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THE INFLUENCE OF A TREATMENT PROCESS ON THE REACTION TO WATER OF DURABLE AND WATER RESISTANT WOOD/PLASTIC COMPOSITES

In this study, the effect of the treatment process on the decay resistance and water absorption behaviour of wood flour/polypropylene composites was investigated. Composites based on ZB-pretreated, ZB-treated-during-manufacturing, and untreated wood flour, polypropylene were exposed to the decay by the white-rot fungus (Trametes versicolor) for 14 weeks. The long-term water absorptions of samples were evaluated by immersing them in water at room temperature for several weeks, and water diffusion coefficients (WDC) were also calculated by evaluating the water absorption isotherms. The morphology of the composites was characterized using scanning electron microscopy (SEM). The results showed that the composite made with ZB-treated wood flour exhibited the same water absorption as the control composite; while the ZB-pretreated wood flour, produced the highest improvements in water absorption performance. The water absorption of WPCs increased after exposure to white- rot fungus, where the water absorption of the decayed UT-WPC, ZB-T-WPC, and ZB-PT-WPC specimens increased by 41.4%, 43.25%, and 36.7%, respectively compared to the undecayed UT-WPC, ZB-T-WPC, and ZB-PT-WPC specimens. The undecayed ZB--treated sample and decayed ZB-pretreated composite exhibited the lowest and highest WDCs, respectively. The SEM micrographs revealed that the outer surface of the wood fibers were coated by some crystalline deposits of zinc borate. Much larger WDC values are observed from the ZB-pretreated and decayed composites when compared to the ZB-treated, control, and undecayed composites. The results of this study help to clarify the effect of T. versicolor on the dynamic moisture absorption process of WPCs impregnated with ZB.

Keywords: zinc borate, pretreatment, in-process, durability, long-term water absorption, wood- polypropylene composites

Introduction

The production and use of wood-plastic composites (WPCs) have been increased extensively in recent years as an engineering material for residential and industrial constructions such as decking, siding, roof tiles, and window frames

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[Li et al. 2014a], with attention to the deterioration caused by fungi, insects, and marine borers [Eaton and Hale 1993; Gardner et al. 2003; Kartal and Green 2003; Stark and Matuana 2006; Tascioglu et al. 2013].

The fungi decay attack was reported to reduce the mechanical properties of wetted WPCs [Li et al. 2014a]. On the other hand, glues used as well as the impregnated preservative for wood materials could be acting as a barrier or slowing down a fungal attack [Simonsen et al. 2004].

The increase in wood flour content of the composite matrix resulted in greater decay susceptibility and mass losses, because the major part of water absorption happened in the wood [Pendelton et al. 2002; Zabihzadeh et al. 2010; Kiani et al. 2011]. The decay of wood and wood based panels caused by fungi as well as other biodeteriorates can be prevented by several borate-based biocides such as zinc borate (ZB), disodium octaborate tetrahydrate, calcium/sodium borate, and boric acid [Laks 1999; Gardner et al. 2003; Tascioglu et al 2013].

Compared with other compounds, ZB has excellent characteristics for less water solubility, high processing temperature, common biocide, and low environmental toxicity and cost [Gardner et al. 2003; Simonsen et al. 2004; Tascioglu et al. 2013]. Two commercial WPC boards containing ZB, absorbed less moisture during exterior exposure [Gnatowski 2009]; also the addition of ZB (1%) to WPCs containing high-density polyethylene (HDPE) decreased moisture diffusion coefficient [Jahadi et al. 2010].

Although the water absorption of the WPCs produced from ZB-treated wood flour was investigated in previous studies [Laks 1999; Schultz and Nicholas 2003; Gnatowski 2009; Jahadi et al. 2010], the model for water diffusion of the WPCs was not extensively studied. In this study, the effect of *Trametes versicolor* on long-term water absorption of the WPC specimens produced from ZB-treated wood flour at different treating processes was investigated. In addition, the water diffusion coefficient of long term immersed WPC specimens depending on the treatment processes and morphological properties of composites based on wood flour and polypropylene was determined.

Materials and methods

The materials used in the production of zinc borate-pretreated wood flour/ polypropylene composites and their sources are presented in table 1. Maleic anhydride polypropylene (MAPP) as Epolene G-3003™ polymer was used as the coupling agent.

Poplar (*Populus deltoids*) wood was cut into small pieces and chopped using a laboratory electrical rotary mill to obtain poplar wood flour (PWF) with a mesh size of 60 (0.25 mm). ZB solution was prepared by dissolving 25 g of ZB in 250 ml ethanol and stirred for 30 min. with a magnetic stirrer. Then a 250 mL suspension of ZB-ethanol solvent was placed into a container with 2250 mL distilled water and stirred for 30 min. in a similar manner. The

concentration level of the ZB was designed to be 1%, based on the weight of oven-dried PWF.

Table 1. Materials used for the production of zinc borate-pretreated wood flour/ polypropylene composites

Material	Property	Source
Polypropylene (PP)	Melt flow index (MI) 10 g/10 min density 0.95 g/cm^3	Tabriz Petrochemical Company of Iran
Reinforcing filler	Fresh poplar wood (*Populus deltoides*)	Amol farms of Iran
Maleic anhydride polypropylene (MAPP)	8% acid anhydride molecular weight 103.500	Eastman Chemical Products, Inc.
Zinc borate) Borogard B (ZB)	–	U.S. Borax Inc., 26877 Tourney Road, Valencia, California 91355

Treatment process of poplar wood flour

Oven-dried PWF was added into the ZB solution and continuously stirred for 24 hours. The ZB-pretreated poplar wood flour (ZB-PTPWF) was filtered and stored at room temperature for 24 hours. After that, the ZB-PTPWF was oven--dried at 80°C until it reached a constant weight, subsequently; it was stored in sealed plastic bags to await blending with PP. Before and after impregnation, the PWF was kept in a drying oven at 103 ±2°C until a constant weight has been achieved. The ZB retention ratio (R, %) was calculated using the following equation (1),

$$R\ (\%) = \frac{M_b - M_a}{M_a} \times 100 \tag{1}$$

where M_a and M_b (g) denote the oven-dry weights of the PWF prior to and after impregnation with ZB treatment, respectively.

Composite preparation

Mixing of the materials was carried out with a Hake internal mixer (HBI System 90, USA) at 180°C and 60 RPM. Table 2 presents the blend design for the ZB--PTPWF composites. It should be noted here, that before preparation of the samples, the PWF was dried in an oven at 65 ±2°C for 24 hours. The production of composite boards was done according to our previously published work [Badritala et al. 2013].

Table 2. Formulation of WPC boards [Badritala et al. 2013]

Code	ZB concentration (wt %)	Wood flour (wt %)	Polypropylene (wt %)	MAPP (wt %)
UT-WPC	0	40	58	2
ZB-T-WPC	1 in powder	39	58	2
ZB-PT-WPC	1 in solution	40	58	2

UT-WPC: untreated wood flour/plastic composite; ZB-T-WPC: zinc borate treated wood flour in manufacturing process/plastic composite; ZB-PT-WPC: zinc borate pretreated wood flour/plastic composite.

Measurements

Water absorption (WA) was measured according to the ASTM D 570-98 [2010]. The water absorption of the decayed and undecayed WPC specimens with *T. versicolor* was determined after 2, 24, 72, 168, 240, 432, 624, 768, and 1000 hr immersion in distilled water (DW) at room temperature. Four specimens of each type of WPC were dried in an oven for 24 h at 105 ±2ºC. The dried specimens were weighed with a precision of 0.001 g and then they were placed in DW. At the end of the immersion periods, the specimens were removed from the DW and the surface water was wiped off using blotting paper. The weight of the specimens was measured at different time intervals during the long-time immersion. The measurements were terminated after the equilibrium weights of the specimens were reached. The mean percentages of WA versus time for each composite (decayed and undecayed) with each of the three formulations were recorded using the following equation (2);

$$WA\ (\%) = \frac{WA_t - WA_0}{WA_0} \times 100 \tag{2}$$

where WA_0 and WA_t denote the oven-dry weight; and weight after t time, respectively.

Results and discussion

Typical absorption curve of UT-WPC, ZB-T-WPC, and ZB-PT-WPC

For water absorption, figure 1 shows that the WA increased as the immersion time was increased. This trend continued up to where no more swelling was attained. The time taken to reach the saturation point was not the same for all the WPC formulations. Initial absorption curves for all the samples exhibited a rapid and a linear water uptake during the first 168 h and even 232 h, which were then followed by slower water uptake that approached an asymptotic equilibrium value.

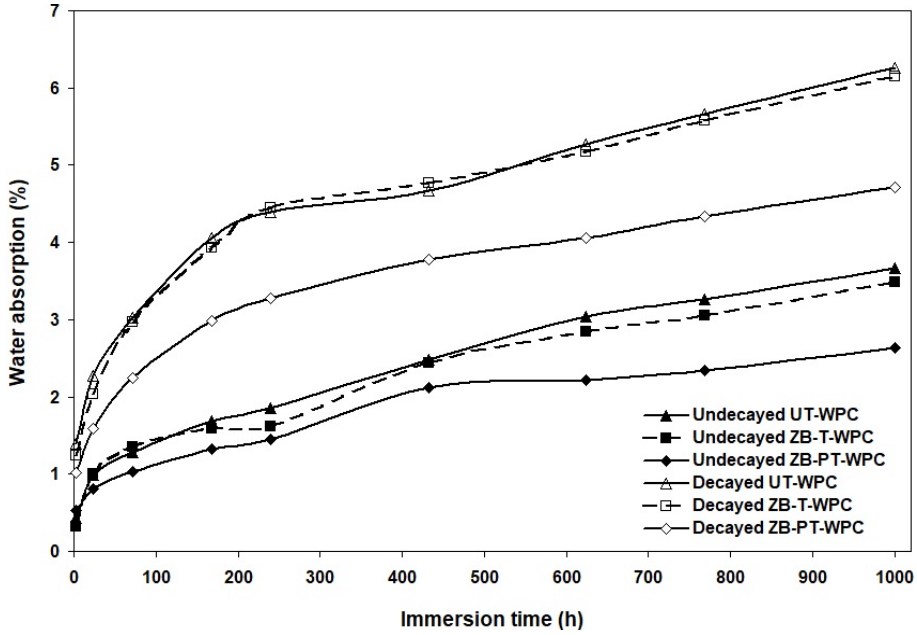

Fig. 1. Typical water absorption curves for selected composite formulations

The chemical characteristics (chemical compositions) of poplar wood such as organic (cellulose, lignin, extractives) and inorganic (ash) content could be affected by the water absorption of WPC where Safdari et al. [2011] were determined by its values that are presented in table 3.

Table 3. Chemical properties of poplar wood [Safdari et al. 2011]

Poplar	Chemical characteristics						Fiber sizes		
	cellulose (%)	ash (%)	lignin (%)	extractives			L (mm)	D (mm)	slenderness ratio (L/D)
				hot water (%)	cold water (%)	organic (%)			
Wood	46.66	2.06	22.33	6	5	4.1	0.30	0.05	5.3

L = mean length; D = mean diameter.

It is very well known that the PP matrix does not absorb any moisture, indicating that moisture is absorbed by the wood component in the composites [Bledzki et al. 2005].

As can be seen in figure 1, the specimens containing the ZB showed lower water absorption (WA) compared to the untreated specimens. The WA of the ZB--T-WPC and ZB-PT-WPC specimens decreased by 4.6% and 27.9%, respectively, compared to the UT-WPC specimens. This is mainly attributed to the less water solubility of zinc borate. Also, the ZB-PT-WPC specimens

actually had a lower average WA than the ZB-T-WPC ones. It seems that various possible reasons for less water absorption and moisture penetration into the ZB--PT-WPC specimens could be proposed, where the main mechanism is the diffusion of water molecules inside the micrographs between the polymer chains [Zabihzadeh et al. 2010; Hosseinihashemi et al. 2011]. It might alternatively be proposed that the precipitation of ZB in the cell lumens and the cell wall of particles, subsequently reduce in the WA. Like the preservative salts, fire retardant salts have also precipitated in the cell cavity and the cell wall [Winandy and Rowell 1984; Badritala et al. 2013]. One of them is a capillary transport of water into the gaps and flaws at the interface between fibers and polymer, and transport by microcracks in the matrix formed during the compounding process. Other possible mechanisms may involve diffusion behaviour (Fickian diffusion process) in the polymer matrix of WPCs. Another interesting result shown in fig. 1 was that the WA increased after exposure to *T. versicolor*, where the WA of the decayed UT-WPC, ZB-T-WPC, and ZB-PT--WPC specimens increased by 41.4%, 43.25%, and 36.7%, respectively, compared to the undecayed UT-WPC, ZB-T-WPC, and ZB-PT-WPC specimens.

Interface evaluation with scanning electron microscope

Figure 2 shows the SEM morphology of the undecayed and decayed samples from the untreated and treated samples.

As shown in figures 2a-b, the undecayed and the decayed untreated control sample had a relatively smooth fiber-plastic interface, where wood components were present on the fracture surface. There were few observed interfacial gaps or separations. In figures 2c-d, it is clear that numerous cracks appeared between the fibers and the matrix. This result is similar to that observed by Li et al. [2014a]. The SEM morphology analysis clearly revealed that the void content in the composite decreased with the addition of zinc borate (figs. 2c-d). Consequently, the filled cell lumens resulted in a slow penetration of water into the depth of the composites. Furthermore, the presence of fewer filled cavities helped to makes the WPC much less accessible for the moisture uptake due to decreasing of the void between the wood flour and polymer matrix (figs. 2e-f).

Mechanism of water absorption

The fitting of experimental data from the 1% ZB-treated WPC is shown in figure 3 as an example.

In general, there are three known mechanisms for water transport in polymer composites: Fickian diffusion, relaxation controlled and non-Fickian or anomalous. The dominant mechanism depends on factors such as the chemical structure of the polymer, the dimensions and morphology of the wood flour and the polymer-filler interfacial adhesion [Comyn 1985; Chiou and Paul 1986;

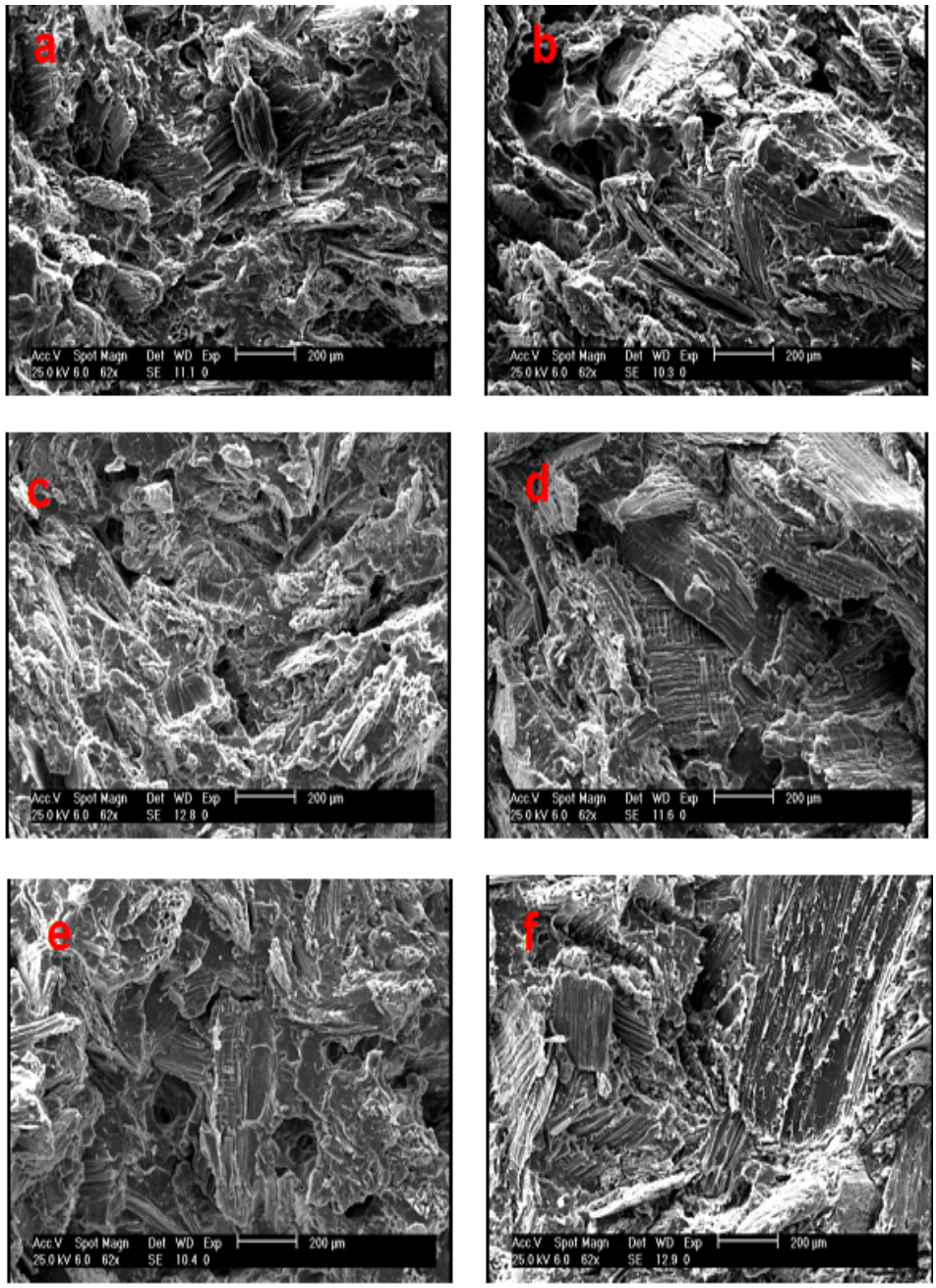

Fig. 2. SEM micrographs of the fracture surfaces in the composites under different treatments: undecayed and decayed UT-WPC (a-b); undecayed and decayed ZB-T--WPC: (c-d); and undecayed and decayed ZB-PT-WPC: (e-f), respectively

Bond and Smith 2006]. These cases can be distinguished theoretically by the shape of the sorption curve represented by the equation (3) [Adhikary et al. 2008; Ghasemi and Kord 2009]:

$$\mathrm{Log}\,\frac{Mt}{M\infty} = \log k + n\log(t) \tag{3}$$

where Mt is the water absorption at time t, $M\infty$ (*Mmax* or *Msat*) is the water absorption at the saturation point (maximum moisture content) and k and n are constants. The amount of the n is different for the following cases: in Fickian diffusion, $n = 0.5$; relaxation $n > 0.5$; and anomalous transport $0.5 < n < 1$.

The coefficients (n and k) were calculated from the slope and intercept of the log plot of $Mt/M\infty$ versus time, which could be drawn from experimental data. An example of the fitting of the experimental data for the heating temperature at different times is given in figure 3.

As shown in table 4, the n value of all composites varied from 0.24 to 0.36 for water absorption.

Thus, the observed n values deviated from the 0.5 value observed for a true Fickian diffusion process. Samples swelling during the absorption probably contributed to the deviation. The data seem to be consistent with previous studies [e.g. Adhikary et al. 2008]. Thus, the absorption process of WPC samples can only be described by Fick's law of diffusion as a first approximation; this is especially true for the water absorption during the immersion process. A higher value of k indicates that the composite requires a shorter time to reach equilibrium. The k values are significantly larger from the undecayed ZB-PT--WPC runs, when compared to those from the undecayed WPC. Thus, the WA from the decayed samples occurred at a much faster rate during absorption under the given fungal exposure conditions.

Maximum WA and diffusion constant

The diffusion constant is the most important parameter of the Fick's model and shows the ability of water molecules to penetrate inside the composite structures. The maximum WA and water diffusion coefficient (WDC) values of all composites from absorption are given in table 4.

The mean maximum WA of the undecayed and decayed control composite samples after water absorption was 3.66% and 6.25%, respectively. The WA varied from 2.64 to 6.15% for treated composites after WA. The use of ZB led to slightly lower WA values in some formulation (e.g. ZB-T-WPC and ZB-PT--WPC); however, there was a consistent effect of treatment on the maximum WA values after water absorption. Composites with 1% ZB in the solution had the lowest WA of 2.64%. Overall, the effect of ZB treatment on the decrease of WA of WPCs from water-soaked at the used levels seems to be small when compared to the control samples.

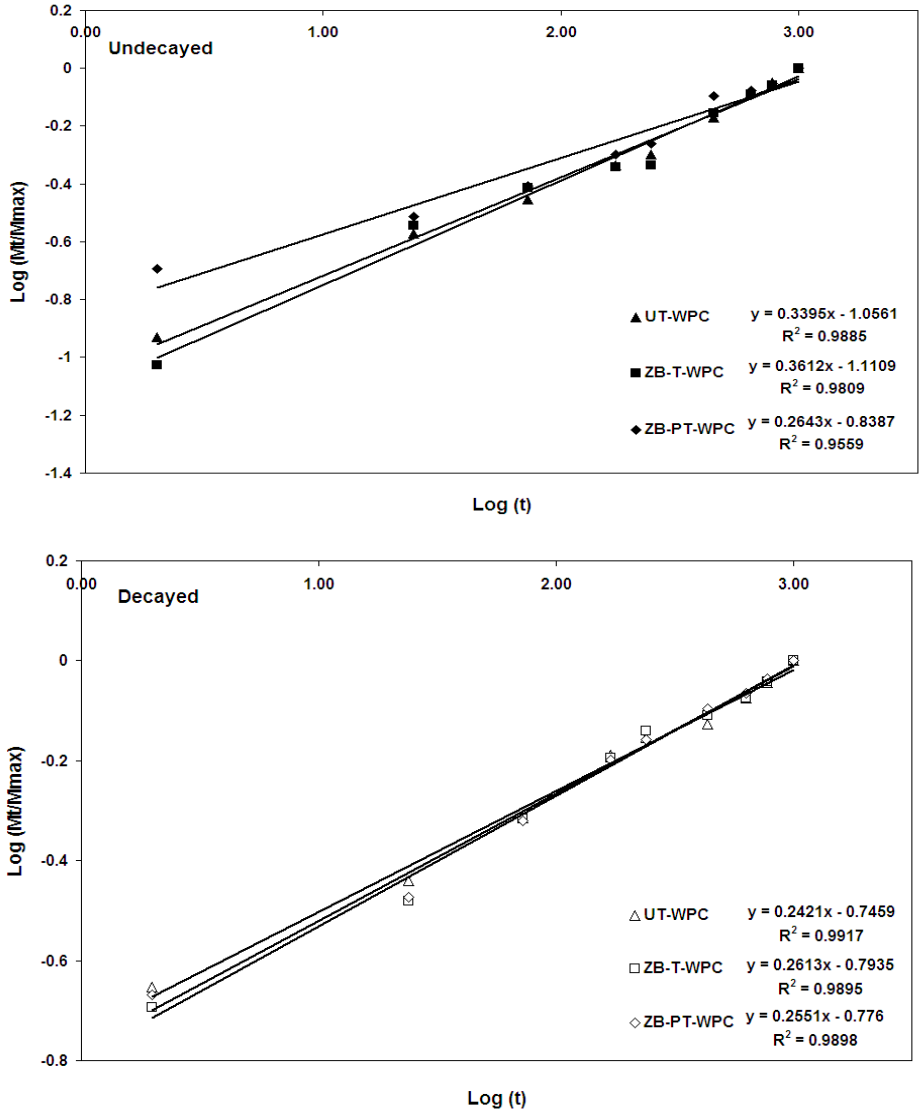

Fig. 3. Fitting of moisture diffusion data for WPC samples with 1% zinc borate

At the early stages and short times (typically $Mt/M\infty \leq 0.5$), the diffusion process is presented as equation (4) [Nadali et al. 2010]:

$$\frac{Mt}{M\infty} = \frac{4t^{1/2}}{(Dt/\pi L^2)^{1/2}} \tag{4}$$

where L is the thickness of the specimen and D is the diffusion coefficient.

Table 4. Weight loss, water diffusion, maximum water absorption, *n* and *k* coefficient for all composites

Sample type	Code	Weight loss (%)	Maximum water absorption (%)	*n*	*K* (hr^2)	Water diffusion coefficient (m^2/s)
Undecayed	UT-WPC	–	3.66	0.34	0.09	7.54×10^{-13}
	ZB-T-WPC	–	3.49	0.36	0.08	8.25×10^{-13}
	ZB-PT-WPC	–	2.64	0.26	0.14	4.90×10^{-13}
Decayed	UT-WPC	1.41	6.25	0.24	0.18	1.23×10^{-12}
	ZB-T-WPC	0.27	6.15	0.26	0.16	1.33×10^{-12}
	ZB-PT-WPC	0.24	4.17	0.25	0.17	1.19×10^{-12}

The water sorption kinetics of the WPCs was studied through the diffusion constants (*k* and *n*) obtained from the data given in table 4. It was observed that the value of *n* is close to 0.5 for all of the composites. This confirms that the Fickian diffusion can be used to adequately describe water transport in the composites, which is consistent with previous studies [Marcovich et al. 2006; Wang et al. 2006]. The higher values of *n* and *k* indicate that the composite needs a shorter time to attain equilibrium water absorption. The value of *k* of the specimens increased with increasing polymer matrix. The value of *k* for the decayed UT-WPCs was higher than that of other WPCs, which could be due to the deterioration of wood cell wall chemical components, namely exposed to the white-rot fungus, which resulted in higher moisture absorption initially. The *k* tends to be lower for the WPCs produced from ZB-PT and ZB-T-wood flour, confirming the reduction in WA. However, the decayed UT-WPCs had a higher *k* value than the WPCs produced with ZB treated wood particles, and the reason for this was not clear. The water diffusion coefficient for the WPCs was plotted as $Mt/M\infty$ against $t^{0.5} \times L^{-1}$ (fig. 4).

The WDCs were obtained from the slopes of the linear part of the plots using the less-squares method. The results showed the WDC of the WPCs decreased with the addition of ZB to the composites. The control WPCs showed the lowest WDCs. The WDCs value of undecayed and decayed composites was 7.54×10^{-13} and 1.23×10^{-12} m^2/s, respectively. The WDCs values from treated composites varied from 8.25×10^{-13} to 1.19×10^{-12} m^2/s. Among these three formulations, the composite with 1% ZB in the solution had the lowest WDC value (4.90×10^{-13} m^2/s in undecayed and 1.19×10^{-12} m^2/s in decayed), while the composites with 1% ZB in the powder had the highest value (8.25×10^{-13} m^2/s and 1.33×10^{-12} m^2/s). The insoluble ZB particles in the composite could lead to additional

micro-gaps between the fiber and the matrix, which in-turn reduced the bonding strength and helped speed up the water diffusion process [Li et al. 2014b].

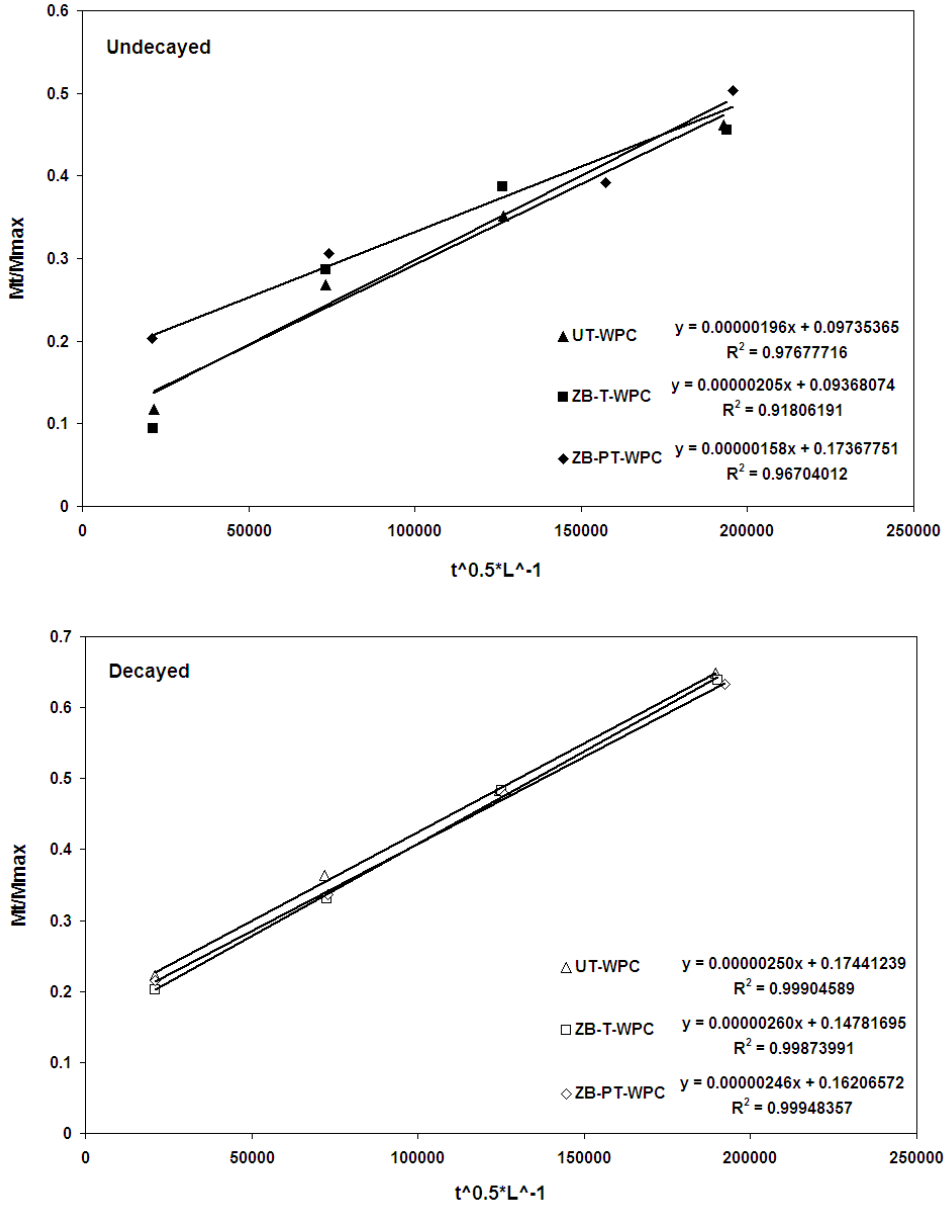

Fig. 4. Water diffusion coefficient data for WPC samples with 1% zinc borate

Conclusions

The long-term water absorption of zinc borate-pretreated wood flour/ polypropylene composites was investigated as affected by white-rot fungus (*Trametes versicolor*) after 14 weeks of incubation. The moisture content of the WPCs increased as immersion time increased. The water absorption curves linear, up to the first 168 h and then asymptotically approached the saturation limit. According to the data in table 4, the samples exposed to fungus (decayed) took much more water.

The initial water absorption curves were also approximately linear; afterwards, the absorption process slowed down until reaching the equilibrium state. The mechanism of water absorption of all the composites deviated from the Fickian diffusion process.

The water absorption of the WPCs increased after exposure to *T. versicolor*, where the water absorption of the decayed UT-WPC, ZB-T-WPC, and ZB-PT--WPC specimens increased by 41.4%, 43.25%, and 36.7%, respectively, compared to the undecayed UT-WPC, ZB-T-WPC, and ZB-PT-WPC specimens.

The undecayed ZB-treated sample and decayed ZB-pretreated composite exhibited the lowest and highest WDCs, respectively. Much larger diffusion coefficient values are observed from the water absorption of ZB-PT-WPC samples *versus* the water absorption of ZB-T-WPC samples.

The measured maximum WA and calculated diffusion coefficients varied with sample formulation, which indicated that the ZB-pretreatment used in the current study did positively affect the water absorption behavior of WPCs.

The results of this study help to clarify the effect of *T. versicolor* on the dynamic moisture absorption process of WPCs impregnated with ZB.

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List of standards

ASTM D 570-98:2010 Standard Test Method for Water Absorption of Plastics, American Society for Testing and Materials (ASTM), West Conshohocken, PA, USA

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