Effects of Heat Treatment, Zn_3BO_6 Addition, and their Combination on the Mechanical Properties and Water Absorption Behavior of Beech Wood/Polypropylene Composites

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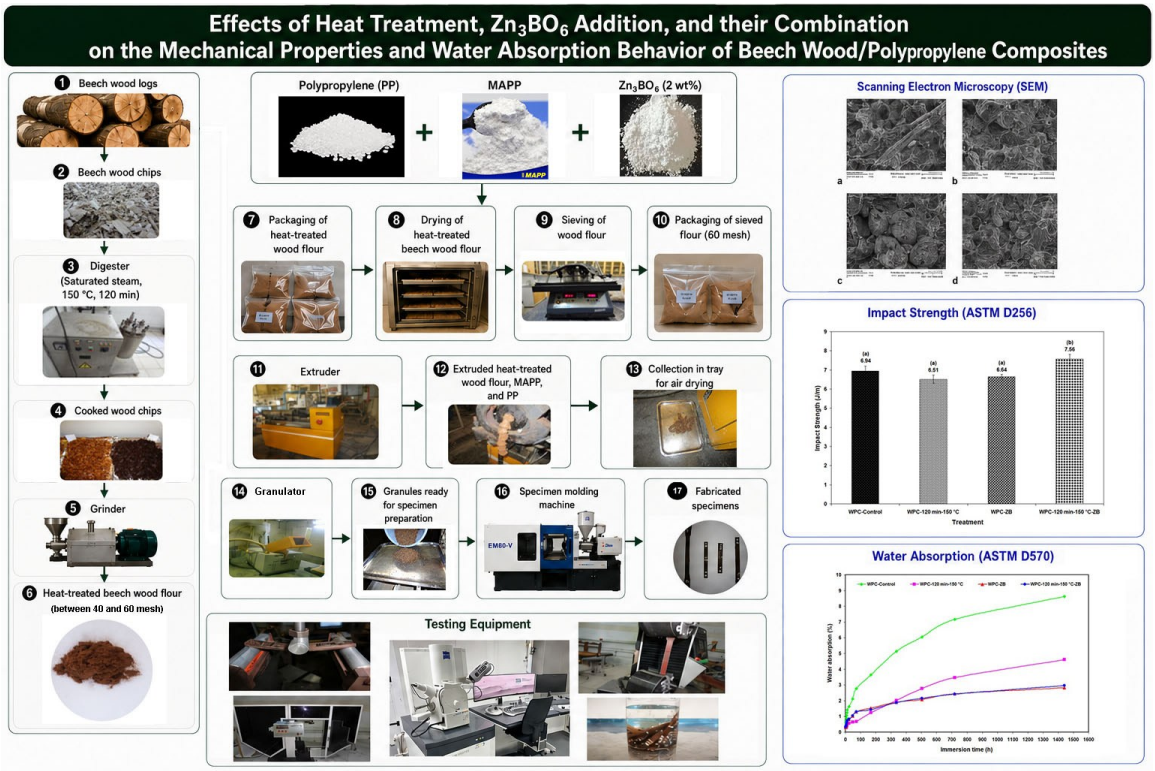
GRAPHICAL ABSTRACT



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Individual and combined effects of thermal treatment and zinc borate (ZB) addition were studied relative to the mechanical properties and water resistance of oriental beech (*Fagus orientalis* Lipsky) wood-plastic composites (WPCs). Wood flour was thermally treated at 150 °C for 120 min under saturated steam before composite manufacturing. Flexural, tensile, impact, and water resistance properties were evaluated, and the data were analyzed using two-way ANOVA ($p < 0.05$). Thermal treatment increased the flexural strength from 57.2 MPa to 75.0 MPa, while the combined treatment produced the highest impact strength (7.56 J/m). Zinc borate-containing composites exhibited lower maximum water absorption (2.8 to 3.0%) than the control (8.6%), despite showing higher moisture diffusion coefficients. This indicated that the diffusion rate and equilibrium moisture uptake were driven by different mechanisms. SEM observations revealed that the combined treatment produced a more cohesive fracture morphology, although these changes were not accompanied by improved flexural or tensile strength. Overall, thermal treatment and zinc borate affected the performance characteristics through distinct mechanisms, providing an effective approach for improving the moisture resistance and durability of wood-plastic composites.

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Keywords: Wood-plastic composites; Thermal treatment; Zinc borate; Water absorption; Diffusion modeling; Mechanical properties; Modeling

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INTRODUCTION

Wood-plastic composites (WPCs) have gained increasing attention as sustainable engineering materials due to their favorable mechanical properties, low maintenance requirements, and recyclability, making them suitable for outdoor and structural applications (Ashori 2008). These composites typically consist of thermoplastic polymers reinforced with lignocellulosic fillers such as wood flour. However, the inherently hydrophilic nature of wood remains a major limitation, as it leads to excessive water absorption, dimensional instability, and deterioration of mechanical properties under humid or wet conditions (Clemons 2002; Hosseinihashemi *et al*. 2016; Hung *et al*. 2017).

The hygroscopic behavior of wood originates from the abundance of hydroxyl groups in cellulose and hemicellulose, which readily form hydrogen bonds with water molecules (Stark and Rowlands 2003; Hill 2006). As a result, WPCs manufactured with untreated wood flour often exhibit swelling, microcracking, and debonding during long-term moisture exposure, ultimately compromising durability (Esteves *et al*. 2007; Toubal *et al*. 2016). Therefore, improving the moisture resistance of WPCs remains a critical challenge for expanding their long-term outdoor applications. Recent review articles have further emphasized that improving moisture durability while maintaining mechanical performance remains one of the principal challenges in the development of structural WPCs (Wang *et al*. 2021; Jian *et al*. 2022).

Thermal treatment has been widely investigated as an effective method to reduce the hygroscopicity of lignocellulosic materials. Controlled heat exposure degrades hemicelluloses and reduces accessible hydroxyl groups, thereby lowering moisture uptake and improving dimensional stability (Ayrilmis *et al*. 2011; Kaboorani and Englund 2011; Hosseinihashemi *et al*. 2022). Previous studies have demonstrated that thermal treatment of beech wood within the temperature range of 160 to 200 °C significantly reduces water absorption in WPCs. However, such severe conditions may also cause excessive degradation of wood polymers and negatively affect mechanical strength (Follrich *et al*. 2006; Fletes and Rodrigue 2021; Broda *et al*. 2024). Consequently, moderate thermal treatment temperatures, such as 150 °C, have been proposed as a compromise between moisture resistance improvement and preservation of mechanical integrity (Källbom *et al*. 2020; Martha *et al*. 2024).

Although many previous studies have focused on treatment temperatures between 160 and 200 °C, several investigations have shown that thermal modification at 150 °C is sufficient to initiate partial degradation of hemicelluloses and reduce hydroxyl group accessibility (Arwinfar *et al*. 2016; Hosseinihashemi *et al*. 2016, 2022). This moderate temperature improves dimensional stability while minimizing the loss of mechanical properties observed at higher treatment temperatures.

In addition to thermal modification, zinc borate (ZB) is commonly incorporated into WPC formulations as a multifunctional additive, primarily for its flame-retardant and biocidal properties. Several studies have also reported that ZB can influence moisture behavior by partially blocking capillaries and interfacial voids, leading to reduced water uptake (Ghasemi and Kord 2009; Yang *et al*. 2017; Hosseinihashemi and Badritala 2017; Mrad *et al*. 2018). Most published studies have evaluated ZB loadings between approximately 1 and 5 wt%, with concentrations around 1 to 2 wt% generally providing improved moisture resistance while minimizing adverse effects on mechanical properties; higher loadings may promote particle agglomeration and reduce interfacial compatibility (Ayrilmis 2013; Wang *et al*. 2020; Cárdenas Oscanoa *et al*. 2021). Accordingly, the present study employed 2 wt% ZB, which falls within the range commonly reported as effective in previous investigations. However, the reported influence of ZB on mechanical properties remains inconsistent, with some studies showing slight reinforcement and others indicating neutral or even negative effects, depending on formulation and processing conditions (Faure *et al*. 2019; Wang *et al*. 2020). Oriental beech (*Fagus orientalis* L.) was selected as the lignocellulosic reinforcement because it is one of the most abundant hardwood species in the region, possesses favorable mechanical characteristics, and has been extensively used in previous WPC studies, thereby facilitating meaningful comparison with earlier research.

Although several previous studies have suggested beneficial combined effects of thermal modification and chemical additives, the interaction between these treatments has not been systematically evaluated, and evidence for true synergistic effects remains limited. Furthermore, it should be noted that the incorporation of the ZB typically requires a slight reduction in wood flour content to maintain processability and constant compatibilizer levels. This formulation adjustment may act as a confounding factor when interpreting changes in moisture resistance and mechanical performance and therefore must be carefully considered in comparative analyses.

Accordingly, this study aimed to evaluate the individual and combined effects of moderate thermal treatment (150 °C for 120 min) and zinc borate addition on the mechanical properties, moisture resistance, and moisture diffusion behavior of polypropylene-based WPCs reinforced with oriental beech wood flour. Particular emphasis was placed on determining whether the interaction between these treatments is property-dependent rather than universally synergistic.

EXPERIMENTAL

Materials

The polymer matrix consisted of V30S polypropylene (PP) with a melt flow index of 16 g/10 min and a density of 0.87 g/cm³, supplied by Marun Petrochemical Co. (Mahshahr, Iran). An Oriental beech (*Fagus orientalis* L.) log with a diameter of 200 to 250 mm was supplied from a local market. The coupling agent, maleic anhydride grafted polypropylene (MAPP), characterized by a melt flow index of 64 g/10 min and density of 0.91 g/cm³ was provided by Kimia Javid Sepahan Co. (Tehran, Iran). A constant level of 3 wt% MAPP was added to ensure consistent interfacial adhesion across all formulations. Zinc borate (ZB, ≥99% purity) with a density of 2.8 g/cm^3 was used as the inorganic additive. The material supplied by Merck (Germany).

Thermal Treatment of Wood Chips

Beech logs were first processed into chips using a drum-type chipper. The formulations used for preparing the wood-plastic composites are presented in Table 1. The wood chips were air-dried at ambient temperature for 24 h before undergoing heat treatment. After cooling in a desiccator, thermal treatment was carried out in a laboratory steam digester using saturated steam at 150 °C for 120 min. Under saturated steam conditions, the corresponding equilibrium pressure was approximately between 70 and 80 bar.

Table 1. Compositions of the Evaluated WPC Formulations

WPC Code	Beech Wood Flour (wt%)	Polypropylene (PP) (wt%)	MAPP* (wt%)	Zinc Borate (ZB) (wt%)
WPC-control	50	47	3	0
WPC-120 min-150 ºC	50	47	3	0
WPC-ZB	47	48	3	2
WPC-120 min-150 ºC-ZB	47	48	3	2

*MAPP = maleic anhydride grafted polypropylene

Then, the treated wood chips were ground into flour. The particle size range of the wood flour was controlled by sieving to minimize variability in composite performance. Beech wood flour passing through a 40-mesh sieve was oven-dried at 103 ± 2 °C for 24 h to achieve a moisture content between 0 and 1%. Then, the polypropylene, beech wood flour and coupling agent were weighed accurately and packaged according to the formulations outlined in Table 1.

For the ZB-treated formulation, the ZB powder (2 wt%) was directly added to the blend during compounding with and without thermal treatment of the wood flour. The thermal treatment temperature of 150 °C was selected based on previous studies reporting that moderate treatment conditions effectively improve some mechanical properties, moisture resistance, and dimensional stability of thermoplastic composites produced with thermally treated beech wood, while minimizing excessive thermal degradation of wood components (Arwinfar *et al*. 2016; Hosseinihashemi *et al*. 2016; Hosseinihashemi *et al*. 2022).

Preparation and Testing of WPCs

The raw materials were dry-blended and then compounded using a counter-rotating, intermeshing, twin-screw extruder (Model T20; Dr Collin GmbH, Germany), with barrel temperatures set at 180 °C across six zones, from the feeding section to the die. The screw speed was maintained at 60 rpm for a duration of 14 min. The resulting molten mixture was cooled to ambient temperature and then ground into granules using a laboratory mill (Wieser, WGLS 200/200 Model, Germany). These granules were subsequently dried at 105 °C for 4 h. Test specimens were fabricated using an injection molding machine (Model EM80, Aslanian Co., Iran) operating at temperatures between 160 and 180 °C. Each molding cycle produced a full set of specimens for various tests. Prior to testing, the WPC samples were conditioned at 23 °C and 50% relative humidity for a minimum of 40 h in accordance with ASTM D618-99 (1999). Water absorption measurements were conducted following the ASTM D570-098 (2010) standard.The sizes of control WPCs samples are shown in Fig. 1.

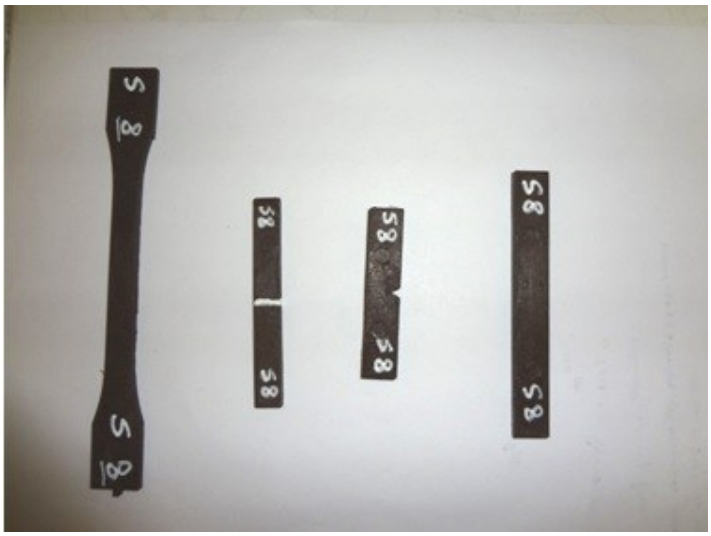

Fig. 1. The sizes of WPCs control samples (HosseiniHashemi and Arwinfar 2023)

Mechanical Testing

Flexural properties were determined according to ASTM D790, tensile properties according to ASTM D638, and impact strength according to ASTM D256. All mechanical tests were performed using the prepared injection-molded specimens under the standard test conditions specified by the respective ASTM methods. At least three replicate specimens were tested for each formulation.

Morphological Analysis of WPCs

The microstructure of the wood-plastic composites was examined using scanning electron microscopy (SEM, Model LEO 440i, Oxford, UK) operated at an accelerating voltage of 15 kV. Prior to imaging, the samples were rapidly frozen in liquid nitrogen and fractured to preserve a clean and undisturbed internal structure. To enhance electrical conductivity during SEM observation, the fractured surfaces were coated with a thin layer of gold.

Water Absorption and Thickness Swelling Tests

Water absorption measurements were recorded according to the ASTM D570-098 (2010) standard. The WPC specimens with nominal dimensions of 5 mm × 11 mm × 80 mm, were immersed in distilled water at room temperature. The absorption was recorded at multiple intervals: 2, 4, 6, 8, 10, 12, 24, 48, 72, 168, 336, 504, 720, and 1440 h. For each type of composite, three samples were oven-dried at 105 ± 2 °C for 24 h before testing. After drying, the specimens were weighed with a precision of 0.001 g and then submerged in distilled water. At each time point, the samples were removed, surface moisture was gently wiped off using blotting paper, and their weights were measured. This process continued until the specimens reached a stable, equilibrium weight.

Water absorption percentage at time t, WA(t), was calculated by Eq. 1,

$$\text{WA}\ (t) = [\frac{W(t) - W(o)}{W(o)}] \times 100 \quad (1)$$

where WA(t), $W(o)$, and $W(t)$ depict the water absorption at time t, the initial weight of specimens, and the weight of specimens at time t, respectively.

Similarly, thickness swelling at time t, TS (t), was determined by Eq. 2,

$$\text{TS}\ (t) = [\frac{T(t) - T(o)}{T(o)}] \times 100 \quad (2)$$

where TS(t), $T\ (o)$, and $T\ (t)$ depict the thickness swelling at time t, the initial thickness of specimens, and the thickness of specimens at time t, respectively.

Further analysis focused on modeling the swelling behavior of the composites. Swelling rate parameters were extracted by fitting model predictions to the experimental data. Shi and Gardner (2006) proposed a model to quantify thickness swelling in WPCs, describing the hygroscopic swelling process in wood-based composites. The model introduces a swelling rate constant K_{SR}, derived from experimental observations, which quantifies the rate of swelling by Eq. 3,

$$T(t) = \frac{Ts}{1 + (\frac{Ts}{To} - 1)e^{-K_{SR}t}} \quad (3)$$

where $T(t)$, T_o, and T_s depict the thickness swelling at time t, the initial, and the equilibrium board thickness, respectively. K_{SR} is a constant referred to as the initial (or intrinsic) relative swelling rate. Its value depends both on the speed at which the composites swell and the final equilibrium swelling reached.

Nonlinear curve fitting techniques were employed to estimate K_{SR} by minimizing the difference between the model predictions and experimental data. This was achieved by reducing the sum of squared residuals, defined as Eq. 4,

$$SS = \sum_{i=1}^{n} (y_i - \bar{y}i)^2 \qquad (4)$$

where SS is the sum of squared difference and y_i and $\bar{y}_i$ are the observed and predicted values of the dependent variable, respectively.

Statistical Analysis

Three replicate specimens were used for each formulation in all mechanical and physical tests, and the results are presented as the mean ± standard deviation. The experimental data were analyzed using two-way analysis of variance (ANOVA) to evaluate the main effects of thermal treatment and zinc borate addition, as well as their interaction, on the measured properties. When significant differences were detected ($p < 0.05$), Duncan's multiple range test was applied for pairwise comparisons of the treatment means. Water absorption and thickness swelling were determined using three replicate specimens for each formulation at each immersion period. All statistical analyses were conducted using IBM SPSS Statistics 24.0 (IBM Corp., Armonk, NY, USA).

RESULTS AND DISCUSSION

Mechanical Properties

Flexural strength and modulus of WPCs

The effects of thermal treatment, ZB addition, as well as their combination, on the flexural strength (FS) of WPCs were evaluated using two-way ANOVA (Table 2). Overall, neither thermal treatment nor ZB addition showed a significant main effect on FS when considered individually. In contrast, a significant interaction between thermal treatment and ZB addition was observed, indicating that the effect of ZB addition on flexural strength depended on the thermal treatment condition. Therefore, the influence of these factors should be interpreted based on their interaction rather than their individual effects.

The evaluation of the flexural performance of the WPCs under different treatment conditions including thermal exposure, ZB addition, and their combination demonstrated varying effects on their mechanical behavior. Thermal treatment at 150 °C for 120 min significantly enhanced the flexural strength, increasing it from 63.8 MPa in the control sample to 74.7 MPa (Fig. 2). This improvement is attributed to improved interfacial bonding between the polymer matrix and wood fibers, as thermal exposure facilitates moisture removal and partial degradation of hemicellulose, thereby promoting better matrix-fiber adhesion and load transfer efficiency (Esteves and Pereira 2009; Wang *et al*. 2021).

Table 2. Summary of Two-way ANOVA Results of the Effect of Thermal Treatment, ZB Addition, and Their Combination, on the Mechanical Properties of Beech Wood Flour/Polypropylene Composites

Property	Source	Sum of Squares	df	Mean Square	F	p-value
Flexural Strength (FS)	Corrected Model	218.448[a]	3	72.816	5.511	0.024
	Intercept	57362.692	1	57362.692	4341.473	0.000
	Thermal	26.078	1	26.078	1.974	0.198
	ZB	0.130	1	0.130	0.010	0.923
	Thermal * ZB	192.240	1	192.240	14.550	0.005
	Error	105.702	8	13.213		
	Total	57686.842	12			
	Corrected Total	324.150	11			
Flexural Modulus (FM)	Corrected Model	746983.000[a]	3	248994.333	1.759	0.232
	Intercept	349207563.000	1	349207563.000	2467.401	0.000
	Thermal	8965.333	1	8965.333	0.063	0.808
	ZB	707616.333	1	707616.333	5.000	0.056
	Thermal * ZB	30401.333	1	30401.333	0.215	0.655
	Error	1132228.000	8	141528.500		
	Total	351086774.000	12			
	Corrected Total	1879211.000	11			
Tensile Strength (TS)	Corrected Model	344.341[a]	3	114.780	49.668	0.000
	Intercept	20097.449	1	20097.449	8696.555	0.000
	Thermal	64.635	1	64.635	27.969	0.001
	ZB	36.436	1	36.436	15.766	0.004
	Thermal * ZB	243.270	1	243.270	105.268	0.000
	Error	18.488	8	2.311		
	Total	20460.278	12			
	Corrected Total	362.829	11			
Tensile Modulus (TM)	Corrected Model	6984472.667[a]	3	2328157.556	2.495	0.134
	Intercept	568398145.300	1	568398145.300	609.012	0.000
	Thermal	6681176.333	1	6681176.333	7.159	0.028
	ZB	180075.000	1	180075.000	0.193	0.672
	Thermal * ZB	123221.333	1	123221.333	0.132	0.726
	Error	7466498.000	8	933312.250		
	Total	582849116.000	12			
	Corrected Total	14450970.670	11			
Impact Strength (IS)	Corrected Model	1.981[a]	3	.660	13.501	0.002
	Intercept	573.392	1	573.392	11721.810	0.000
	Thermal	0.185	1	0.185	3.782	0.088
	ZB	0.429	1	0.429	8.778	0.018
	Thermal * ZB	1.367	1	1.367	27.943	0.001
	Error	0.391	8	0.049		
	Total	575.765	12			
	Corrected Total	2.373	11			

The addition of ZB also improved flexural strength, reaching 71.6 MPa. This can be ascribed to the role of ZB not only as a flame retardant and biocide but also as a filler that reinforces the composite structure by reducing voids and promoting particle dispersion within the matrix (Horrocks and Kandola 2005; Xie *et al*. 2010). However, when thermal treatment was combined with ZB, the flexural strength decreased to 66.5 MPa, which was

lower than either treatment applied alone. This suggests a possible antagonistic interaction, where the high-temperature process may alter the chemical form, distribution, or interfacial behavior of ZB, thereby reducing its reinforcing effect (Lu *et al*. 2019).

For flexural modulus (FM), the two-way ANOVA indicated that the overall model was not statistically significant. Neither thermal treatment nor the interaction between thermal treatment and ZB addition had a significant effect on FM (Table 2). Although the main effect of ZB addition approached statistical significance, it did not reach the 0.05 significance level. These findings indicate that the stiffness of the WPCs was not significantly affected by thermal treatment, ZB addition, or their interaction.

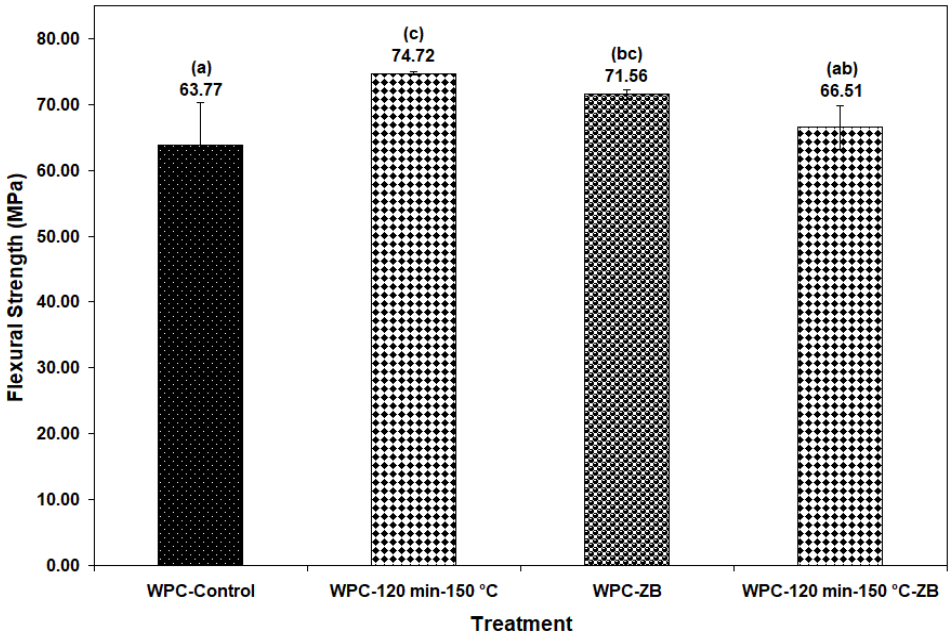

Fig. 2. The average and standard deviation of the flexural strength of the WPCs. The same letters in each bar show that there was no significant difference ($p<0.05$) between the WPC groups.

In contrast to strength, the flexural modulus values exhibited only minor, statistically insignificant variations across treatments, ranging from 5130 MPa (control) to 5720 MPa (ZB-treated) (Fig. 3). Despite slight numerical increases in stiffness in the ZB-containing samples, these were not significant according to the statistical grouping. This indicates that the treatments had a greater impact on strength than on stiffness. The limited change in flexural modulus could be due to the modest extent of matrix reinforcement provided by ZB or the counterbalancing effects of thermal degradation on the wood component's stiffness (Santos 2000; Esteves *et al*. 2007). Overall, these findings suggest that while thermal and chemical modifications can substantially enhance flexural strength, they may have limited influence on stiffness unless further formulation optimization is pursued.

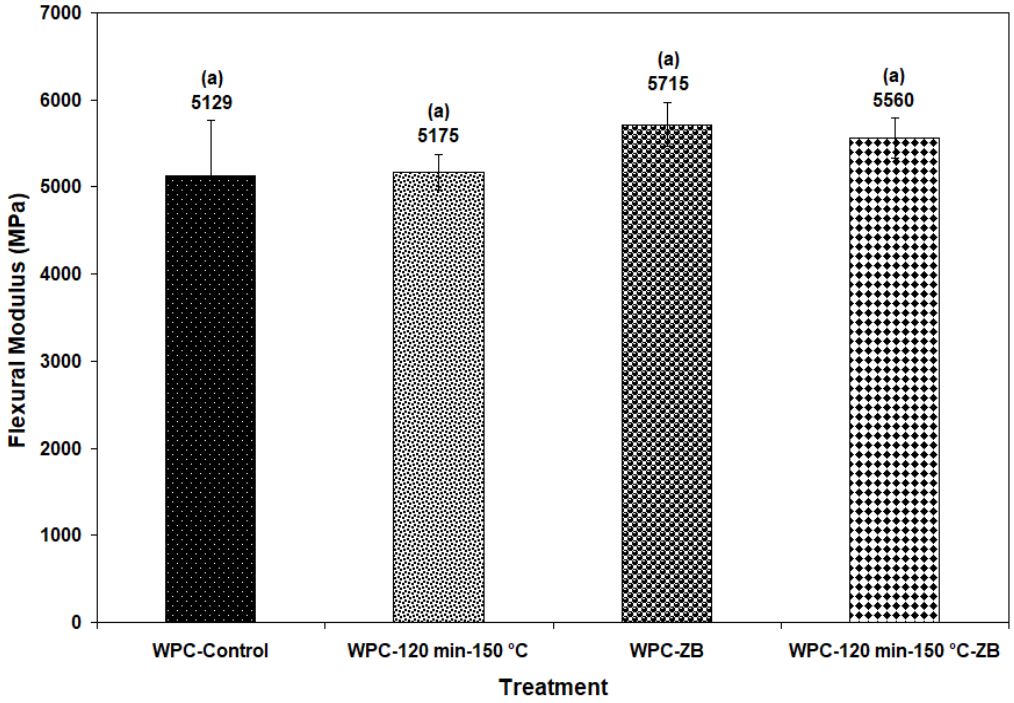

Fig. 3. The average and standard deviation of the flexural modulus of the WPCs. The same letters in each bar show that there was no significant difference ($p<0.05$) between the WPC groups.

The mechanical performance of WPCs was affected more prominently in terms of flexural strength than flexural modulus. Thermal treatment proved to be the most effective single strategy for enhancing strength, while ZB also had a positive effect when used independently. However, the combination of thermal treatment and ZB did not produce synergistic benefits and, in fact, resulted in decreased strength compared to the individual treatments. The flexural modulus remained statistically unchanged across all conditions, indicating that stiffness is less sensitive to these modifications. These results highlight the need to consider the interactive effects of thermal and chemical treatments carefully when designing WPCs for improved mechanical properties.

Tensile strength and modulus of WPCs

For tensile strength (TS), the two-way ANOVA revealed that both thermal treatment and ZB addition had significant main effects (Table 2). In addition, a highly significant interaction between thermal treatment and ZB addition was detected, indicating that the effect of ZB addition on TS was dependent on the thermal treatment condition.

Notably, Thermal treatment at 150 °C for 120 min increased tensile strength to 49.5 MPa, compared to 35.8 MPa in the untreated control (Fig. 4). This enhancement is attributable to improved fiber-matrix adhesion, resulting from moisture removal and partial depolymerization of hemicellulose, which improves load transfer efficiency (Jian *et al*. 2022).

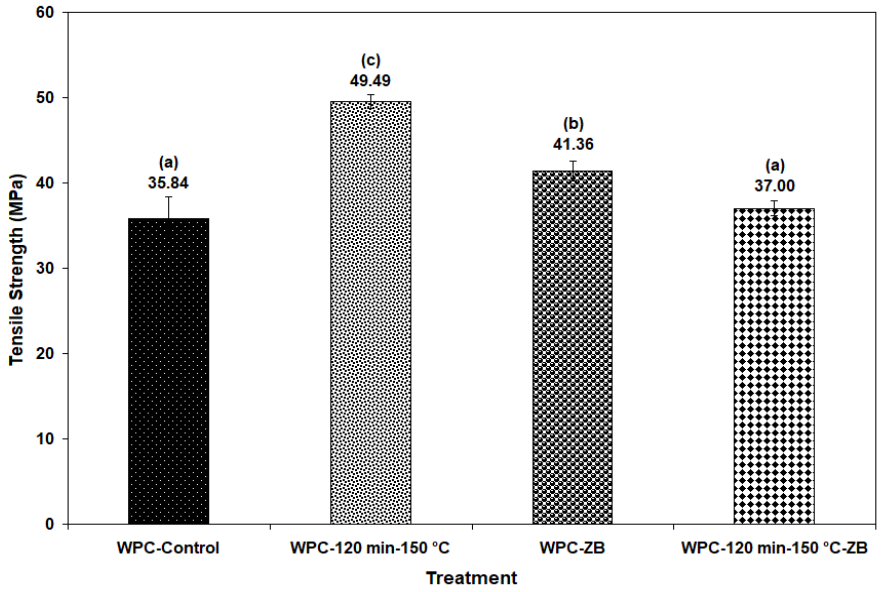

Fig. 4. The average and standard deviation of the tensile strength of the WPCs; The same letters in each bar show that there is no significant difference ($p<0.05$) between the WPC groups.

The ZB supplementation alone also raised tensile strength to 41.4 MPa. Recent studies confirm that low concentrations (1 to 2 wt%) of ZB can offer modest reinforcement effects by filling microvoids and distributing stress, while also imparting antifungal benefits (Zhao *et al*. 2025). However, some findings, such as those reported, suggest that ZB's mechanical role is limited. ZB has no effect on the mechanical properties of untreated WPCs, suggesting that its impact depends heavily on the method and amount used (Cárdenas Oscanoa *et al*. 2021).

When combining thermal treatment with ZB, tensile strength decreased to 37.0 MPa, statistically similar to the control implying a negative interaction. This could be due to ZB degradation at elevated temperatures or its impaired distribution within the polymer, which restricts reinforcement and may introduce interfacial defects (Badritala *et al*. 2013).

For tensile modulus (TM), thermal treatment had a significant main effect, whereas neither ZB addition nor the interaction between thermal treatment and ZB addition was statistically significant (Table 2). These results indicate that variations in TM were primarily associated with thermal treatment, while ZB addition did not significantly influence this property either independently or through interaction with thermal treatment.

Meanwhile, tensile modulus showed no significant differences across treatments, although numerical values ranged from 6160 MPa (control) to 7850 MPa (thermal-treated) (Fig. 5). The same statistical grouping across all bars indicates no real improvement. While thermal treatment might induce slight densification and fiber realignment (Lee *et al*. 2019), and the increase in the aggregates of $ZnHBO_3$ particles in a polymer matrix might marginally restrict polymer mobility (Yerlesen and Tasdemir 2015), the inherent stiffness of the WPC remained largely unaffected under these specific conditions. In summary, the tensile strength proved more responsive to both thermal and chemical modifications, whereas tensile stiffness remained stable underscoring different sensitivity scales for these mechanical properties.

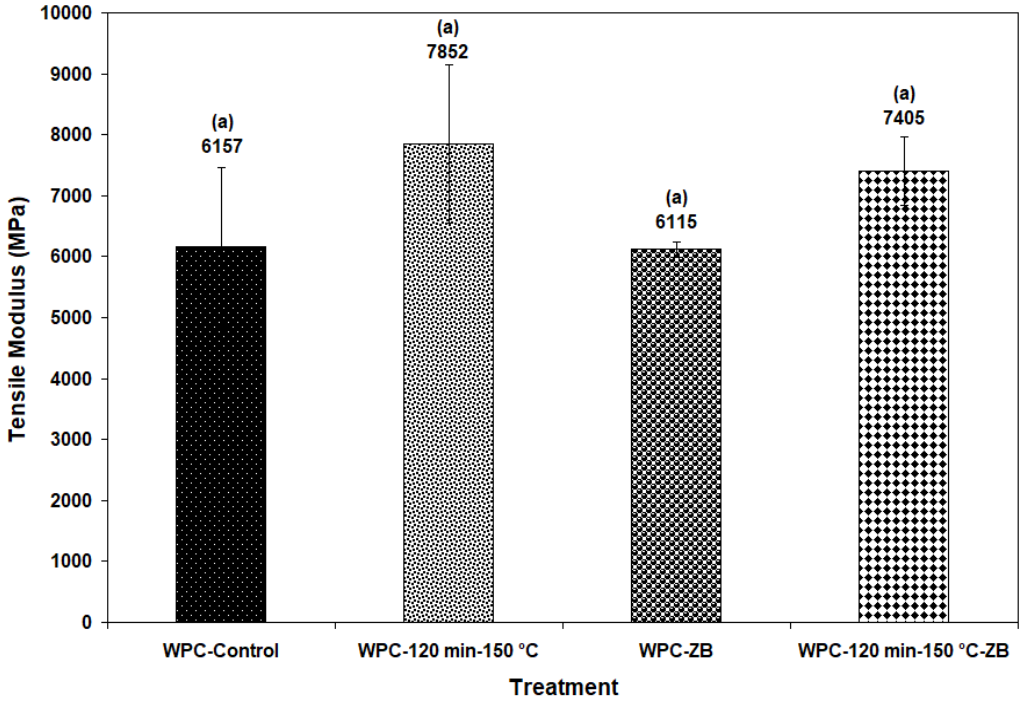

Fig. 5. The average and standard deviation of the tensile modulus of the WPCs; The same letters in each bar show that there is no significant difference ($p<0.05$) between the WPC groups.

Thermal treatment at 150 °C significantly enhanced the tensile strength of WPCs, which can be attributed to improved fiber-matrix interface quality. The ZB also contributed positively when used alone, though less effectively than heat. However, combining the two treatments yielded no synergistic benefits and actually reduced tensile strength to control levels. In contrast, tensile modulus was not statistically affected by any treatment, which reinforces the observation that stiffness is less sensitive to these specific modifications. These findings highlight the necessity of optimizing both additive type and processing conditions to enhance WPC mechanical performance effectively.

Impact strength of WPCs

The impact strength (IS) of the WPCs is presented in Fig. 6. Two-way ANOVA revealed a significant interaction between thermal treatment and zinc borate (ZB) addition, indicating that the effect of ZB depended on the thermal treatment condition (Table 2). Thermal treatment or ZB addition alone did not significantly affect impact strength, yielding values of 6.51 and 6.64 J/m, respectively, compared with 6.94 J/m for the control. However, the combined treatment significantly increased the impact strength to 7.56 J/m ($p < 0.05$).

The absence of improvement with ZB alone agrees with previous studies reporting that ZB has little or no positive effect on the impact performance of WPCs because of limited interfacial compatibility between the additive and polymer matrix (Suppakarn and Jarukumjorn 2009; Ayrilmis 2013; Turku *et al*. 2014; HosseiniHashemi *et al*. 2025). Likewise, the slight reduction after thermal treatment alone is consistent with the increased brittleness of heat-modified lignocellulosic materials. The enhanced impact strength observed only for the combined treatment suggests that the interaction between thermal

modification and ZB altered the failure behavior of the composite, although this beneficial effect was not reflected in the flexural or tensile properties.

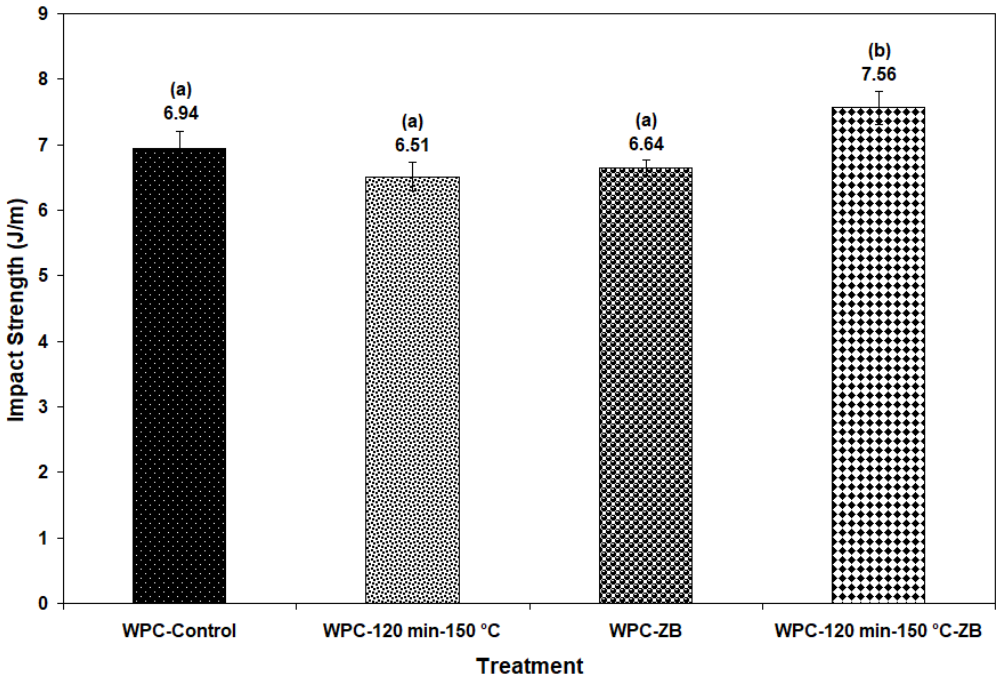

Fig. 6. The average and standard deviation of the impact strength of the WPCs. The same letters in each bar show that there is no significant difference ($p<0.05$) between the WPC groups.

The SEM micrographs (Fig. 13) revealed clear differences in the fracture morphology of the composites. The control sample exhibited pronounced fiber pull-out and interfacial gaps, indicating weak fiber-matrix adhesion. Thermal treatment resulted in a more compact fracture surface, although localized voids remained. The ZB-treated composite showed a relatively brittle fracture morphology with fragmented regions, suggesting the presence of rigid inorganic particles. In contrast, the combined thermal and ZB treatment produced reduced fiber pull-out and a more cohesive fracture surface, indicating improved interfacial contact between the wood flour and the polymer matrix.

Although these morphological changes were consistent with the improved impact strength of the combined treatment, they were not accompanied by corresponding improvements in flexural or tensile properties. Therefore, the enhanced impact resistance is attributed to changes in fracture behavior and localized energy dissipation rather than a general improvement in the mechanical performance of the composites.

Physical Properties

Long-term water absorption

Water absorption ratio of WPCs during immersion in water over 1440 h is presented in Fig. 7. Thermal treatment reduces OH groups, limiting hydrophilicity and capillary absorption (Ayrilmis *et al*. 2012; Priadi and Hiziroglu 2013). The ZB also acts as a hydrophobic filler, further decreasing water pathways. Pelaez-Samaniego *et al*. (2013) reported similar reductions in WPCs treated *via* heat and chemical agents. It is worth mentioning that the dual-treated group shows no major difference from ZB alone, suggesting ZB dominates in long-term resistance to water absorption.

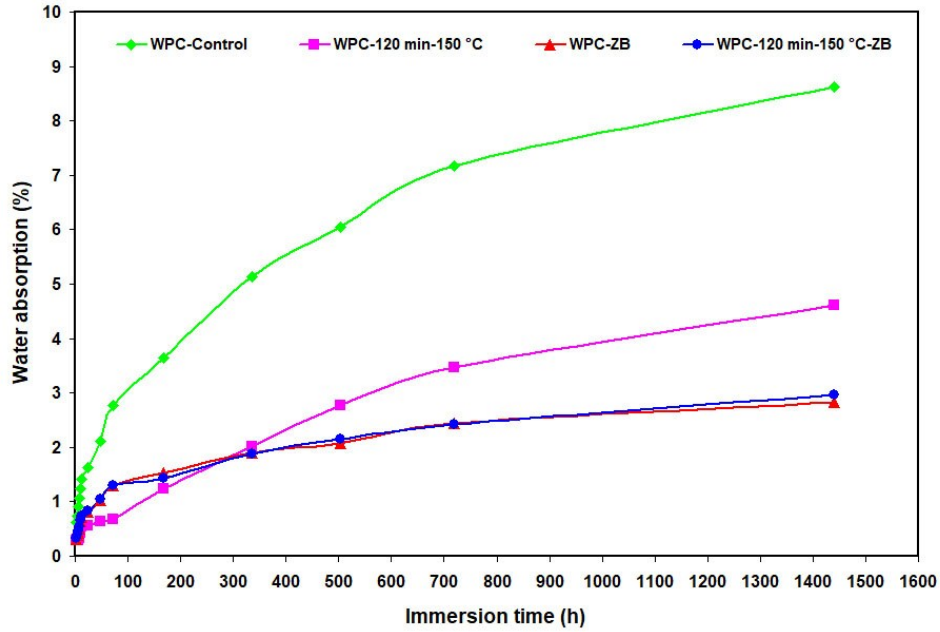

Fig. 7. Effect of thermal and ZB treatments on the long-term water absorption of WPCs

The reduction in water absorption observed in the ZB-containing composites may be attributed to several factors. First, the fine ZB particles may partially occupy interfacial voids within the composite structure, thereby reducing the available space for water accumulation. Second, the combined effect of thermal treatment and ZB addition may have altered the moisture transport pathways and limited the equilibrium water uptake. Furthermore, thermal treatment is known to reduce the number of accessible hydroxyl groups in wood by degrading hemicelluloses, thereby decreasing the hydrophilicity of the lignocellulosic filler. Consequently, although the ZB-containing composites exhibited higher diffusion coefficients, this parameter reflects the rate of moisture transport rather than the final amount of absorbed water, explaining the apparent discrepancy between diffusion behavior and equilibrium water absorption.

Long-term thickness swelling of WPCs

Figure 8 illustrates thickness swelling ratio of WPCs during immersion in water over 1440 h. All groups showed swelling between 4.9% and 5.0%, but WPC-Control showed faster initial swelling. The ZB-treated and thermally treated composites exhibited slower swelling rates over time. Although the maximum swelling was similar across all groups, the swelling kinetics was significantly altered. Thermal treatment reduces water-holding capacity by degrading hemicellulose and collapsing micropores, delaying water penetration. Thermal treatment also showed that treated lignocellulosic composites exhibited slow swelling behavior but stable over long exposure times, confirming these trends (Silveira *et al*. 2016). The ZB contributed by blocking capillaries and voids within the matrix (Cheng *et al*. 2019).

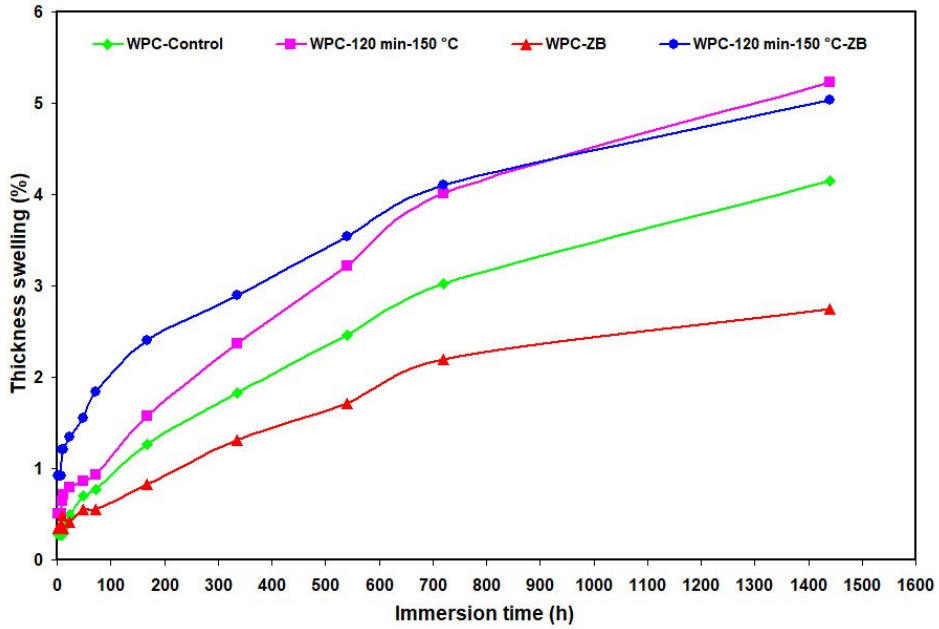

Fig. 8. Effect of thermal and ZB treatments on the long-term thickness swelling of WPCs

Fickian Diffusion Fitting

All the WPC samples exhibited Fickian or quasi-Fickian behavior. The control samples have higher slopes, indicating faster water ingress. The significantly lower n value (0.06) for thermally treated WPCs shows that the heat markedly reduces initial water diffusion (Fig. 9).

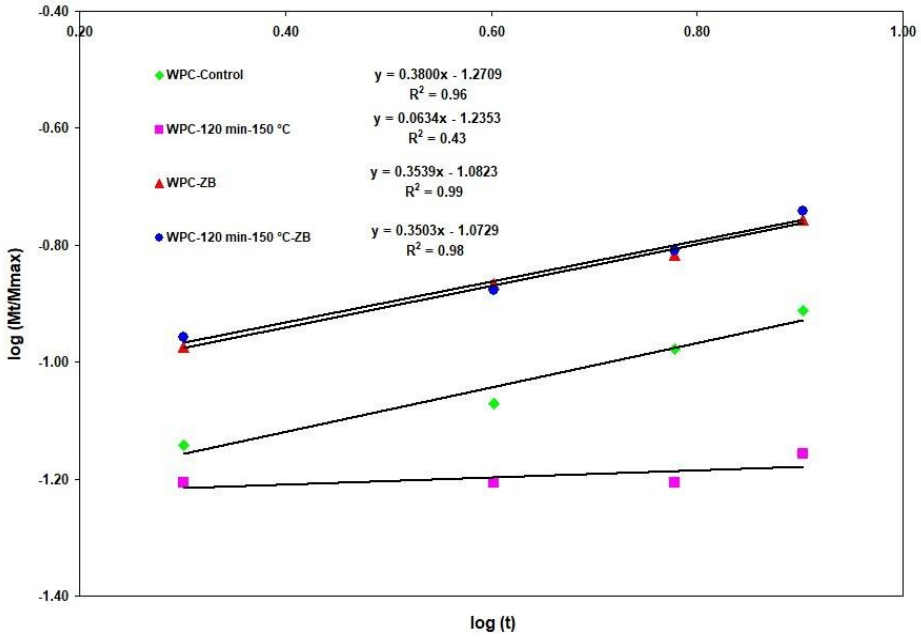

Fig. 9. Effect of thermal and ZB treatments on the diffusion case fitting for WPCs (t: immersion time (h); Mt: the water absorption at time t (%); M_{max}: the water absorption at the saturation point (%)

Similar low *n* values in the heat-treated wood were reported in previous studies (Adhikary *et al*. 2008), indicating delayed sorption due to fiber densification and microstructural collapse. The poor Fickian fit for the thermally treated sample ($R^2 = 0.43$, see Fig. 9) can be partly explained by the wood's sorption properties, which are affected by thermal treatment. This is because the treatment reduces the number of accessible hydroxyl (OH) groups and alters the wood's chemical composition. Initial drying also significantly impacts the accessibility of hydroxyl groups and the sorption properties of heat-treated wood (Kyyrö *et al.* 2021).

Water Diffusion Coefficient Analysis

It was observed that the diffusion coefficients of the ZB-treated and dual-treated samples were higher than those of the control sample, even though their overall water absorption was lower. This indicates faster initial absorption, likely due to the fine particle dispersion of the ZB, but with limited capacity (low M_{max}). This can be explained by the fact that the ZB affects interfacial porosity, creates micro-capillaries, and alters tortuosity of diffusion paths (Ayrilmis *et al*. 2012). In contrast, thermal treatment reduces both the initial rate and the total absorption, which is consistent with a strong Fickian barrier effect (Table 3 and Fig. 10) (Marcovich *et al*. 1999; Li *et al*. 2014).

Table 3. Water Diffusion, Maximum Water Absorption, *n*, and *k* Coefficient for All Composites

WPC Code	Maximum Water Absorption (%)	*N*	*k* (hr^2)	Water Diffusion Coefficient (m^2/s)
WPC-control	8.63	0.38	0.055	9.41×10^{-13}
WPC-120 min- 150 ºC	4.61	0.06	0.059	1.54×10^{-14}
WPC-ZB	2.84	0.35	0.083	1.58×10^{-12}
WPC-120 min-150 ºC-ZB	2.96	0.35	0.086	1.65×10^{-12}

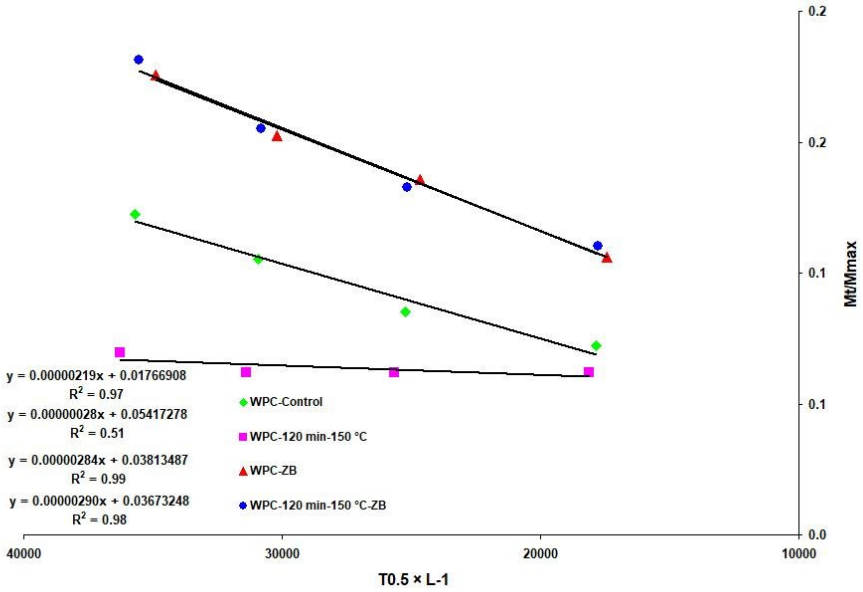

Fig. 10. Effect of thermal and ZB treatments on the water diffusion coefficient for WPCs ($t^{0.5}$: time (s); L^{-1}: thickness of specimen (m))

This contrast highlights how diffusion rate and total absorption are governed by different mechanisms.

Predicted *vs.* Experimental Thickness Swelling

Figure 11 compares the modeled swelling curves (based on Fickian fitting) with the experimental data for each WPC formulation.

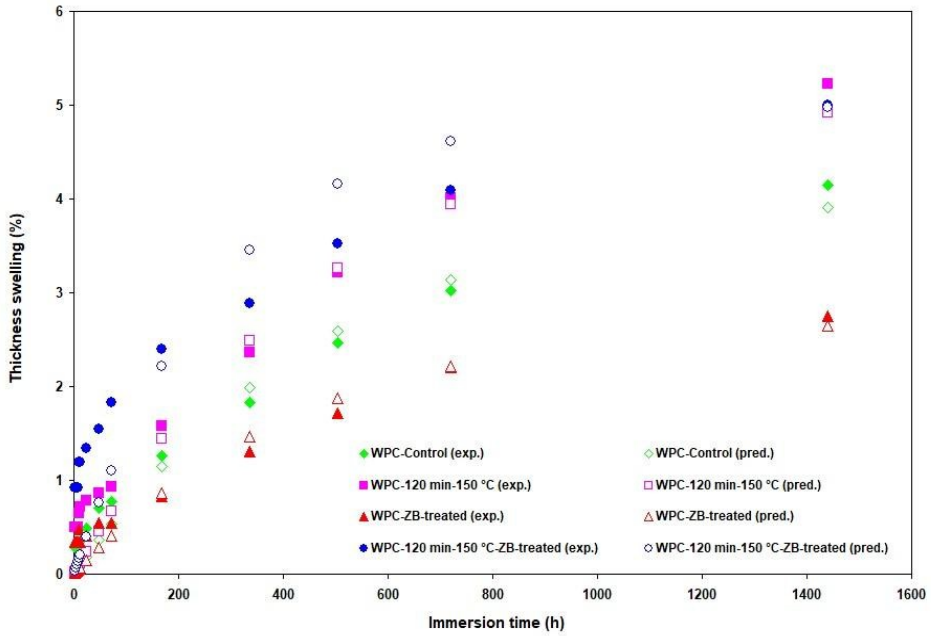

Fig. 11. Fitting predicted thickness swelling with experimental data for WPC specimens produced with thermally- and ZB-treated wood particles

The high correlation confirms that Fick's diffusion model accurately describes thickness swelling in WPCs, especially in the long-term region. Marcovich *et al*. (1999) and Clemons (2002) also showed excellent prediction accuracy using Fick-based models in similarly treated WPCs. Slight deviations at early times likely arise from surface relaxation or polymer chain mobility, especially in untreated samples. A strong ($R^2 = 0.99$) correlation between predicted and experimental thickness swelling was determined for WPC-control, WPC-120 min-150 ºC, WPC-ZB, and WPC-120 min-150 ºC-ZB, respectively. In addition, $R^2 = 0.98$ correlation was found in the specimens of WPC-120 min-150 ºC-ZB group.

Relationship Between Water Absorption and Thickness Swelling

A strong correlation exists between absorbed water and resulting thickness swelling (Fig. 12). The highest slope (1.59) in the dual-treated group suggests a steeper swelling response per unit of water, possibly due to early-stage micro-swelling of densified cell walls. This matched the results by Kokta *et al*. (1990), who also observed the variations in the slope based on treatment. The high R^2 values showed that swelling could be predicted based on water absorption, and that sorption-swelling was dependent on structure (Geffert *et al*. 2017).

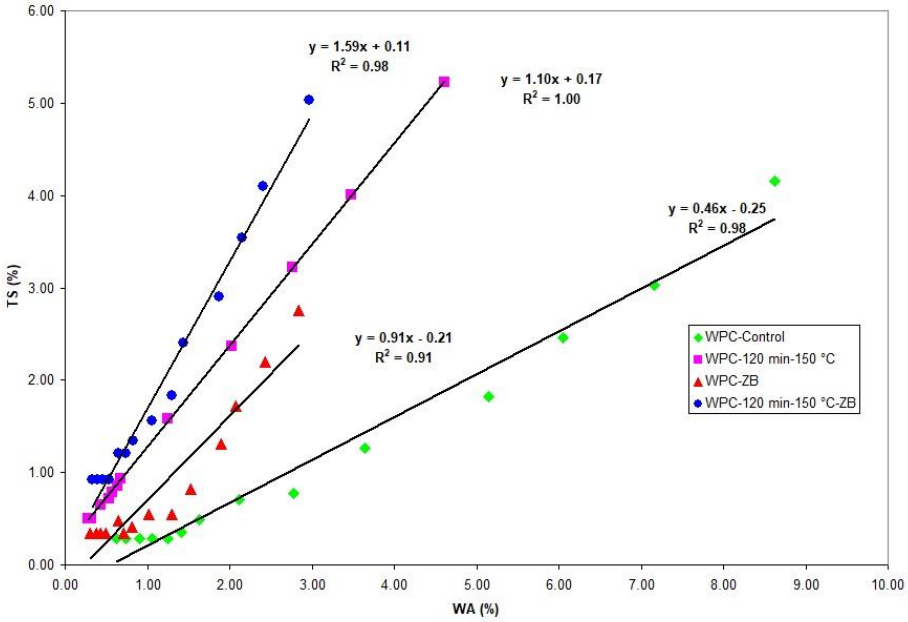

Fig. 12. Relationship between long-term water absorption (WA) and thickness swelling (TS) in studied composites

The observation that ZB-containing composites exhibited higher apparent diffusion coefficients despite lower maximum water absorption requires careful interpretation. Diffusion coefficient primarily reflects the initial rate of water ingress, whereas maximum water absorption (M_{max}) represents the total sorption capacity of the composite. The presence of inorganic particles, such as zinc borate, may introduce localized interfacial regions that facilitate early-stage water penetration, leading to an increased apparent diffusion rate. However, the overall hygroscopic capacity of the composite is reduced due to the lower wood flour content and the partial occupation of sorption sites by inorganic particles, limiting total moisture uptake. Similar distinctions between diffusion kinetics and equilibrium absorption capacity in wood-plastic composites and filled polymer systems have been reported in previous studies (Hill 2006; Ayrilmis *et al*. 2011; Tamrakar *et al*. 2011). These findings indicate that diffusion kinetics and equilibrium moisture content are governed by different structural factors, explaining why ZB-treated composites can exhibit relatively higher diffusion coefficients while maintaining lower equilibrium water absorption.

Water Absorption and Diffusion Parameters

The WPC-ZB and thermally treated WPC-ZB composites exhibited higher diffusion coefficients (D) than the control, whereas the control showed the lowest diffusion coefficient (Table 3). Thermal treatment significantly reduced both waterabsorption and water diffusion in lignocellulosic composites, indicating effective structural modification. It is worth mentioning that ZB-treated samples exhibited a lower water diffusion rate, although water diffusion remained relatively high, suggesting rapid water ingress followed by early saturation. A similar result for the WPCs containing zinc borate was observed by Ayrilmis (2013). The diffusion exponent (n) values below 0.5 further confirm Fickian diffusion or pseudo-Fickian behavior, particularly in thermally treated composites.

Swelling Rate Constants and Model Fit

The control group exhibited the highest swelling rate (K_{SR}), while all R^2 values exceed 0.97, indicating an excellent fit to swelling kinetics models. Dual-treated samples demonstrate a moderate K_{SR}, likely due to rapid initial water absorption followed by early saturation, reflecting enhanced dimensional stability. This behavior confirms that swelling is influenced by both diffusion dynamics and matrix-filler interactions, as observed in previous studies (Kord and Kiaeifar 2010).

Model Parameters for Swelling *vs.* Water Absorption

The high R^2 values indicateda strong correlation between water absorption and swelling (Fig. 12). WPC-120 min-150 °C-ZB had the steepest slope (1.59), indicating that for each unit water absorbed, thickness swelling increases more than in other samples. This behavior could be due to microstructural densification from thermal treatment, causing more dimensional change per water molecule absorbed. These trends validate using linear regression to predict swelling from water absorption, especially for long-term performance modeling (López-Naranjo *et al*. 2014).

SEM Analysis of WPCs Fracture Surfaces

The SEM images revealed distinct differences in fracture surface morphology between untreated and modified WPC samples (Fig. 13). These morphological variations highlight the effects of thermal and chemical surface treatments on interfacial bonding and fracture behavior. The control sample exhibits a rough, porous surface characterized by significant fiber pull-outs and interfacial voids, indicative of poor compatibility between the hydrophilic wood fibers and the hydrophobic polymer matrix. Such weak interfacial adhesion impairs effective stress transfer, often leading to premature mechanical failure (Debabeche *et al*. 2024). In contrast, the thermally treated sample exhibited a denser, more compact morphology. When lignocellulosic fibers are heated to high temperatures (for example, 150 °C for 2 h), the hemicellulose and volatile extractives are removed. This improves the way the fibers interact with the material around them (Wang and Howard 2018). As a result, the interfacial bonding becomes more efficient, though minor gaps remain, indicating partial enhancement.

The ZB-treated surface displays more brittle fracture features, with finely fragmented zones. The application of the ZB may chemically modify the surface polarity of wood particles, enhancing wettability and dispersion within the polymer matrix (Kazemi Najafi *et al*. 2007; Xie *et al*. 2013). However, the stiffer interface might reduce fracture toughness, favoring crack propagation through the filler-matrix interface. Among all samples, the combined thermally + ZB-treated specimen showed the most cohesive and smooth fracture surface. The absence of visible pull-outs and the uniform dispersion of particles indicate a synergistic effect: thermal treatment improves mechanical interlocking, while ZB enhances chemical bonding (Rowell *et al*. 1997). This morphology is associated with optimized load transfer and lower stress concentration, which could significantly enhance tensile and flexural performance (Rowell *et al*. 1997).

The dual-treated specimen exhibited a relatively smoother and more cohesive fracture surface, indicating improved interfacial continuity and reduced void formation. However, this morphological feature did not correspond to improved tensile and flexural strength, as the mechanical tests showed lower strength values for the dual-treated composite. This finding suggests that, besides fracture morphology, factors such as thermal

degradation of hemicellulose and changes in the wood-polymer interface also influenced the mechanical performance.

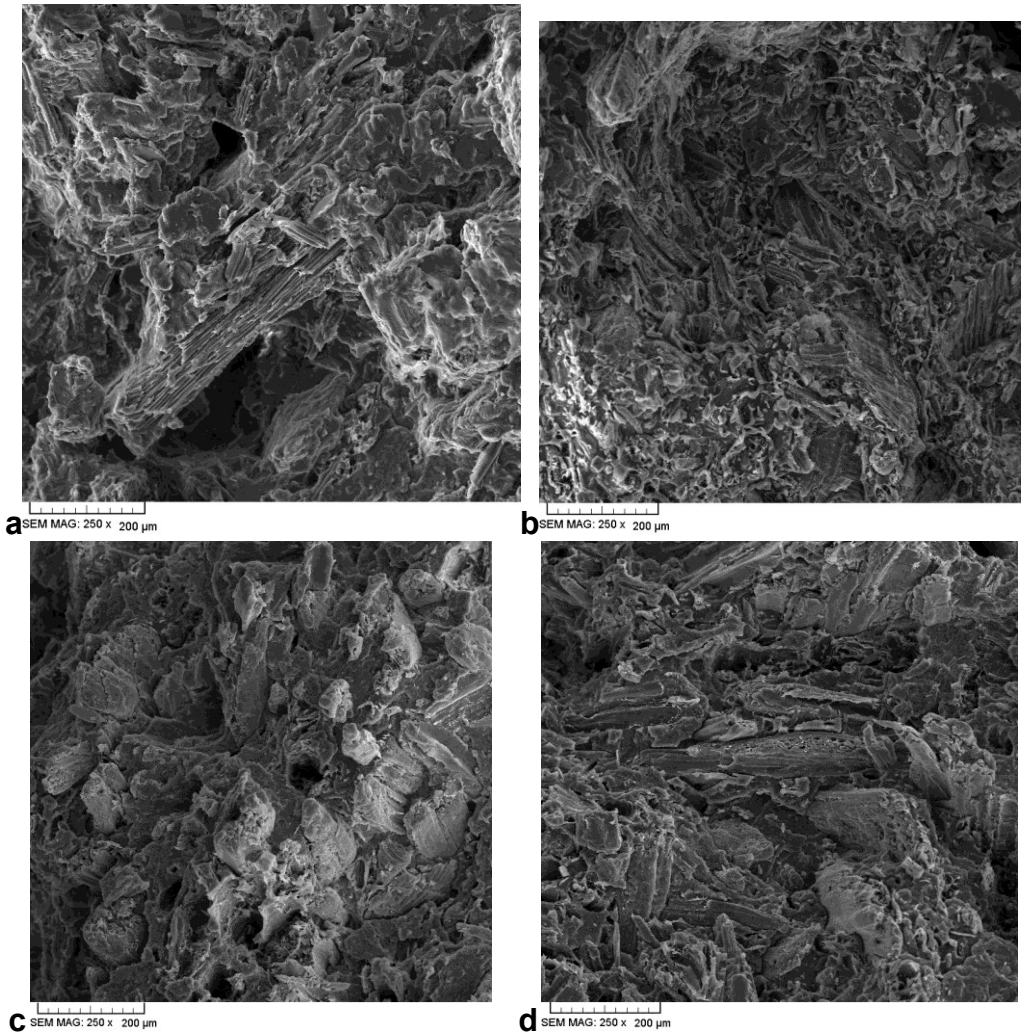

Fig. 13. SEM micrograph of the fracture surfaces in the WPC specimens produced with thermally- and ZB-treated wood particles: (a) control specimen, (b) 120 min-150 ºC, (c) ZB, and (d) 120 min-150 ºC-ZB

CONCLUSIONS

1. This study demonstrated that thermal treatment and zinc borate (ZB) addition influenced the performance of wood-plastic composites through different mechanisms.
2. Thermal treatment improved the flexural strength from 57.2 MPa in the control composite to 75.0 MPa, while the combined thermal treatment and ZB addition produced the highest impact strength (7.56 J/m). In contrast, the combined treatment did not improve tensile and flexural strength compared with thermal treatment alone.

3. Moisture diffusion analysis showed that ZB-containing composites exhibited higher diffusion coefficients (1.58 to 1.65 × 10^{-12} m^2/s) than the control (9.41 × 10^{-13} m^2/s), indicating that diffusion behavior should be interpreted separately from the overall moisture uptake.
4. Scanning electron microscope (SEM) observations confirmed that the combined treatment altered the fracture morphology; however, these microstructural changes were not directly reflected in improvements in tensile or flexural performance. Overall, the results indicate that the effects of thermal treatment and ZB addition are property-dependent and should be evaluated according to the specific performance requirements of WPC applications.
5. Although zinc borate is primarily used as a flame-retardant additive, the slight improvement observed in the flexural strength of the ZB-containing composite may be attributed to improved filler dispersion and reduced interfacial defects. Nevertheless, the statistical analysis indicated that the interaction between thermal treatment and ZB addition played a more important role than the main effect of ZB alone.

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