

Sonia Correa Jurado¹, José Guadalupe Rutiaga Quiñones¹, Nancy Eloísa Rodríguez Olalde¹, José Juan Alvarado Flores¹, Faustino Ruiz Aquino², Javier Ramón Sotomayor Castellanos^{*1}

Effect of Wood Ash and Boron Salts on Hygroscopicity of Sawdust Composites Bonded with Wheat Protein

Utjecaj drvnog pepela i soli bora na higroskopnost kompozita od piljevine lijepljenih pšeničnim proteinom

ORIGINAL SCIENTIFIC PAPER

Izvorni znanstveni rad

Received – prispjelo: 3. 1. 2026.

Accepted – prihvaćeno: 27. 2. 2026.

UDK: 674.812; 674.82

<https://doi.org/10.5552/drvind.2026.0320>

© 2026 by the author(s).

Licensee University of Zagreb Faculty of Forestry and Wood Technology.

This article is an open access article distributed

under the terms and conditions of the

Creative Commons Attribution (CC BY) license.

ABSTRACT • Bio-based wood composites bonded with wheat protein represent a sustainable alternative to conventional formaldehyde-based panels; however, their high moisture sensitivity limits dimensional stability and functional reliability. This study comparatively evaluated the effect of incorporating equivalent low dosages (5 wt.% dry basis) of wood ash and boron salts on the hygroscopic behavior of sawdust composites manufactured under identical formulation and processing conditions. By maintaining constant raw material, adhesive system, and consolidation parameters, the experimental design enabled direct assessment of additive performance within the same lignocellulosic matrix. Hygroscopic response was characterized through short-term water immersion tests, analyzing density, hygroexpansion, and water absorption index. Relative to the unmodified control, both additives produced statistically significant improvements with large effect sizes. Wood ash increased bulk density and markedly reduced water uptake, indicating microstructural modification and partial pore blocking. Boron salts achieved the greatest reduction in hygroexpansion, suggesting chemical stabilization of cell-wall polymers. The results demonstrate that even low mineral additions can substantially modify short-term moisture response without compromising adhesive consolidation. These findings provide controlled comparative evidence supporting the use of low-cost inorganic additives to enhance the dimensional stability of sustainable wood composites intended for interior applications.

KEYWORDS: dimensional stability; moisture sensitivity; inorganic additives; hygroexpansion; water absorption

SAŽETAK • Biokompoziti od drva lijepljeni pšeničnim proteinom održiva su alternativa konvencionalnim pločama na bazi formaldehida. Međutim, njihova visoka osjetljivost na vlagu utječe na njihovu dimenzijsku stabilnost i funkcionalnost. U ovoj studiji usporedno je procijenjen utjecaj dodavanja ekvivalentnih niskih doza (5 %-tni

* Corresponding authors

¹ Authors are researchers at Michoacán University of San Nicolás de Hidalgo, Avenida F. J. Múgica s/n, Morelia, Michoacán, México. <https://orcid.org/0009-0002-8401-5885>, <https://orcid.org/0000-0002-8617-8947>, <https://orcid.org/0000-0003-2921-0725>, <https://orcid.org/0000-0002-5756-0960>, <https://orcid.org/0000-0002-1527-8801>

² Author is researcher at University of Sierra de Juárez, Avenida Universidad s/n, Ixtlán de Juárez, Oaxaca, México. <https://orcid.org/0000-0001-6506-4441>

maseni udio suhe tvari) drvenog pepela i soli bora na higroskopnost kompozita od piljevine proizvedenih s istom formulacijom ljepila i u jednakim uvjetima prešanja. Održavanjem konstantnog sastava sirovine, sustava ljepila i parametara konsolidacije eksperiment je omogućio izravnu procjenu učinkovitosti aditiva unutar iste lignocelulozne matrice. Higroskopnost je proučavana kratkotrajnim testovima uranjanja u vodu, analiziranjem gustoće, promjenom dimenzija i indeksa upijanja vode. U odnosu prema nemodificiranome kontrolnom uzorku, aditivi su znatno poboljšali svojstva kompozita. Drveni je pepeo povećao gustoću i znatno smanjio upijanje vode, što upućuje na mikrostrukturnu modifikaciju i djelomično zapunjavanje pora. Dodavanjem soli bora postignuta je najbolja dimenzijska stabilnost, što upućuje na kemijsku stabilizaciju polimera staničnih stijenki. Rezultati pokazuju da čak i niski dodatci minerala mogu znatno promijeniti kratkotrajni odgovor na vlagu bez ugrožavanja konsolidacije ljepila. Ti rezultati daju kontrolirane usporedne dokaze koji podupiru upotrebu jeftinih anorganskih aditiva za poboljšanje dimenzijske stabilnosti održivih drvenih kompozita namijenjenih za unutarnju primjenu.

KLJUČNE RIJEČI: dimenzijska stabilnost; osjetljivost na vlagu; anorganski aditivi; promjene dimenzija; upijanje vode

1 INTRODUCTION

1. UVOD

Wood particle composite materials are a fundamental axis in the valorization of lignocellulosic waste for applications in furniture and interior components (Iždinský *et al.*, 2020). However, composites made with natural adhesives, such as wheat protein, exhibit marked hygroscopicity, which translates into dimensional instability and a reduction in their service life (Ferdosian *et al.*, 2017). Water-material interaction dominates the hygroscopic behavior and, consequently, the mechanical strength and durability of these systems, with moisture variations being responsible for hygroexpansion and losses of stiffness (Thybring *et al.*, 2022).

Recent studies indicate that sorption and hysteresis determine the hygroscopic equilibrium, especially under fluctuating conditions (Fredriksson *et al.*, 2023; Thybring *et al.*, 2022). Consequently, current research in wood technology is oriented towards the incorporation of sustainable mineral or ionic additives to mitigate hygroscopicity in lignocellulosic composites (Gonçalves *et al.*, 2021).

In the development of sustainable composites, protein adhesives derived from wheat gluten have been established as bio-based alternatives to synthetic resins that release formaldehyde in particleboards (Raydan *et al.*, 2021). Various studies have shown that gluten dispersions, especially when modified or cross-linked, can achieve adequate bond strength and dimensional stability for interior applications, provided pressing conditions are optimized (Aluvihare Gedara *et al.*, 2021; Calvez *et al.*, 2024).

In this sense, protein denaturation and chemical modification can alter the accessibility and reactivity of polar groups, and in some cases reduce net water sorption by promoting intra- and intermolecular crosslinks that reduce chain mobility and porosity; however, the net effect depends on the denaturation method and on crosslink density (Aluvihare Gedara *et al.*, 2021; Calvez *et al.*, 2024). Consequently, the use of

wheat protein as a sustainable adhesive provides an ideal basis for analyzing the comparative influence of inorganic additives, such as wood ash and boron salts, on the hygroscopicity of bio-based lignocellulosic matrices (Raydan *et al.*, 2021).

Wood ash, composed mainly of mineral oxides, has been used as a pozzolanic additive and alkalizing agent in cemented wood composites, due to its influence on the reactivity and stability of the matrix (Tekercan *et al.*, 2023). Research on wood-cement boards with partial substitution of cement by ash shows a progressive increase in absorption capacity by increasing its fraction, identifying an optimal range of 10-30 % to balance mechanical and physical properties (Vu *et al.*, 2019). The effects of ash on hygroscopicity derive from multiple interrelated mechanisms: modification of pH and local ionic strength, alteration of porosity and effective surface area by the incorporation of fine particles, and the presence of soluble fractions (alkaline salts, carbonates) that regulate capillary absorption and vapor sorption (Sigvardsen, 2019; Tekercan *et al.*, 2023; Vu *et al.*, 2019).

Although the use of wood ash has been predominantly investigated as a pozzolanic additive in wood-cement composites, its role in matrices bonded with bio-based adhesives remains insufficiently explored. In cement-based systems, ash fractions between 10-30 % have been observed to increase absorption capacity, an effect attributed to the modification of porosity and the presence of soluble salts. However, in a protein matrix such as the one proposed in the present study, it is postulated that the ash acts primarily as an inorganic filler, modifying the capillary network of the composite (Mwango and Kambole, 2019; Martínez-García *et al.*, 2022). This raises the question of whether, at low concentrations, it can physically hinder water diffusion pathways without compromising the integrity of the adhesive, a mechanism fundamentally different from that reported in inorganic matrices.

Boron compounds, mainly boric acid (H_3BO_3) and borax ($Na_2B_4O_7 \cdot 10H_2O$), are traditionally used as

low-toxicity preservatives in lignocellulosic products, providing protection against wood-destroying organisms. In addition to their biocidal action, various studies have shown that boron salts influence the hygroscopic and dimensional properties of wood composites, depending on their form, concentration, and method of incorporation (Khademibami and Bobadilha, 2022).

Impregnation tests with concentrations of 1-8 % boron have shown significant reductions in equilibrium moisture content and hygroexpansion, as in clones of *Eucalyptus* spp. treated with 4 % solutions (Baraúna *et al.*, 2020). This effect is attributed to the ability of borates to form complexes with hydroxyl groups of polysaccharides, their own intrinsic hygroscopicity, and their influence on the pH and curing reactions in adhesives (Ayrilmis, 2020; Khademibami and Bobadilha, 2022).

Thus, empirical evidence supports the effectiveness of boron compounds, such as boric acid and borax, in reducing hygroexpansion in wood. However, most of this research uses vacuum-pressure impregnation methods on solid wood, achieving significant reductions in equilibrium moisture content. This approach differs from that proposed in the present investigation, which evaluates the direct addition of borates into the sawdust matrix before pressing. Consequently, it is expected that the dominant mechanisms not only include the formation of complexes with polysaccharides, but also the alteration of the adhesive rheology and the interfacial properties between particles, an aspect not yet clarified in the literature.

The literature review shows that both ashes and borates have been widely studied, although generally in matrices, concentrations, and incorporation methods that are not comparable (Ayrilmis, 2020; Teker Ercan *et al.*, 2023; Vu *et al.*, 2019). While cemented boards use ash fractions between 10-30 % and boron treatments are usually applied by impregnation in ranges of 1-8 %, there are few studies that evaluate both additives in the same lignocellulosic matrix under homogeneous experimental conditions. Direct comparisons using a low dosage (5 % w/w on dry mass) of wood ash and boron salts in systems bonded with bio-based adhesives have barely been explored (Baraúna *et al.*, 2020; Khademibami and Bobadilha, 2022; Vu *et al.*, 2019). This experimental gap limits the understanding of their relative effectiveness in reducing hygroscopicity in composites made with sawdust.

Despite the industrial relevance of controlling moisture absorption in lignocellulosic composites, the literature lacks comparative studies that evaluate, in the same matrix and with the same natural wheat protein adhesive, the effects of homogeneous and low additions of wood ash or boron salts on the material's hygroscopicity. This lack of evidence generates uncertainty regarding the relative efficacy of these inorganic

additives in mitigating the hygroscopic instability of these bio-composites.

The research hypothesis proposes that the incorporation of 5 % by mass of ash or boron salts will reduce the hygroscopicity of the wood sawdust composites compared to the control composite without additives. Consequently, the present research aims to quantitatively determine and compare the effect of adding 5 % ash or 5 % boron salts on the hygroscopicity of wood sawdust composites, through an experimental design that allows contrasting their effects under identical composition and processing conditions.

The concentration of 5 % by weight was strategically selected as a low and constant dosage for both additives. This choice allows a direct comparison of their efficacy, avoiding the adhesive dilution effects or alterations in composite density that would be observed with larger additions. In this way, the experimental design focuses on elucidating whether, at this level of addition, the hygroscopic benefits outweigh any possible compromise in the functional properties of the final composite.

2 MATERIALS AND METHODS

2. MATERIJALI I METODE

2.1 Materials and board manufacture

2.1. Materijali i proizvodnja ploča

Three types of wheat protein-bonded sawdust composites were prepared under controlled laboratory conditions. The control formulation consisted of sawdust from Mexican white pine (*Pinus pseudostrabus* Lindl. var. *pseudostrabus*) (Figure 1) with a defined



Figure 1 Micrograph (10x magnification) of *P. pseudostrabus* particles from the fraction retained on a 850 μm sieve
Slika 1. Mikrografija (uz uvećanje od 10 puta) čestica drva *P. pseudostrabus* iz frakcije zadržane na situ od 850 μm

particle size distribution: > 850 μm (50 %), 425–850 μm (25 %), 250–425 μm (12.5 %) and < 250 μm (12.5 %). Prior to blending, the sawdust was oven-dried at $(103 \pm 2)^\circ\text{C}$ to constant mass (mass variation < 0.1 % between two measurements separated by 2 h) to obtain absolute dry material. All formulation proportions were calculated on an oven-dry sawdust basis.

The adhesive was prepared from commercial wheat flour (10 % protein content), following the protein quality characteristics described by Moreno-Araiza *et al.* (2020). The formulation consisted of 30 wt.% wheat flour and 200 wt.% distilled water, both expressed relative to the oven-dry mass of sawdust. The high-water content was required to ensure adequate protein gelatinization and homogeneous dispersion within the sawdust matrix. The mixture was stirred continuously for 2 min and heated to 98–100 $^\circ\text{C}$ until gelatinization occurred and a homogeneous, high-viscosity paste was obtained.

Two modified formulations were produced by incorporating inorganic additives at 5 wt.% relative to the oven-dry mass of sawdust (dry/dry basis). In the second composite (MC Ash), wood ash obtained from controlled combustion of *P. pseudostrobus* residues at 600 $^\circ\text{C}$ for 4 h (ISO 18122:2022) was incorporated. In the third composite (MC Boron), a mixture of boric acid (H_3BO_3 , 39.4 %) and sodium borate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$, 60.6 %) was added. In all cases, the dry components were blended prior to adhesive addition to ensure homogeneous distribution.

The furnish was manually pre-formed and subsequently compacted in a steel mold (2 cm \times 17 cm \times 17 cm) using a laboratory hydraulic press at 2.5 MPa for 5 min to minimize void formation and improve thickness uniformity (Figure 2). The wet plates were subjected to a stepped thermal schedule to promote adhesive gelation and moisture removal: 65 $^\circ\text{C}$ for 24 h, followed by 85 $^\circ\text{C}$ for 24 h, and finally 103 $^\circ\text{C}$ for 24 h.



Figure 2 Composite material specimens during the drying process in an oven at 103 $^\circ\text{C}$

Slika 2. Uzorci kompozitnih materijala tijekom procesa sušenja u sušioniku na 103 $^\circ\text{C}$

After drying, twelve specimens (2 cm \times 2 cm \times 5 cm) were cut from each board. The specimens were subsequently oven-dried at $(103 \pm 2)^\circ\text{C}$ to constant mass, defined as a mass variation of less than 0.1 % between two consecutive measurements separated by 2 h. This condition was considered the oven-dry reference state ($\text{MC} \approx 0\%$ on a wet basis), and all subsequent moisture contents were calculated relative to this oven-dry mass.

The complete formulation and manufacturing parameters are summarized in Table 1.

2.2 Methods

2.2. Metode

The absorption tests started with the specimens in oven-dry condition. These were immersed in water at a temperature of 20 $^\circ\text{C}$ and, at time intervals (t) of 10 minutes up to 120 minutes. Moisture content was determined using ISO 13061-1:2014. Short-term water uptake was measured by immersing specimens in deionized water at $(20 \pm 1)^\circ\text{C}$; at each time point specimens were removed, surface water was removed by gentle blotting with a damp lint-free cloth, and the sample was immediately weighed (balance accuracy ± 0.01 g). The 120-minute interval was selected to simulate short-term exposure and identify initial absorption differences between treatments. Density (ρ_{MC}) was calculated according to standard ISO 13061-2:2014. Weight and volume were determined by direct measurement at each time interval, considering the total expansion of the specimen. The wood ash was prepared according to standard ISO 18122:2022.

Hygroexpansion was calculated with Eq. 1 (Fu *et al.*, 2019):

$$\alpha = \left(\frac{v_s - v_i}{v_i} \right) \cdot 100 \quad (1)$$

Where α – hygroexpansion (%), v_s – volume corresponding to $t = 120$ min (m^3), v_i – initial volume corresponding to $t = 0$ min (m^3).

The water absorption index was calculated using Eq. 2 (Yang and Liu, 2020):

$$\text{WAI} = \left(\frac{w_s - w_i}{w_i} \right) \cdot 100 \quad (2)$$

Where WAI – water absorption index (%), w_s – weight corresponding to $t = 120$ min (g), w_i – weight corresponding to $t = 50$ min (g).

2.3 Experimental design

2.3. Postavke eksperimenta

A completely randomized experiment was designed. The composite formulation was considered with the variation factor at three levels: Control (base formulation), Ash (formulation with 5 % ash) and Boron (formulation with 5 % boron salts). The total number of units was 36 (3 treatments \times 12 independent

Table 1 Composition and manufacturing parameters of wheat protein–bonded sawdust composites**Tablica 1.** Sastav i parametri proizvodnje kompozita od piljevine lijepjenih pšeničnim proteinom

Parameter Parametar	MC Control Uzorak za kontrolu sadržaja vode	MC Ash (5 wt.%) Sadržaj vode uzorka s dodatkom pepela (5 %-tni maseni udio)	MC Boron (5 wt.%) Sadržaj vode uzorka s dodatkom soli bora (5 %-tni maseni udio)
Wood species vrsta drva	<i>Pinus pseudostrobus</i> Lindl. var. <i>pseudostrobus</i>	Same as control jednako kao u kontrolnom uzorku	Same as control jednako kao u kontrolnom uzorku
Sawdust condition svojstva piljevine	Oven-dry ((103 ± 2) °C, constant mass) sušenje u sušioniku (103 ± 2 °C, konstantna masa)	Same as control jednako kao u kontrolnom uzorku	Same as control jednako kao u kontrolnom uzorku
Particle size distribution distribucija veličine čestica	> 850 μm (50 %); 425 – 850 μm (25 %); 250–425 μm (12.5 %); < 250 μm (12.5 %)	Same as control jednako kao u kontrolnom uzorku	Same as control jednako kao u kontrolnom uzorku
Adhesive type vrsta ljepila	Wheat flour (10 % protein) pšenično brašno (10 % proteina)	Same as control jednako kao u kontrolnom uzorku	Same as control jednako kao u kontrolnom uzorku
Adhesive formulation ¹ formulacija ljepila ¹	30 wt.% flour + 200 wt.% water 30 masenih postotaka brašna + 200 masenih postotaka vode	Same as control jednako kao u kontrolnom uzorku	Same as control jednako kao u kontrolnom uzorku
Additive type vrsta dodataka	—	Wood ash / drvni pepeo	Boric acid + sodium borate borna kiselina + natrijev borat
Additive dosage ² količina aditiva ²	—	5 wt.%	5 wt.%
Ash preparation priprema pepela	—	600 °C, 4 h (ISO 18122:2022)	—
Mat forming formiranje tepiha	Manual pre-forming	Same as control jednako kao u kontrolnom uzorku	Same as control jednako kao u kontrolnom uzorku
Pressing conditions parametri prešanja	2.5 MPa, 5 min	Same as control jednako kao u kontrolnom uzorku	Same as control jednako kao u kontrolnom uzorku
Plate dimensions dimenzije ploče	2 cm × 17 cm × 17 cm	Same as control jednako kao u kontrolnom uzorku	Same as control jednako kao u kontrolnom uzorku
Drying schedule raspored sušenja	65 °C × 24 h → 85 °C × 24 h → 103 °C × 24 h	Same as control jednako kao u kontrolnom uzorku	Same as control jednako kao u kontrolnom uzorku
Specimen dimensions dimenzije uzoraka	2 cm × 2 cm × 5 cm	Same as control jednako kao u kontrolnom uzorku	Same as control jednako kao u kontrolnom uzorku
Number of specimens (n) broj uzoraka (n)	12	12	12

¹Expressed relative to oven-dry sawdust mass / u odnosu prema masi piljevine osušene u sušioniku²Expressed on oven-dry sawdust basis (dry/dry) / u odnosu prema masi piljevine osušene u sušioniku (suho/suho)

replicates). The response variables were density ρ_{MC} (kg/m³), hygroexpansion α (%) and water absorption index WAI (%).

Descriptive statistics mean (μ), standard deviation (σ) and coefficient of variation ($CV = \sigma/\mu$) were calculated. The assumptions of normality (Shapiro-Wilk): evaluated individually by group ($p > 0.05$ indicates normal distribution); and homoscedasticity (Levene): verification of equality of variances between groups ($p > 0.05$ indicates homogeneity) were verified. One-way analyses of variance (ANOVA) were per-

formed, when the ANOVA was significant ($p < 0.05$), as well as multiple comparisons with Tukey's Honestly Significant Difference (HSD) tests. In the case of hygroexpansion (α), the data did not meet the parametric assumptions. Therefore, the analysis of hygroexpansion (α %) was conducted using Welch's ANOVA followed by the Games-Howell post-hoc test. Effect sizes (η^2 and ω^2) and power ($1 - \beta$) were calculated from the observed effect size ($\eta^2 \rightarrow f$), the total sample size and for $\alpha = 0.05$. Adequate power was considered when $1 - \beta \geq 0.80$.

3 RESULTS AND DISCUSSION

3. REZULTATI I RASPRAVA

The physical properties of the wheat protein-bonded sawdust composites are summarized in Table 2. Results are expressed as mean \pm standard deviation ($n = 12$). Statistically significant differences among formulations are indicated by superscript letters ($\alpha = 0.05$).

Regarding density, the ash-modified composite exhibited the highest mean value (435 kg/m^3), differing significantly from both the control and the boron-modified composite, which did not differ from each other. The relatively low dispersion values indicate consistent manufacturing across treatments. The significant F-value ($27.93, p < 0.001$) confirms that composite formulation influenced bulk density. The effect size ($\eta^2 = 0.629$) indicates that approximately 63 % of total density variance is attributable to additive incorporation, evidencing a strong structural impact of ash addition.

For hygroexpansion, the control composite showed the highest volumetric swelling (21.7 %), whereas both modified composites presented significantly lower values (14.9 % for ash and 13.0 % for boron), without statistical difference between them. The overall model was significant (Welch-adjusted $F = 18.19, p < 0.001$). The effect size ($\eta^2 = 0.524$) reveals that more than half of the variability in swelling behavior is governed by formulation, demonstrating that both additives substantially improve dimensional stability relative to the unmodified matrix.

In the case of water absorption index, all formulations differed significantly, following the order Control > Ash > Boron. The control composite exhibited the highest short-term water uptake (177 %), while the boron-modified composite showed the lowest value (148 %). The large F-statistic ($96.80, p < 0.001$) and very high effect size ($\eta^2 = 0.854$) indicate that formulation explains more than 85 % of the variance in water absorption. This represents the strongest treatment effect among the evaluated properties and confirms the

dominant role of additive incorporation in regulating short-term capillary water uptake.

Overall, the consolidated presentation integrates descriptive and inferential statistics in a single table, providing a concise yet statistically rigorous evaluation of treatment effects. The magnitude of the observed effect sizes demonstrates that even a low additive dosage (5 wt.% dry basis) produces substantial and practically meaningful modifications in the hygroscopic performance of wheat protein-bonded sawdust composites under short-term immersion conditions.

Hygroscopicity is a decisive factor in the durability and dimensional stability of lignocellulosic composite materials, as moisture variations cause volumetric deformations and reduce mechanical performance. According to the experimental results, the MC control, without additives, showed the highest hygroexpansion and water absorption index, in addition to high variability, which reflects its sensitivity to moisture. The statistical significance of the analysis of variance confirms that the differences between the groups are due to the modifications introduced (Rahim *et al.*, 2024). Likewise, the incorporation of 5 % ash or boron salts was compatible with the wheat protein adhesive, allowing adequate consolidation of the material. Consequently, the effects on hygroscopic properties are attributed to the additives, consolidating their role in improving the stability and functionality of sawdust composites.

The effect of wood ash on hygroscopicity is complex and depends on the balance between pore filling and the introduction of hygroscopic soluble salts. The results of this study (Table 2) show that, with a low concentration of 5 %, the ash had a positive effect. The ash-modified composite showed a significant and important reduction in hygroexpansion compared to the control, as well as in the water absorption index. The significant difference in the density of the composite containing ash, compared to the control and the composite containing boron, supported by the Tukey HSD test, suggests a modification in the microstructure of the composite. This modification may be due to an improvement in

Table 2 Physical properties of wheat protein-bonded sawdust composites (mean \pm SD, $n = 12$)

Tablica 2. Fizička svojstva kompozita od piljevine lijepljenih pšeničnim proteinom (srednja vrijednost \pm SD, $n = 12$)

Property Svojstvo	MC control Kontrolni uzorci	MC ash (5 wt.%) Uzorci s dodatkom drvnog pepela	MC boron (5 wt.%) Uzorci s dodatkom soli bora	F	p-value	η^2
Density, kg/m^3 / gustoća, kg/m^3	414 \pm 12.9 ^b	435 \pm 13.5 ^a	408 \pm 17.5 ^b	27.93	< 0.001	0.629
Hygroexpansion, % promjene dimenzija, %	21.7 \pm 6.4 ^a	14.9 \pm 2.7 ^b	13.0 \pm 2.2 ^b	18.19*	< 0.001	0.524
Water absorption index, % indeks upijanja vode, %	177 \pm 1.5 ^a	160 \pm 1.4 ^b	148 \pm 0.8 ^c	96.80	< 0.001	0.854

Notes: Different superscript letters within the same row indicate statistically significant differences at $\alpha = 0.05$; Density and WAI were analyzed by one-way ANOVA followed by Tukey's HSD test; Hygroexpansion was analyzed using Welch's ANOVA and Games-Howell post-hoc test due to heteroscedasticity; $\eta^2 =$ proportion of total variance explained by formulation (effect size).

Napomena: Različita slova u natpisu brojeva unutar istog retka označavaju statistički značajne razlike pri $\alpha = 0,05$; gustoća i indeks upijanja vode analizirani su jednosmjernom ANOVA-om, nakon čega je slijedio Tukeyjev HSD test; promjene dimenzija su zbog heteroskedastičnosti analizirane Welchovom ANOVA-om i Games-Howell post-hoc testom; $\eta^2 =$ udio ukupne varijance objašnjen formulacijom (veličina učinka).

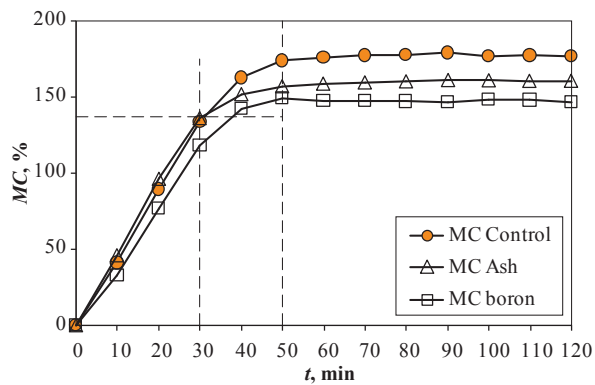


Figure 3 Variation of moisture content (MC) as a function of immersion time (t), each point represents mean \pm standard deviation ($n = 12$)

Slika 3. Varijacija sadržaja vode (MC) kao funkcija vremena uranjanja (t); svaka točka predoduje srednju vrijednost \pm standardnu devijaciju ($n = 12$)

compaction or a change in particle distribution, which in turn contributes to the reduction in water absorption.

The composite containing boron showed the greatest decrease in hygroexpansion, and this difference is statistically significant compared to the control. This supports the idea that borates can work well with the lignocellulosic matrix to limit hygroexpansion (Ay-rilmis, 2020; Teker Ercan *et al.*, 2023; Vu *et al.*, 2019). However, its performance in water absorption was intermediate, notably higher than that of ash and notably lower than that of the control. This result indicates a dual behavior: while borates are effective in restricting hygroexpansion caused by moisture adsorbed in the cell wall, their hygroscopic nature could be favoring the retention of a greater amount of liquid water in the material, which is reflected in a water absorption index higher than that observed in the case of ash.

The moisture content of the three analyzed composites showed an evolution as a function of the immersion time, evidencing a general trend of rapid increase in the initial stages of exposure (0-50 minutes), followed by a stabilization phase between 50 and 120 minutes (Figure 3). The profile of these trends resembles that documented by Fu *et al.* (2019) for *Pinus radiata* D. Don wood, with immersion periods of 300 hours, and that reported by Widiastuti *et al.* (2023) for wood-plastic composites, with periods of 240 hours. The materials analyzed and the immersion times in water documented by the authors cited differ from those examined in the present investigation. The current study employed a 120-minute immersion to investigate short-term capillary uptake and near-surface saturation, thus limiting comparisons with multi-day immersion studies; nonetheless, the early uptake kinetics is similar in structure and permit inferences about initial sorption mechanisms (capillarity versus cell-wall sorption). Nonetheless, the trends in hygroscopic behavior are analogous and corroborate the interpretation of Figure 3.

4 CONCLUSIONS

4. ZAKLJUČAK

This study comparatively evaluated the effect of low and equivalent dosages (5 wt.% dry basis) of wood ash and boron salts on the hygroscopic performance of wheat protein-bonded sawdust composites manufactured under identical formulation and processing conditions. By controlling all other variables, the experimental design allowed a direct assessment of the relative efficacy of both inorganic additives within the same lignocellulosic matrix.

The unmodified control composite exhibited pronounced hygroexpansion and water absorption, confirming the inherent moisture sensitivity of protein-bonded systems. In contrast, both modified composites showed statistically significant improvements, supported by large effect sizes. Wood ash incorporation increased bulk density and markedly reduced water absorption, indicating a predominantly microstructural mechanism associated with partial pore filling and modification of capillary pathways. Boron salts produced the greatest reduction in hygroexpansion, suggesting that chemical interactions with hydroxyl groups in the cell-wall polymers contributed to restricting moisture-induced dimensional changes.

The results demonstrate that even at a relatively low concentration (5 wt.%), mineral additives can substantially modify short-term moisture response without impairing adhesive consolidation. The distinct yet complementary mechanisms identified for ash and boron highlight that additive selection can be tailored depending on whether the primary objective is to reduce water uptake or to minimize dimensional instability.

Although the findings are limited to one species, one adhesive system, a single additive concentration, and short-term immersion conditions, they provide controlled comparative evidence supporting the feasibility of low-cost inorganic additives as functional modifiers in sustainable, bio-based wood composites intended for interior applications.

Acknowledgements – Zahvala

The research was sponsored by the Michoacana University of San Nicolás de Hidalgo (UMSNH, Mexico), the Institute of Science, Technology, and Innovation of the State of Michoacán (ICTI, Mexico), Mexico and the Secretariat of Science, Humanities, Technology, and Innovation (SECIHTI, Mexico).

5 REFERENCES

5. LITERATURA

1. Aluvihare Gedara, A. K.; Chianella, I.; Bhattacharyya, D.; Endrino, J. L.; Zhang, Q., 2021: Alkali-treated wheat gluten cross-linked with sodium alginate as a bio-based

- wood adhesive for interior grade particleboard. *BioResources*, 16 (4): 7916-7934. <https://doi.org/10.15376/biores.16.4.7916-7934>
2. Ayrlimis, N., 2020: Effect of boron and phosphorus compounds on fire and technological properties of oriented strandboard. *Materials International*, 2: 117-122. <https://doi.org/10.33263/Materials22.117122>
 3. Baraúna, E. E. P.; Paes, J. B.; Christoforo, A. L.; Lahr, F. A. R., 2020: Influence of the impregnation with boron compounds on the physical properties of Eucalyptus wood. *Scientia Forestalis*, 48 (128): e3383. <https://doi.org/10.18671/scifor.v48n128.09>
 4. Calvez, I.; Pizzi, A.; Amirou, S.; Mansouri, H. R., 2024: Recent advances in bio-based adhesives and formaldehyde-free technologies for wood-based panel manufacturing. *Current Forestry Reports*, 10 (5): 386-400. <https://doi.org/10.1007/s40725-024-00227-3>
 5. Ferdosian, F.; Pan, Z.; Gao, G.; Zhao, B., 2017: Bio-based adhesives and evaluation for wood composites application. *Polymers*, 9 (2): 70. <https://doi.org/10.3390/polym9020070>
 6. Fredriksson, M.; Thybring, E. E.; Zelinka, S. L., 2023: Water sorption in wood cell walls – data exploration of the influential physicochemical characteristics. *Cellulose*, 30: 1857-1871. <https://doi.org/10.1007/s10570-022-04973-0>
 7. Fu, Z.; Zhou, Y.; Gao, L.; Zhao, R.; Tu, D., 2019: Changes of water related properties in radiata pine wood due to heat treatment. *Construction and Building Materials*, 227: 116692. <https://doi.org/10.1016/j.conbuildmat.2019.116692>
 8. Gonçalves, D.; Paiva, N.; Ferraz, J. M.; Martins, J.; Magalhães, F. D.; Carvalho, L. H., 2021: Non-formaldehyde, bio-based adhesives for use in wood-based panel manufacturing industry – A review. *Polymers*, 13 (23): 4086. <https://doi.org/10.3390/polym13234086>
 9. Iždinský, J.; Vidhodlová, Z.; Reinprecht, L., 2020: Particleboards from recycled wood. *Forests*, 11 (11): 1166. <https://doi.org/10.3390/f11111166>
 10. Khademibami, L.; Bobadilha, G. S., 2022: Recent developments studies on wood protection research in academia: A review. *Frontiers in Forests and Global Change*, 5: 793177. <https://doi.org/10.3389/ffgc.2022.793177>
 11. Martínez-García, R.; Alaejos, P.; Vegas, I.; Frías, M., 2022: The present state of the use of waste wood ash as an eco-efficient construction material: A review. *Materials*, 15 (15): 5349. <https://doi.org/10.3390/ma15155349>
 12. Moreno-Araiza, O.; López, A.; Ramírez, J.; Rodríguez, M., 2020: Protein quality in roller-milling fractions of wheat (*Triticum aestivum*) at a commercial scale. *Biotechnia*, 22 (3): 53-60. <https://doi.org/10.18633/biotechnia.v22i3.1201>
 13. Mwango, A.; Kambole, C., 2019: Engineering characteristics and potential increased utilisation of sawdust composites in construction – A review. *Journal of Building Construction and Planning Research*, 7: 59-88. <https://doi.org/10.4236/jbopr.2019.73005>
 14. Rahim, N. L.; Hassan, M. F.; Zain, M. F. M.; Abdullah, S.; Ismail, N., 2024: Development of eco-friendly particleboard composite using fly ash. *E3S Web of Conferences*, 589: 04002. <https://doi.org/10.1051/e3s-conf/202458904002>
 15. Raydan, N. D. V.; Silva, R. A.; Oliveira, J. R.; Santos, L. M., 2021: Recent advances on the development of protein-based adhesives for wood composite materials – A review. *Molecules*, 26 (24): 7617. <https://doi.org/10.3390/molecules26247617>
 16. Sigvardsen, N. M.; Jensen, P. A.; Dam-Johansen, K.; Ahrenfeldt, J., 2019: Impact of production parameters on physicochemical characteristics of wood ash for possible utilisation in cement-based materials. *Resources, Conservation and Recycling*, 145: 230-240. <https://doi.org/10.1016/j.resconrec.2019.02.034>
 17. Teker Ercan, E. E.; Kaya, A.; Yilmaz, H.; Demir, I., 2023: Wood ash as sustainable alternative raw material for the production of concrete – A review. *Materials*, 16 (7): 2557. <https://doi.org/10.3390/ma16072557>
 18. Thybring, E. E.; Fredriksson, M.; Englund, E. T., 2022: Water in wood: A review of current understanding and knowledge gaps. *Forests*, 13 (12): 2051. <https://doi.org/10.3390/f13122051>
 19. Vu, V.-A.; Nguyen, T. H.; Tran, Q. T.; Pham, D. H., 2019: The effect of wood ash as a partial cement replacement material for making wood-cement panels. *Materials*, 12 (17): 2766. <https://doi.org/10.3390/ma12172766>
 20. Widiastuti, I.; Suryanegara, L.; Hadi, Y. S.; Santoso, A., 2023: Optimizing the water absorption behaviour and natural weathering resistance of compatibilized iron-wood-based recycled polypropylene composites. *Composites. Part C: Open Access*, 12: 100423. <https://doi.org/10.1016/j.jcomc.2023.100423>
 21. Yang, L.; Liu, H.-H., 2020: Effect of a combination of moderate-temperature heat treatment and subsequent wax impregnation on wood hygroscopicity, dimensional stability and mechanical properties. *Forests*, 11 (9): 920. <https://doi.org/10.3390/f11090920>
 22. ***ISO 13061-1, 2014: Wood – Physical and mechanical properties of wood. Test methods for small clear wood specimens. Part 1: Determination of moisture content for physical and mechanical tests. International Organization for Standardization, Geneva.
 23. ***ISO 13061-2, 2014: Wood – Physical and mechanical properties of wood. Test methods for small clear wood specimens. Part 2: Determination of density for physical and mechanical tests. International Organization for Standardization, Geneva.
 24. ***ISO 18122, 2022: Solid biofuels – Determination of ash content. International Organization for Standardization, Geneva.

Corresponding address:

JAVIER RAMÓN SOTOMAYOR CASTELLANOS

Michoacán University of San Nicolás de Hidalgo, Avenida Francisco J. Múgica s/n, Morelia, Michoacán, MÉXICO, e-mail: javier.sotomayor@umich.mx