gardner 2008). The degradation behavior and degradability characteristics of the composite materials after accelerated weathering are greatly influenced by the characterization of natural and artificial polymers and their distribution in the matrix. Even though all wood components are sensitive to weathering effects, lignin is the most sensitive polymer among the wood components which absorbs UV and is sensitive to weathering effects (Fengel and Wegener 1983; Stark and Gardner 2008). It is stated that weathering causes an increase in surface oxidation and a decrease in lignin content in wood polymer composites (Fabiyi *et al.* 2007). Since lignin can influence color change in wood polymer composites, using lignin free fibers in producing wood-based polymer composites might help to understand the mechanism of weathering.

Before designing and improving a new material, it is very important to consider the environmental conditions to which it will be exposed. The durability of TP panels might be of great importance because of their service life and maintenance. Therefore, there is a great interest in developing a new, alternative material in the panel industry. The objective of this study was to evaluate fungal and photo-degradation mechanisms of the panel specimens produced from waste beverage packaging cartons in moisture and decay resistance and weathering tests by using FT-IR spectroscopy. Termite resistance, fire performance, thermal characteristics, and some physical properties of the panels with and without zinc borate (ZnB) were also studied.

EXPERIMENTAL

Panels Manufacturing

Shredded waste Tetra Pak® (TP) packaging cartons were supplied by Yekasan Yeniden Kazanma A.S., a converting factory in Izmir, Turkey. A blend of shredded particles without any biocide or additives was compounded in a closed mixing blender with a constant blender revolution of 30 rpm.

Panels were made using a mold (60 mm by 60 mm) that was hot pressed at 170 to 180 °C for 5 to 7 min at 25 kg/cm^2 and then cooled at room temperature. The resulting panels were cut to 50 mm by 50 mm by 18 mm. Treated panels with 1% and 10% ZnB were used in biological performance tests.

In fire resistance tests and water absorption and thickness swelling tests, 10% zinc borate treated panels only were tested (Fig. 1a)

(a)   (b)

Fig. 1. (a) Tetra pak® panel specimen; (b) Ground material from panels

Chemical Analysis

Chemical characterization of paper used in TP beverage cartons was carried out to understand the degradation behavior of natural polymer in panels. Klason lignin content of paper was determined based on Runkel and Wilke (1951). Carbohydrates content of the hydrolysates was determined by anion exchange high performance liquid chromatography (HPLC) using pulsed amperometric detection (Davis 1998). Wood sugars were quantitated using an internal standard method and results were reported in terms of percentage of original specimen mass.

SEM Investigations

Scanning electron microscopy (SEM) on TP panel specimens were performed using a Carl Zeiss EVO40 scanning electron microscope (Carl Zeiss SMT, Inc., Thornwood, New York).

Water and Moisture Absorption and Swelling Tests

Water sorption and swelling tests

TP panels (one control and one with 10% ZnB) were cut into 20 mm by 20 mm specimens. Specimens were soaked in reversed osmosis water for 14 days at room temperature. At various intervals, the weight and dimensional changes were then measured.

Moisture sorption

All specimens were oven dried at 105 °C in a forced draft oven for 24 h, cooled 1 h in a desiccator, weighed, and then put in a controlled conditioning room set at 27 °C and 90% relative humidity (RH). Weight measurements were taken at various time intervals. The percentage moisture sorption was then calculated.

Biological Performance of TP Panels

Decay resistance tests – ASTM D2017-05

Specimens (19 mm by 19 mm by thickness) were tested against two brown-rot and two white-rot fungi in soil block tests based on the ASTM D2017-05 standard method (ASTM, 2010). In the tests, southern pine feeders were inoculated with the brown-rot fungi *Tyromyces palustris (Fomitopsis)* (FFPRI – Forestry and Forest Products Research Institute- 0507) and *Gloeophyllum trabeum* MAD 617, and maple feeders were

inoculated with the white-rot fungus *Trametes versicolor* MAD 697 and *Irpex lacteus*. Following incubation, average percentage mass loss was calculated for each specimen.

Decay resistance tests – JIS K 1571

Specimens with the dimension of 20 mm by 20 mm by thickness were prepared for decay resistance tests. A monoculture decay test was conducted according to JIS K 1571 (JIS, 2004) using the brown-rot fungus, *T. palustris* and the white-rot fungus, *T. versicolor* FFPRI 1030 (JIS, 2004). The test jars were then incubated at 27 °C for 12 weeks. The extent of the fungal attack was expressed as the percentage of weight loss.

Termite resistance tests – JIS K 1571

Test specimens (20 mm by 20 mm by thickness) were exposed to the subterranean termites, *Coptotermes formosanus* Shiraki, according to the JIS K 1571 standard method (JIS 2004). 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. 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 and termite mortality and material consumption rates were determined.

Termite resistance tests - ASTM D3345-08

A no-choice termite resistance test with *Reticulitermes flavipes* Kollar (Eastern subterranean termites) was performed using five specimens (25 mm by 25 mm by thickness). Termites were collected from Janesville, WI, USA. The containers were maintained at 27 °C and 85% RH for four weeks based on the ASTM D3345-08 standard method (ASTM 2010). At the end of the test, specimens were reweighed to calculate mass losses.

Mold resistance tests

The specimens (10 mm by thickness 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 3×10^7 spores mL^{-1}. The spray bottle was adjusted to deliver 1 mL inoculum per spray.

Ten specimens 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.

Accelerated Weathering Tests

Accelerated weathering tests were performed in a Xenon arc radiation cabinet (Atlas Xenotest Alpha+, ATLAS), which included water spraying according to ISO 4892-2-A1 (ISO, 2006). The radiation source was a Xenon arc lamp (300 to 400 nm), using borosilicate filters. The test consisted of cycles of 102 min of radiation, a black body temperature of 65 ± 2 °C, and relative humidity of $60\pm5\%$, followed by 18 min of water spray at the same radiation conditions, and a relative approximately of 100%. The total duration of the test was 576 h (144 h x 4 periods) for 3 specimens (45 by 135 mm) per each test group.

Fire Performance Test

Treated and untreated TP panel specimens, along with commercial plywood and solid wood (*Pinus sylvestris*) specimens, were investigated for fire properties using a Mass Loss Calorimeter (with thermopile and chimney) (Fire Testing Technology Ltd, UK) in accordance with ISO 13927 considering the following parameters: Time to ignition (TTI), peak heat release rate (PHRR), average heat release rates (AHRR) for 180 seconds, and 300 seconds and mass loss percentage (MLP) (ISO 13927, 2001). Specimens were conditioned at 23 °C, 50% RH prior to testing. Specimens (100 mm by 100 mm by 9 mm) were tested in horizontal orientation with the conical radiant electric heater located above the specimen. The temperature of the conical heater was adjusted to produce an external heat flux of 35 kW/m^2 on exposed specimens. Heat release rates, mass loss percentages, total heat released, and ignition times from samples were measured at the external heat flux of 35 kW/m^2.

Thermal Analysis

The effect of ZB loading on the thermal properties and thermal stability of TP panel were investigated by a thermo gravimetric analysis (TG). Characteristic temperatures associated with thermal degradation reactions and residues of the composites after the degradation were determined. Thermal analysis of ZnB, TP panel, and 10% ZnB treated TP panel were carried out using the Perkin Elmer Diamond thermal analysis instrument, which was calibrated using the melting points of indium (T_m=156.6 °C) and tin (T_m=231.9 °C) under the same conditions as the sample. The analyses were carried out with ground samples from panels at 10 °C/min in an atmosphere of N_2 that had a constant flow rate of 200 mL/min. The samples (~5 mg) were allowed to settle in standard platinum crucibles and heated up to 700 °C. Ground samples were prepared with the same procedure based on cryogenic grinding described below.

FTIR-ATR Analysis

The IR absorption data were obtained by 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}. Ground specimens were put in the cell, and the analyses were performed on control specimens and specimens exposed to the fungi and weathering experiment. The spectra were baseline corrected and normalized to the highest peak. FT-IR spectroscopy was improved by an attenuated total reflectance (ATR) unit, which provided direct interaction of the measuring beam with the sample and reflected the attenuated radiation to the spectrometer and increased the sensitivity of FT-IR-based analyses.

Preparation of specimens for FT-IR analyses

Due to the non-uniform nature of composite materials, it is an important task to obtain a homogenous material that represents the properties of the whole specimen as much as possible. It was thought that grinding the samples would be the right way to obtain homogenous specimens; however the panels contained three different components possessing entirely different hardening properties. These three components, -paper, LDPE, and aluminum-, vary in terms of their specific surface area, density, micro-hardness, compressive strength, tensile strength, hygroscopicity, adsorption, and absorption properties. Moreover, thermoplastics are particularly difficult to grind to a small particle size at room temperature. Thus, cryogenic grinding known as freezer milling was used in this study to eliminate the disadvantages of the samples as mentioned (Nguyen *et al.* 2007). According to the procedure, samples were divided into small pieces and put into the special jars, then were cooled by liquid nitrogen and beaten in the machine (Fig. 1b). Due to the differences in degradability between biological attacks and weathering effects, two different procedures were followed in grinding the test samples: Since fungal attack started in the surface of the panels and continued to advance into the inside of the samples, the whole samples exposed to fungi were ground. In the second procedure, the specimens obtained from the surface layers of the samples were cut and then ground, since the effect of UV degradation is limited to a 1 to 2 mm thick surface layer.

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

Sample Characterization

TP packaging materials are composed of three different components organized in six layers: duplex paper (75% -cellulose fibers), low-density polyethylene (LDPE) (20%), and aluminum (5%). The primary element of TP panels is cellulosic fibers, averaging 75%, and the second primary element is LDPE, which is known as a thermoplastic material, 20%. The third component is Al, which is a metal. Even though it is accepted that 75% of the material is composed of cellulose fibers, it is not pure cellulose. The paper used in TP packaging material is being produced by the kraft process, also known as the kraft pulping, which is a chemical process. It is accepted that more than 95% of the lignin in wood is removed by the kraft process. After the kraft process, there are still some polyoses such as xylose and mannose in addition to some lignin. Table 1 shows the chemical characterization of paper used in TP packaging.

Table 1. Chemical Characterization of Paper Used in Tetra Pak® Packaging Material

Lignin %	Arabinan %	Galactan %	Glucan %	Xylan %	Mannan %	Total Carbohy. %	Total Yield %
3.9	0.1	0.82	81.65	8.83	4.7	96.1	100

Water Absorption and Thickness Swelling Tests Results

Results of the water absorption and thickness swelling of TP panel specimens are given in Figs. 2 and 3, respectively. The specimens absorbed water quickly in the first 48 h; however, water uptake continued over the time of immersion. Plotting water absorption versus time showed that water absorption curves continued increasing linearly until reaching a pseudo-equilibrium level which is beyond 336 h immersion (Fig. 2). Control specimens showed slightly higher water absorption than ZnB treated specimens. Thickness swell curves for control and ZnB treated specimens were essentially identical to the water absorption curves obtained.
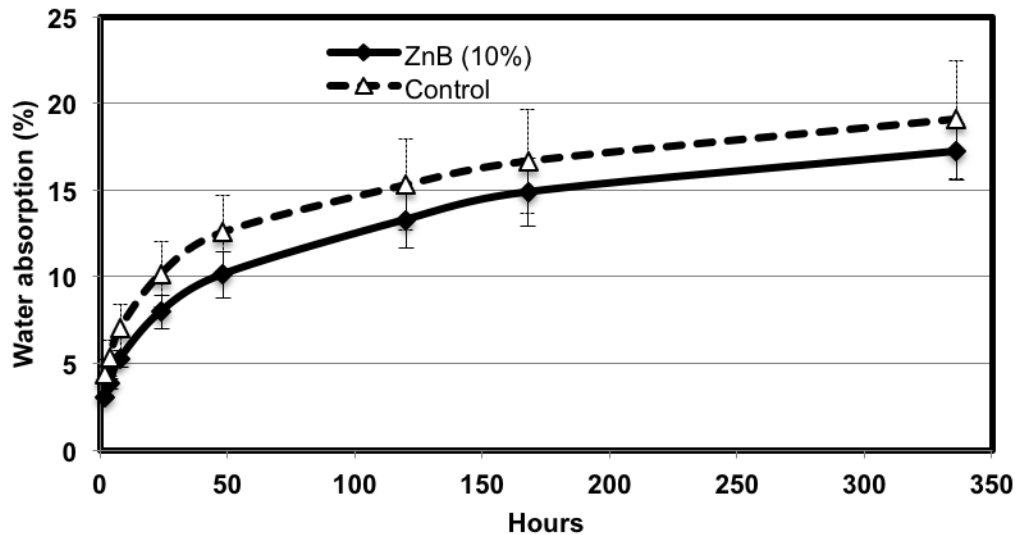

Fig. 2. Comparison of water absorption of TP panel and panel with 10% ZnB
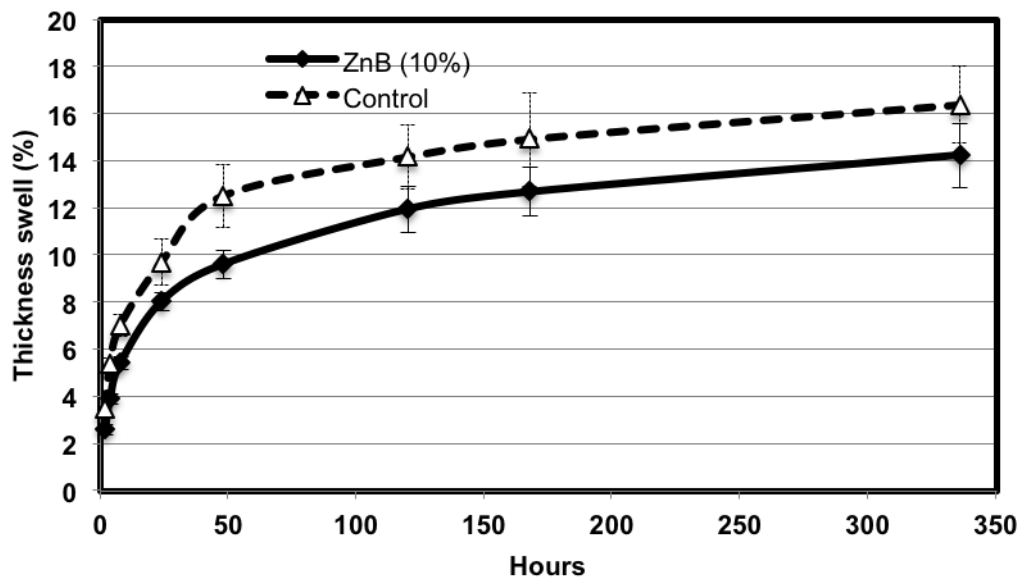

Fig. 3. Comparison of thickness swelling of TP panel and panel with 10% ZnB

It is known that the content of composite materials has an effect on water absorption and thickness swelling ability. Water absorption is greatly related to fiber

type, fiber volume, and fiber area of exposed water, besides many factors such as temperature, interfacial bonding, diffusivity, and reaction between water and matrix (Deng *et al.* 2010; Stamboulis *et al.* 2001). It is also obvious that water absorption is being achieved by cellulosic fibers only in TP panel specimens because of the hydrophobic character of LDPE.

Absorption of water in composite materials takes place in three different ways (Espert *et al.* 2004). The first and main process is penetration of water molecules through the micro-gaps between polymer chains. The second way is capillary transport of water through the small cracks and fractures at the interfaces between natural fibers and polymer, and the third way is absorption of water by micro-gaps and cracks in the structure formed during processing of the composite material (Espert *et al.* 2004; Lin *et al.* 2002).

Among wood cell components, cellulose is the most hydrophilic polymer, and absorption of water by cellulose depends on the number of free OH-groups (Fengel and Wegener 1983). The hydrophilic character of cellulose fibers is responsible for the water absorption in the composite matrix; there might be a linear correlation between the natural fiber content and water absorption ability of panels (Espert *et al.* 2004). Kartal *et al.* (2013) studied composite panels with different wood and polypropylene blends of 50%/50% and 70%/30%. According to their findings, while 50% of wood included panels absorbed 16.43% of water, 70% of wood included panels absorbed 23.16% of water after 720 h, and in the case of ZnB treatments, water absorption in specimens with 70% wood flour decreased by 5.2%. In that study, 336 h water absorption value was estimated as 22%.

In our study TP panel specimens with 75% cellulose fiber absorbed 19.01% water, while specimens with ZnB absorbed 17.2% water after 336 h immersion (Fig. 2). Apart from the type of material in the composite matrix, *i.e.* wood, cellulose, *etc.*, there are many factors that affect water absorption and thickness swell: natural polymers' shape and particle size, application conditions, and whether any pretreatment is applied on the components of composite material, *etc.*

In the present study, the paper part of TP beverage cartons was composed of cellulose fibers as long strips in the composite, while smaller particles such as wood flour are employed in wood-polymer composites. Since the kraft process, an alkaline procedure, has been used to obtain those cellulose fibers, the fibers in the composite are no longer natural cellulose. Thus, alkali treatment may be favorable in encapsulation of fibers by LDPE in a TP matrix, resulting in a decrease in water uptake. While the thickness swell rate was 16.4% in the control specimen, it was 14.3% in the specimen with ZnB (Fig. 3). Adding ZnB into the panels might have resulted in less accessibility of water into the fibers.

Figure 4(a) and (c) show the appearance of the panels before water absorption, and Fig. 4(b) and (d) show the panels after water absorption. The entrance of water may have taken place through the small cracks and fractures in the structure formed during processing of the panels. Moreover, transportation and penetration of water might have been achieved through the interfaces between natural fibers and polymer. Figure 4(a) shows that the fibers were not encapsulated sufficiently by melted LDPE, and the penetration of water might have occurred in those regions (Fig. 4b).
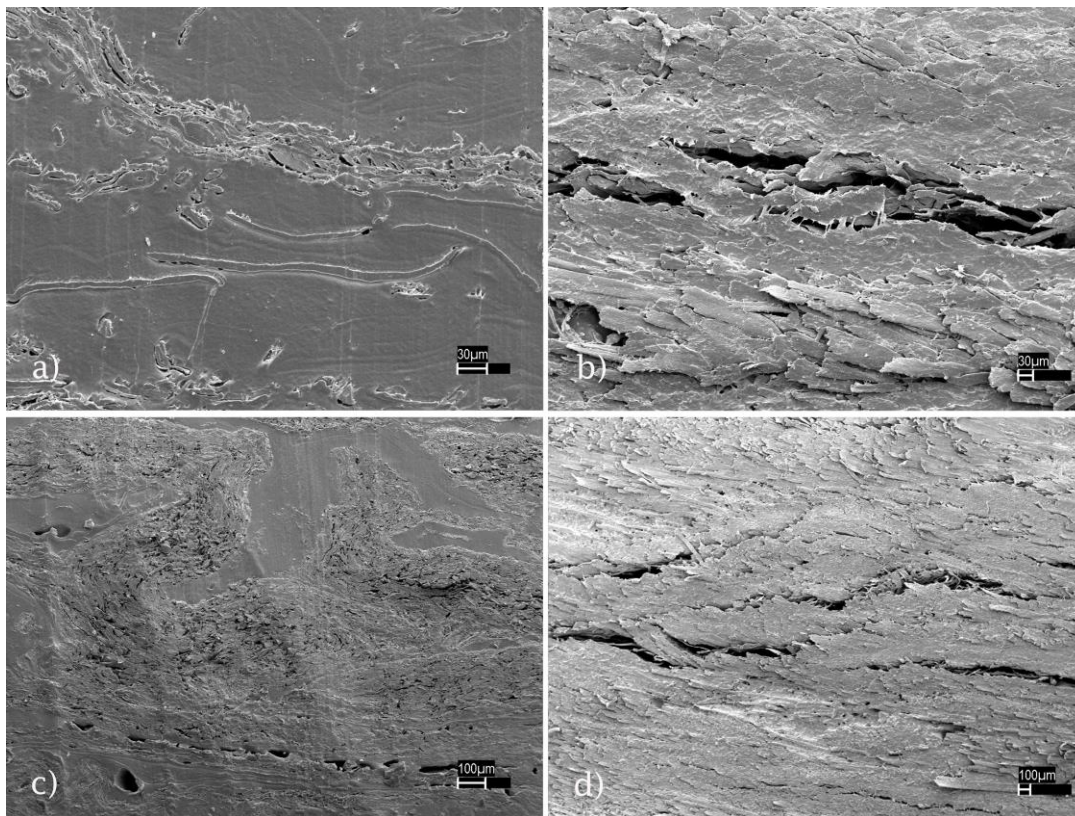
Fig. 4. Micrographs of dry panels (a and c) water soaked panels (b and d)

Biological Performance

Mass losses in the specimens from TP panels and wood specimens exposed to the two white and two brown rot fungi for 12 weeks based on the ASTM standard test method and one white and one brown rot fungi based on the JIS standard test method are given in Table 2. In the ASTM standard test method, the lowest mass losses were brought about by the white rot fungus, *C. versicolor*. The fungus causing the highest mass losses in TP panel specimens was the other white rot fungus, *I. lacteus*. When compared to mass losses that occurred in the SYP solid wood specimens, TP panel specimens were resistant to the test fungi in laboratory conditions. Similarly, considerable low mass losses were obtained in TP panel specimens when compared to sugi sapwood specimens exposed to the two test fungi based on the JIS K standard test method. Incorporation of boron into the matrix at both treatment levels lowered mass losses considerably. As expected, TP panel specimens showed increased resistance against the decay and mold fungi tested in the study. The composition of TP panel specimens may have a significant effect on biological performance properties. In general, the degree of fungal attack in test specimens is closely related to their moisture content during decay process, and mass losses are in general associated by increases in moisture content, since water is a key factor for colonization and decomposition of lignocellulosics by fungi (Guzman 2003; Lomelí-Ramírez *et al.* 2009). Lomelí-Ramírez *et al.* (2009) state that the proper moisture content to start decay by fungi is about 20%, and preferably this is 30%; however, Guzman (2003) recommends moisture levels higher than 30% for decay. Zabel and Morrell (1992) also state wood moisture content of 25 to 30% for fungal decay in wood specimens exposed to biological performance tests. Even though the polyethylene (PE)

material in TP panel specimens may have encapsulated paper particles in the matrix, the paper component may have reached moisture levels that are proper for fungal decay due to un-encapsulated paper particles near the specimen surface. Thus, we have seen highly severe fungal attack near the surfaces of the specimens exposed to *I. lacteus, T. palustris,* and *G. trabeum*. Wang and Morrell (2004) and Silva *et al*. (2001) state that degradation by fungi is likely to be limited to the surfaces of wood plastic composites due to encapsulation of wood fibers, and the inner parts of the composites remain generally unaffected until adequate moisture content is reached. Lomelí-Ramírez *et al*. (2009) also point out that fungal respiration results in metabolism of wood components and water formation during the decay process. Furthermore, hyphae are able to transport water from high humidity media into drier parts of specimens (Ammer 1964; Muller *et al.* 2001). In the present tests, TP panel specimens did not show complete protection against the fungi tested except for *C. versicolor* in both standard decay test methods, indicating that the specimens were able to absorb some amount of water during decay process even though they contained around 20% LDPE and 5% aluminum by weight in the matrix. In addition, the chemistry of LDPE part may play a role in decreased mass losses in the TP panel specimens. Silva Guzman (2003) states that the resistance of ordinary plastics to fungal degradation is due mainly to their low surface area and relative impermeability, and high molecular weight. Microorganisms in general tend to attack the ends of large molecules. Because the number of the ends is inversely proportional to the molecular weight, it would be necessary to break large molecules into very small fragments with a large surface area in order to make plastic degradable (Naghipour 1996). In addition, the fungi do not secrete enzymes capable of degrading such plastics. Thus, LDPE, polystyrene, and polyvinyl chloride (PVC) were not susceptible to fungal attack (Naghipour 1996).

Table 2. Mass Losses in Decay Resistance Tests - Based on the ASTM D2017-05 and JIS K 1571 Standard Methods

Samples	Method	Mass Losses (%)			
		C. versicolor	*I. lacteus*	*T. palustris*	*G. trabeum*
TP control	ASTM	0.06 B (0.14)	19.92 B (1.94)	11.19 B (2.05)	14.88 B (1.36)
TP 1% ZnB	ASTM	0.00 B (0.00)	0.57 C (0.52)	2.25 C (2.64)	0.20 C (0.27)
TP 10% ZnB	ASTM	0.00 B (0.00)	0.08 D (0.13)	0.24 D (0.20)	0.09 D (0.15)
Solid wood (SYP sapwood)	ASTM	27.02 A (2.12)	24.78 A (3.09)	47.56 A (3.67)	38.89 A (6.12)
TP control	JIS	1.03 B (0.23)	-	6.23 B (0.89)	-
TP 1% ZnB	JIS	0.00 C (0.00)	-	1.11 C (0.03)	-
TP 10% ZnB	JIS	0.00 C (0.00)	-	0.34 D (0.04)	-
Solid wood (sugi sapwood)	JIS	19.09 A (1.86)	-	46.86 A (7.74)	-
The same letters in each column in each method indicated that there was no statistical difference among the specimens according to Duncan's multiple range test. Numbers in parentheses are the standard deviations.					

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In both termite resistance tests based on the ASTM and JIS standards, similar results were obtained (Table 3). Mass losses by the termites in TP panel specimens were lower than those in solid sapwood specimens. No termite mortalities were observed in either of the specimen types. Boron addition into the matrix caused significant low mass losses and 100% termite mortalities in the tests based on the ASTM standards; however, in the JIS K standard methods, lower termite mortalities and higher mass losses were obtained. The termites in the JISK test method were able to deteriorate cellulose and LDPE parts in the matrix by the action of their mandibles while searching for food. The outer surfaces of the specimens showed small nibbles by the termites; however in the AWPA tests, no surface deterioration in the specimens was observed. Klyosov (2007) states that wood-plastic materials are commonly resistant to termites. Termites cannot get into the plastic matrix in composites easily and can only slightly trim wood fibers at the surface. This was also shown in our study, and mass losses in the TP panel specimens due to termite attack were highly lower than those in solid wood specimens.

Table 3. Mass Losses and Termite Mortalities in Termite Resistance Tests - Based on the ASTM D3345-08 and JIS K 1571 Standard Methods

Samples	Method	Termite species	Test duration	Average mass loss (%)	Average termite mortality (%)
TP control	ASTM	*Reticulitermes flavipes*	4 weeks	3.53 B (0.54)	0.00 B (0.00)
TP 1% ZnB	ASTM	*Reticulitermes flavipes*	4 weeks	0.89 C (0.35)	100.00 A (0.00)
TP 10% ZnB	ASTM	*Reticulitermes flavipes*	4 weeks	0.20 D (0.02)	100.00 A (0.00)
Solid wood (SYP sapwood)	ASTM	*Reticulitermes flavipes*	4 weeks	36.30 A (4.21)	0.00 B (0.00)
TP control	JIS	*Coptotermes formosanus*	3 weeks	7.57 B (0.55)	0.00 C (0.00)
TP 1% ZnB	JIS	*Coptotermes formosanus*	3 weeks	6.44 BC (0.97)	21.80 B (1.80)
TP 10% ZnB	JIS	*Coptotermes formosanus*	3 weeks	4.23 C (1.03)	85.17 A (5.84)
Solid wood (sugi sapwood)	JIS	*Coptotermes formosanus*	3 weeks	23.45 A (1.61)	0.00 C (0.00)
The same letters in each column in each method indicated that there was no statistical difference among the specimens according to Duncan's multiple range test. Numbers in parentheses are the standard deviations.					

TP specimens with and without boron addition were also resistant against mold growth on their surfaces when compared to solid wood specimens even after a 12-week-exposure period. No mold growth on TP panel specimens was observed in the tests even though the solid wood specimens were completely covered by the mold fungi at the end of tests (Table 4). These results suggested that TP panel specimens did not provide any suitable growth conditions for mold growth on their surfaces in terms of moisture content and food. Mold fungi need higher moisture content than 20 to 30% that is essential for decay fungi to grow on material surfaces and water movement through inside the composites with plastic takes a long time. In addition, the wood-discoloring molds and

staining fungi live generally on nutrients in the parenchyma cells such as sugar, starch, and protein.

Table 4. Average Mold Growth Ratings

Samples	Week 4	Week 8	Week 12
TP control	0 B	0 B	0.2 B
TP 1% ZnB	0 B	0 B	0 B
TP 10% ZnB	0 B	0 B	0 B
Solid wood (SYP sapwood)	3.4 A	3.6 A	4.4 A
The same letters in each column indicate that there was no statistical difference among the specimens according to Duncan's multiple range test. Specimens were visually rated on a scale of 0 to 5 with 0 indicating the specimen is completely free of mold growth and 5 indicating complete coverage. 0: no growth, 1: 20% coverage with mold fungi, 2: 40%, 3: 60%, 4: 80%, 5: 100%. Each value is the average of ten specimens.			

Fire Performance

According to the PHRR values, there was a significant difference between untreated and 10% ZnB treated TP panel specimens (Table 5). The PHRR values in treated TP panel specimens were about 11% lower than those of untreated TP panel specimens. When compared to ZnB treated specimens, the ignition time of commercial plywood was 40% longer than that of TP panel specimens. On the other hand, the PHRR values for the plywood specimens were observed as 21.7% higher than that in ZnB treated TP panel specimens. The highest mass loss ratio and PHRR values were recorded in untreated solid wood specimens followed by commercial plywood specimen, while the lowest PHRR, AHRR, and mass losses were observed in the ZnB treated specimens (Table 5).

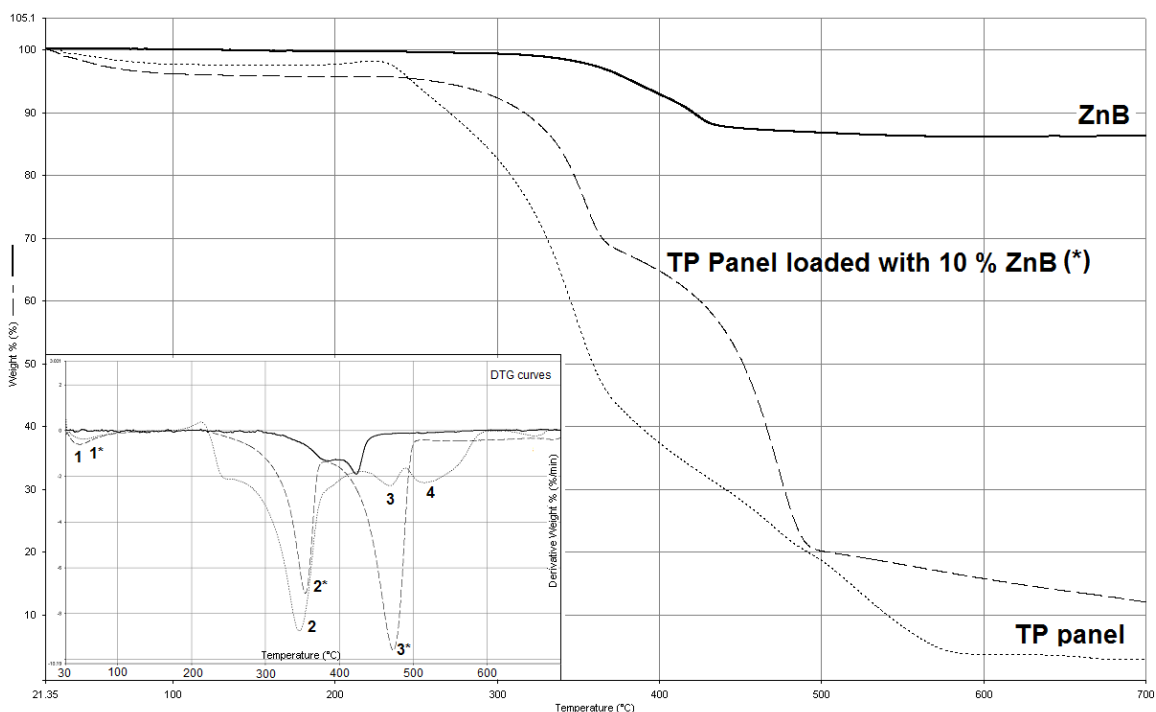**Table 5.** Mass Loss Calorimeter Test Results

Specimens	Time to ignition (s)	Mass loss (%)	Peak heat release rate (PHRR) (kW/m^2)	Average heat release rates (after ignition) (AHRR) (kW/m^2)	
				180 s	300 s
TP control	54	72.6	121.28	97	95
TP 10% ZnB	47	62.5	108.47	88	83
Plywood	78	73.6	138.99	88	98
Solid wood	66	84.8	227.00	110	114

Thermal Properties of Thermal Analyses

Figure 5 shows the thermal analysis curves and its derivative, which enable the determination of the mass changes as a function temperature of ZnB, TP panel, and 10% ZnB treated TP panel. The shape of the TG curves is interesting to explain the thermal degradation characteristic of TP panel and 10% ZnB treated TP panel. Considering each set of composites, the profile of TG curves exhibit entirely different degradation behavior due to the ZnB addition. The thermal degradation of ZnB occurred in two consequent steps within the 298.99 to 430.01 °C range, corresponding to a weight loss of about 14%. Reactions contributed to loss of 3 mole water and led to a thermally stable material as anhydrous ZnB.

It can be seen from the TG and DTG curves that degradation of TP panel specimens could be divided into 4 stages (Kanturk Figen *et al.* 2013). The first stage was the moisture evaporation (30.83 to 217.11 °C, weight loss=1.8%); the second stage was the major weight loss stage (217.11 to 425.13 °C, weight loss=65.4%), for which the weight loss was fast and a large amount of gas was produced; the third and fourth stages were consecutive reactions at the 425.13 to 599.61 °C temperature range with total weight loss 28.9%.

The TG/DTG curves of 10% ZnB treated TP panel can be divided into three weight loss stages: a first stage was the moisture evaporation (33.03 to 120.44 °C, weight loss=2.9%); the second stage appeared within the 225.45 to 380.14 °C temperature range with 28.4% weight loss; the last weight loss stage was the major mass stage with the 47.3% loss (380.77 to 502.13 °C). The residue values, corresponding non-degraded TP panel, were determined as 3.1% and 22.8% (not include remaining anhydrous ZnB) for TP panel and 10% ZnB treated TP panel, respectively. It can be easily seen that ZnB addition changed the degradation mechanism and led to improved degradation characteristics.

Fig. 5. TG and DTG curves of ZnB, TP panel and 10 % ZnB treated TP panel
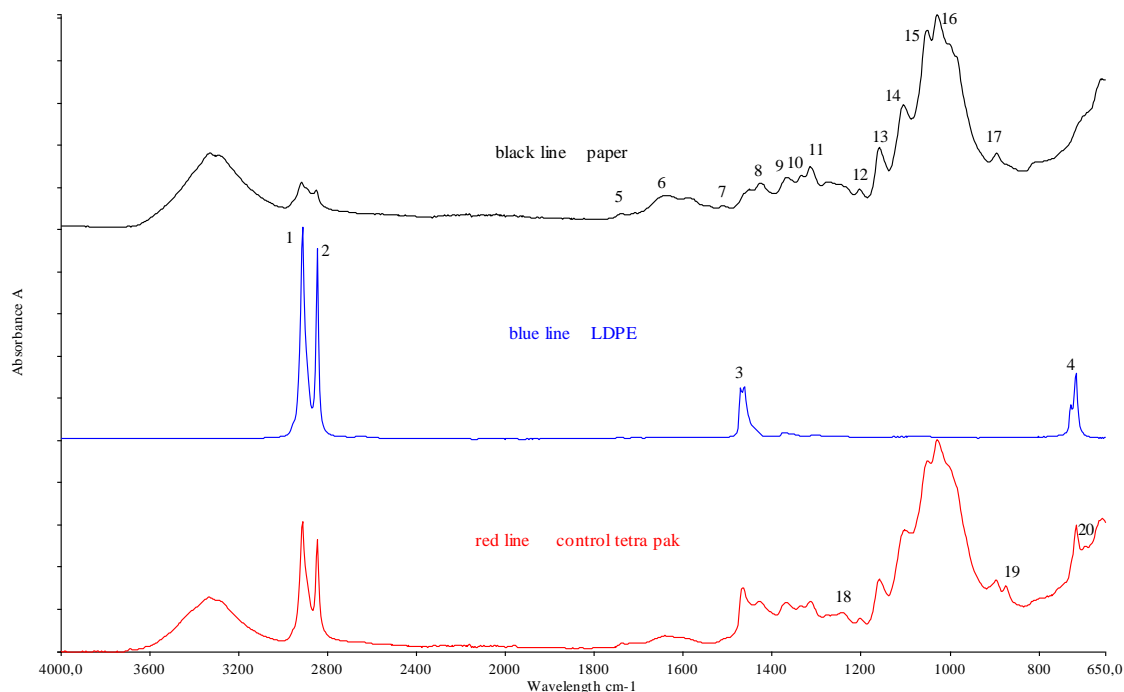
FT-IR ATR Evaluations

Evaluation of TP panels and layers

In order to observe the degradation behavior and accelerated weathering behavior of TP panels, the layers of TP beverage cartons were separated, and the spectra of the paper and LDPE layers were obtained as can be seen in Fig. 6. Control TP spectra represent TP panel specimens. In the figure, it is clearly apparent that control TP specimen spectrums reflect the characteristic bands both of the polymers except the absorption bands observed at 875 cm^{-1} (19) and 698 cm^{-1} (20) (Fig. 6.) These spectral

bands did not appear among the characteristic spectrum bands of the paper or LDPE. The source of these bands might be generated by heat during thermo-pressing in the course of sample preparation. Even though the paper was produced with the kraft process in which it is assumed that lignin is almost completely removed, polyoses in wood such as xylans and glucomannans remain in small quantities, and these components were observed both in paper and TP packaging material absorption bands. Actually chemical characterization of the paper confirmed that there were still lignin and polyoses, even though in small amounts (Table 1).

Fig. 6. IR spectrum of TP panel specimen, paper (cellulose) layer and LDPE layer

The peaks shown as numbers (1, 2, 3, 4) are coming from LDPE. There were strong absorption bands at 2906 and 2840 cm^{-1}, which were attributed to the alkane CH stretching vibrations of the methylene groups of LDPE (Naranjo *et al.* 2013; Xu *et al.* 2006). The next strong absorption band was observed at around 1465 cm^{-1} (3) attributed to the shearing activities of methyl and methylene and CH$_2$ bending deformation (Xu *et al.* 2006; Oldak *et al.* 2005). The band observed at 720 cm^{-1} (4) was referred to the C-H bending of characteristic LDPE crystal and rocking deformation (Xu *et al.* 2006; Oldak *et al.* 2005; Silverstein *et al.* 1974). On the other hand the band observed at 3336 cm^{-1} is contributed by the OH groups in the paper layer and also is present in TP panels (Harrington *et al.* 1964; Pandey and Pitman 2003). The absorption band at 1739 cm^{-1} (5) attributed C=O stretching in xylans was detected as a very small peak in the paper layer and TP panel specimen (Fig. 6) (Harrington *et al.* 1964; Pandey and Pitman 2003). Even though the source of the paper is not known, this peak was attributed to the acetyl group coming from xylan in hardwood and glucomannan in softwood (Fabiyi *et al.* 2011). The peak was seen in both specimens paper and packaging material at 1641 cm^{-1} (6) was attributed to H-O-H deformation vibration of absorbed water (Harrington *et al.* 1964). The tiny peak observed at 1504 cm^{-1} (7) is characteristic for lignin and it is more clearly seen in paper specimen than in control TP panel specimen (Faix 1991; Pandey and

Pitman 2003). The small peak identified at 1426 cm^{-1} (8) in both paper and TP panel specimen was attributed to C-H deformation in lignin and carbohydrates (Faix 1991; Pandey and Pitman 2003; Oldak *et al.* 2005). The bands at 1365 cm^{-1} (9) and 1332 cm^{-1} (10) can be attributed to the C-H deformation in cellulose, the hemicelluloses, and the C-H vibration in cellulose and C$_1$-O vibration in syringyl derivatives, respectively (Faix 1991; Pandey and Pitman 2003). The significant absorption bands detected at 1158 cm^{-1} (13), 1103 cm^{-1} (14), 1049 cm^{-1} (15), and 1029 cm^{-1} (16) were all attributed to the presence of cellulose and hemicelluloses (Harrington *et al.* 1964; Pandey and Pitman 2003; Oldak *et al.* 2005). The absorbance at 897 cm^{-1} (17) is a significant sign of C-H deformation in cellulose (Pandey and Pitman 2003; Oldak *et al.* 2005). On the other hand new peaks appear in TP panel specimen spectrum band at 1244 cm^{-1} (18) (attributed syringyl ring and C–O stretch in lignin and xylan), at 875 cm^{-1} (19) and 698 cm^{-1} (20), which were not detected either in paper or LDPE spectra (Pandey and Pitman 2003). The spectral band at 1244 cm^{-1} (18) was also detected in paper specimen at low intensity, but it became more remarkable in TP panel specimens than in the paper specimen. The peaks detected at 1312 cm^{-1} (11) and 1198 cm^{-1} (12) could not be attributed to any functional groups.

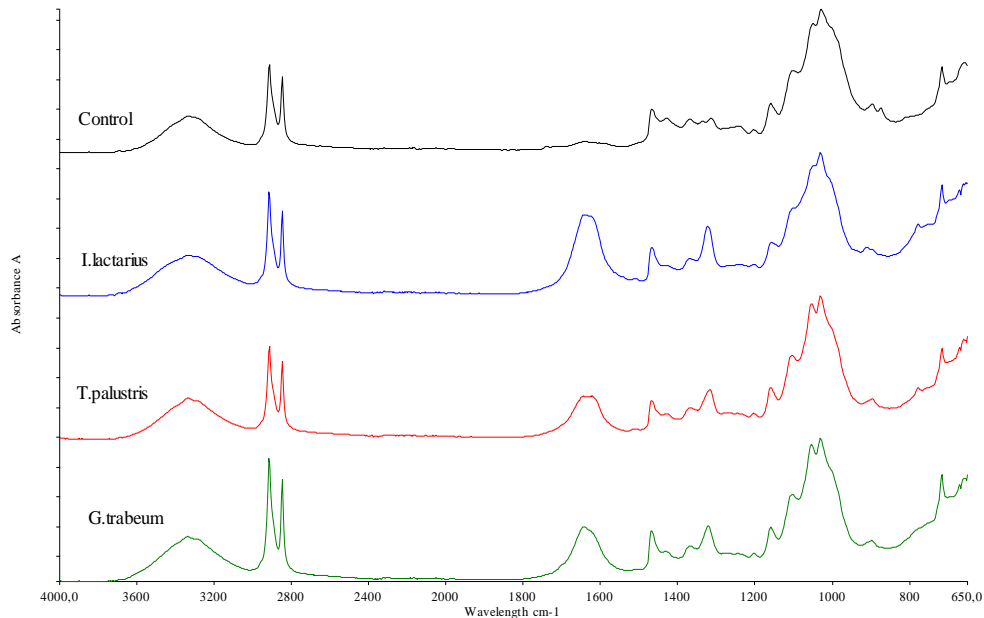*Evaluation of TP panel specimens after biological degradation*

There have been numerous studies on biological deterioration of wood-polymer composites. But the fungal decay mechanism in WPC is still not understood, since wood is composed of different types of polymers such as cellulose, hemicellulose, and lignin having different deterioration mechanisms when exposed to biological agents. This paper might serve to understand more about the decay mechanism of composites, since the natural polymer of the material is composed mostly of cellulose instead of other wood components. As indicated by Pandey *et al*. (2005), the preparation techniques and evaluation of mechanical properties of thermoplastics have been studied by many scientists, but composites with thermoplastics were not studied enough concerning their durability.

Since mass losses in TP control panel specimens by *C. versicolor* were considerably lower than those in the specimens decayed by the other fungi, this fungus was excluded, and FTIR evaluations were performed only for the fungi, *I. lacteus*, *T. palustris*, and *G. trabeum*.
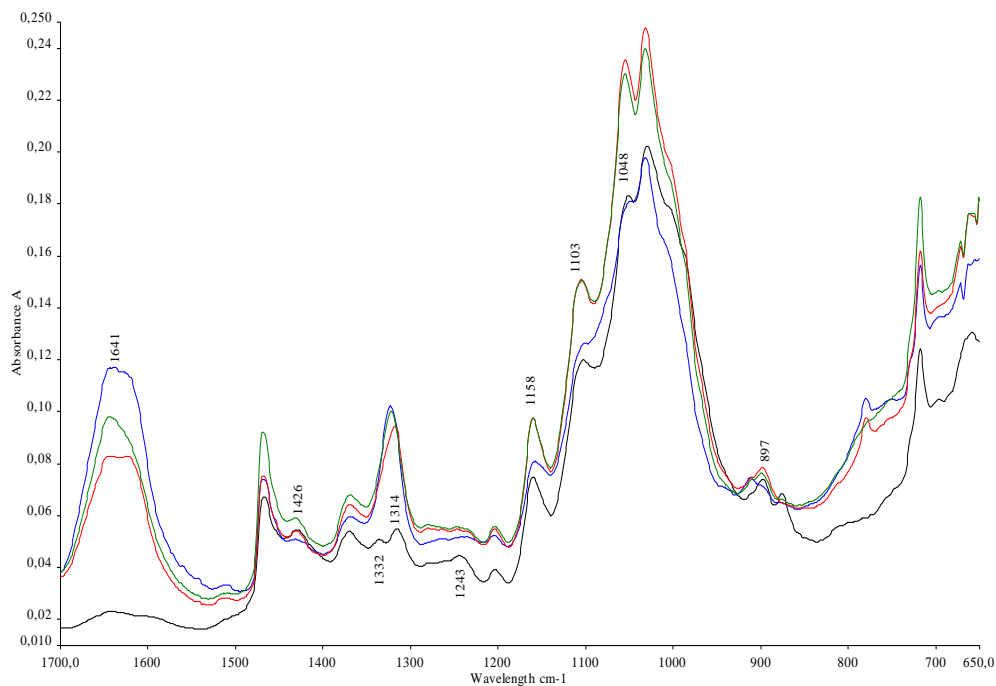
It is observed that there were not any changes in LDPE spectral features (2906 cm^{-1}, 2840 cm^{-1}, 1465 cm^{-1}, and 720 cm^{-1}) of panels, but some changes were detected in the IR spectra of the paper component of the panels as a result of both white and brown rot fungal activities (Figs. 7 and 8). The absorbance of the peak detected at 1641 cm^{-1}, which represents absorbed water, was significantly increased by the activity of all fungi. Even though a significant increase was observed at the band 1641 cm^{-1} in all specimens exposed to fungi, the white rot fungus *I. lacteus* caused a more significant increase in this band compared to brown rot fungi. It seems that *I. lacteus* exposure caused more moisture penetration in the specimen than the other fungi *G. trabeum* and *T. palustris*. As indicated by Lomeli-Ramirez *et al*. (2009), fungal activity causes water formation during a decay process. This finding is compatible with the mass loss result of *I. lacteus*, which exhibited the highest mass loss among the other fungi, as can be seen in Table 2. It is thought that the degradation of cellulose and polyoses might have caused new functional groups such as OH to emerge, and as a result the moisture content of the specimen increased. Even though the preference of wood components of white and brown rot fungi

might be different, it was detected that the peak at 1739 cm⁻¹, which represents C=O stretching in xylans, was completely lost in all the specimens that were exposed to fungal attack (Fig. 7). Besides, the band at 1332 cm⁻¹ attributed to the C-H vibration in cellulose and the band at 1244 cm⁻¹ C-O stretch in xylan were also completely lost in all specimens exposed to fungi attack. The absorbance at 1426 cm⁻¹ decreased and became a shoulder after *I. lacteus* exposure. On the other hand, the intensity of spectral bands at 1158 cm⁻¹ and 1103 cm⁻¹ attributed to the presence of cellulose and hemicellulose were significantly decreased in the specimen exposed to *I. lacteus* (Figs. 7 and 8).

Fig. 7. Comparison of control specimen and specimen decayed by the brown and white rot fungi

Fig. 8. Comparison of control specimen and specimens decayed by the brown and white rot fungi in the finger print region

Evaluation of TP panel specimens after accelerated weathering

It is important to know the weathering performance of panels for use outdoors. It is well known that long time duration of weathering causes many chemical changes in polymers, both natural and artificial. In this study the specimen consisted of two different polymers –cellulose (natural) and LDPE (plastic) - and their behavior due to weathering effect was expected to be different. Figure 9 shows the spectra obtained from ATR, which is known as a surface characterization and depth profiling technique (Oldak *et al.* 2005). However, because of the non-homogeneous characteristic of TP panel specimens, the surface investigation could be illusive even by ATR investigation. The surface investigation of samples subjected to accelerated weathering was performed by cutting the surface as a very thin layer and then grinding this thin layer under liquid nitrogen as mentioned previously.

FTIR-ATR spectra showed remarkable increases in the LDPE bands as seen in Fig. 9. The absorption bands around 2906 and 2840 cm^{-1}, which can be assigned to the alkane CH stretching vibrations of the methylene groups of LDPE, increased in intensity after accelerated aging (Fig. 9). The intensity of the absorption band at 1465 cm^{-1} attributed to the shearing activities of methyl and methylene and CH$_2$ bending deformation increased also after accelerated weathering compared to the control specimen. The similar alteration was observed in the band at 720 cm^{-1}, which can be attributed to the C-H bending of characteristic LDPE crystal and rocking deformation (Fig. 9). It can be clearly seen that after accelerated weathering there were significant increases in the noted spectral bands representing LDPE. Gulmine *et al.* (2003) pointed out that since the amorphous area is in LDPE higher than that in high density polyethylene (HDPE) and linear low density polyethylene (LLDPE), LDPE is more sensitive to oxidative degradation and cross-linking. The maximum heights of the bands were increased after accelerated weathering 233%, 105%, and 86% of the bands at 2906 cm^{-1}, 1465 cm^{-1}, and 720 cm^{-1}, respectively. On the other hand, the spectral bands representing natural polymer consisting of mostly of cellulose, and also very small amount of polyoses and lignin showed small changes due to accelerated weathering (Fig. 10).
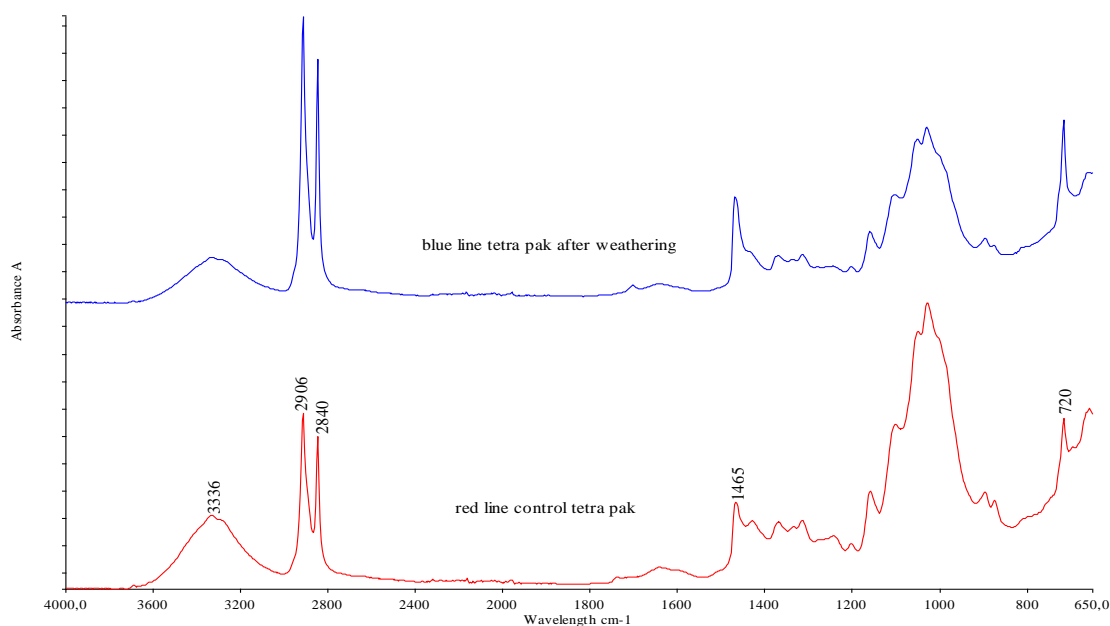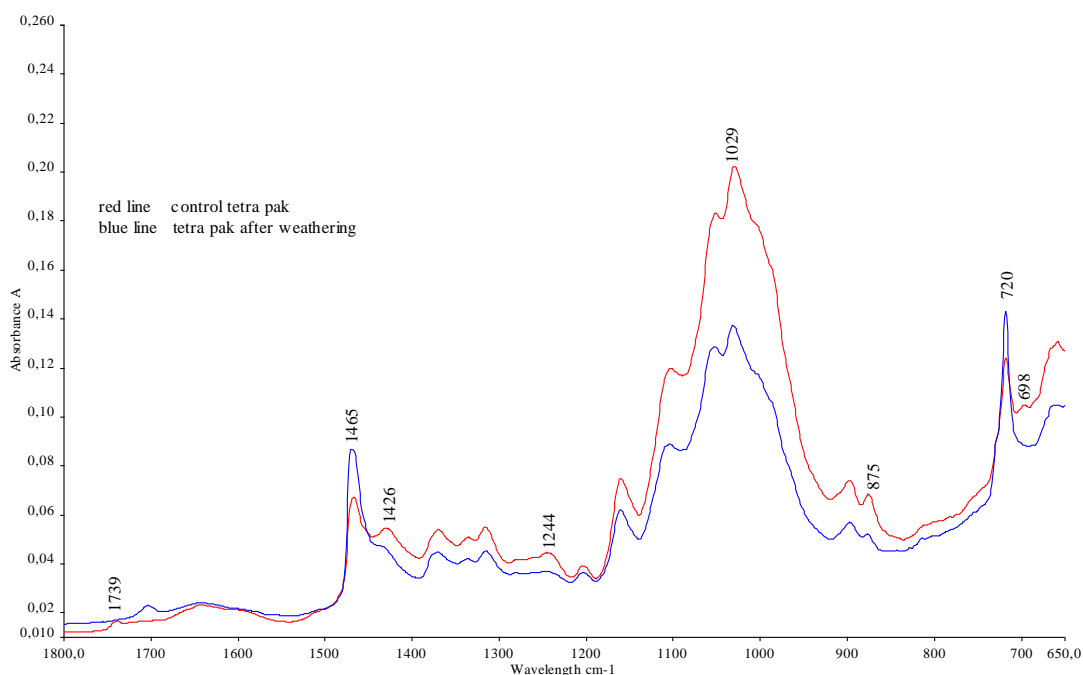

Fig. 9. Comparison of control TP specimen and specimen after accelerated weathering

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Cellulose is known for its high photo-stability compared to lignin in wood polymers, and the present observations confirm this. Changes observed in the absorption spectra in the 1800 to 650 cm^{-1} region were due to the impurities (lignin and polyoses) of the sample. The absorption band around 1739 cm^{-1}, which was attributed to C=O stretching in xylans, was lost, but a new and more intensive band appeared at 1701 cm^{-1} after accelerated weathering (Fig. 10).

Fig. 10. Comparison of control TP specimen and specimen after accelerated weathering in the finger print region

This band might have been in LDPE due to the weathering effect because of photo-oxidation of the plastics (La Mantia and Morreale 2008). On the other hand, no difference between control and accelerated weathered samples in absorption bands near 1641 cm^{-1}, which is related to the moisture content of the samples, was detected. The tiny band at 1504 cm^{-1} indicated that the presence of lignin was completely lost after accelerated weathering, as it was expected. It is known that lignin is the most sensitive of the natural polymers in wood to UV radiation. The remarkable increase observed at 1465 cm^{-1} is thought to be caused by the significant decrease in the next spectral band at 1426 cm^{-1} after accelerated weathering and it might be related also to the photo-degradation of LDPE (Figs. 9 and 10). On the other hand, cellulose was the main component of the panels, and it was represented by the spectral bands at 1158 cm^{-1}, 1049 cm^{-1}, 1029 cm^{-1}, and 897 cm^{-1}; based on these results, cellulose seemed the most resistant component of the composite material to the weathering process in this study. Except for the spectral band observed at 1029 cm^{-1}, the other spectrum bands' intensities did not show any change; however, a significant decrease was observed in the intensity of the spectrum band at 1029 cm^{-1} after the accelerated weathering experiment. Besides, the intensity of absorbance of the spectrum band at 875 cm^{-1} also decreased after accelerated weathering (Figs. 9, 10). The decrease of OH groups and the spectral band at 3336 cm^{-1}, which indicates OH groups in the specimens, is a characteristic of the weathering effect on

thermoplastics. When the intensities of the spectral bands at 3336 cm^{-1} in the control specimen and the specimen after weathering were compared, it is apparent that the intensity of this band in the specimen after weathering decreased, as shown in Fig. 9.

CONCLUSIONS

1. The study revealed that cellulose and LDPE components of the composite panels from waste TP packaging material showed different behavior in terms of resistance against fungal decay and photo-degradation. While photo-degradation occurred in the surfaces of the specimens only, the biological degradation appeared through the whole specimen.

2. Even though FT-IR ATR is known to be the best instrument to examine degradation characteristics of a given material, non-uniformity of composite materials constitutes a great drawback to obtain accurate results. This study confirmed the importance of obtaining homogenous samples before FT-IR evaluations by grinding non-uniform composite specimens under liquid nitrogen preferentially.

3. The mass losses in the specimens in fungal decay resistance tests and the decreases in the intensities of cellulose and polyoses bands in FT-IR spectra indicated that the highest decomposition occurred in the specimens exposed to the white rot fungus, *I. lacteus*. While the white rot fungus, *I. lacteus* caused 19.9% mass loss in test specimens, the brown rot fungi, *G. trabeum* and *T. palustris* resulted in 14.9% and 11.2% mass losses, respectively.

4. The accelerated weathering seemed to cause a decrease in the intensity of OH bands, but it also resulted in an increase in the intensities of LDPE bands' absorbance.

5. Furthermore, biological performance tests revealed that panel specimens without boron loading can be used in indoor applications where dry service conditions exist; however, 1% ZnB treatment will be beneficial to increase durability of such products when they are used outdoors. Considering fire resistance tests, more than 10% ZnB loadings will be necessary for desirable performance against fire. Indoor applications, however, seem more suitable usage for untreated panels from TP packaging materials due to their weak fungal resistance and water absorption properties.

6. Thermal degradation reactions of treated TP panel finished at the lower temperature (~500°C) compared with untreated (~700 °C). The main weight loss stage occurred at the higher temperatures with the treated TP panel. Thermal degradation reactions occurred within 3 stages while the untreated samples decomposed at 4 stages.

7. 10% ZnB treated TP panel lost 78.6% weight during the reactions, and this leads to the degradation of a smaller amount of TP compared with 96.9% for untreated. The char obtained after the untreated TP panel was 3.1%, and the treated TP panel specimen was 22.8% for treated TP panel. This means that a smaller amount of TP degraded during the thermal application compared with the untreated.

8. ZnB acts as a protective barrier against the degradation reactions while it is dehydrated during the heating in the TP panels. The bigger residue implies the ZnB is a good degradation resistor in the TP panels.

ACKNOWLEDGMENTS

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