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PRELIMINARY EVALUATION OF COPPER LEACHING FROM COMMERCIALLY AVAILABLE WOOD SPECIES IN JAPAN TREATED WITH COPPER AZOLE WOOD PRESERVATIVE

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ABSTRACT

This study examined copper fixation and leaching in wood treated with copper azole (CuAz) and copper sulfate ($CuSO_4$), emphasizing the effects of wood species and post-treatment conditions. Five Japanese softwoods, including Sugi, Hinoki, and Douglas fir, were assessed under three conditions: unfixed, ambient-fixed, and heat-treated (60°C for 3 days). Copper fixation was defined as the proportion of non-leachable copper remaining after standardized leaching, with release rates measured via leachate analysis. Results showed that post-treatment condition is the dominant factor controlling copper stabilization. Heat treatment significantly enhanced fixation and reduced leaching across all species and preservative types, indicating improved bonding between copper ions and wood components. Ambient fixation was often insufficient. Sugi sapwood showed the highest leaching, while Hinoki heartwood exhibited the greatest retention. Overall, CuAz outperformed $CuSO_4$, underscoring the importance of formulation in improving copper stability and durability.

KEYWORDS: Copper, leaching, fixation, Cu azole, $CuSO_4$.

INTRODUCTION

Leaching of wood preservatives is a complex, multistep process involving chemical fixation to the wood matrix, mobilization into the aqueous phase, and transport through the porous wood structure (Lupsea et al. 2013). Understanding these mechanisms is crucial for life-cycle management and designing safer preservative systems. Historically, chromated copper arsenate (CCA) enhanced biological resistance, but risks associated with arsenic and chromium leaching led to regulatory restrictions in North America and Europe in the early 2000s (Yıldız et al. 2004; Temiz et al. 2006; Barbero-López et al. 2021; Morais et al. 2021; Başkal et al. 2023). This prompted the development of arsenic- and chromium-free alternatives based on copper, azoles, quaternary ammonium compounds, and boron (Freeman and McIntyre 2008; Hasan et al. 2010; Kartal et al. 2015; Teleginski et al. 2016). Copper amine–based preservatives provide long-term protection comparable to CCA (Evans 2003; Westin et al. 2010; Temiz et al. 2014; Oh and Kim 2020); however, higher copper loadings often result in poor fixation and increased leaching (Eisler 1998; Tascioglu et al. 2005, 2008; Ung and Cooper 2005; Temiz et al. 2006). Inadequate fixation is attributed to the predominance of copper–amine complexes and limited reactive binding sites in wood, leading to incomplete precipitation of insoluble copper (Zhang and Kamdem 2000a,b; Jiang and Ruddick 2004; Lee and Cooper 2010a,b; Lee and Cooper 2012; Oh et al. 2025).

Wood species strongly influence copper uptake, fixation, and leaching due to variations in anatomy, permeability, chemical composition, and pH. Comparative studies of alkaline copper quaternary (ACQ), copper azole (CuAz), and related systems demonstrate pronounced species-dependent variations in copper retention and loss (Humar et al. 2007; Lin et al. 2009; Bakir et al. 2022; Başkal et al. 2023). For instance, copper leaching from copper–ethanolamine preservatives was lower in Norway spruce (*Picea abies*) than in beech (*Fagus sylvatica*), a trend linked to differences in lignin reactivity, hemicellulose content, and ethanolamine-induced lignin depolymerization (Humar et al. 2007; Humar and Žlindra 2007). In copper–boron–amine systems, copper release reflects a pH-dependent competition between fixation to lignin/hemicellulose and complexation with extractives in the leachate (Lupsea et al. 2013). Broadly, variations in lignin structure, hemicellulose composition, cellulose crystallinity, extractives, and intrinsic wood pH govern species-specific preservative behavior (Kartal 1999; Kartal and Lebow 2001; Hwang et al. 2007; Kartal et al. 2007; Lupsea et al. 2013; Ali and Hashim 2019).

In Japan, Sugi (*Cryptomeria japonica*), Hinoki (*Chamaecyparis obtusa*), Western red cedar, and Douglas-fir are primary structural species, with CuAz preservatives commonly applied for outdoor durability. Studies on treated softwoods indicate that Japanese larch (*Larix kaempferi*) often fails to meet target copper retentions and exhibits high leaching, likely because resinous tracheids and extractives interfere with copper penetration and binding (Lin et al. 2009). Differences between sapwood and heartwood further complicate performance, as heartwood contains naturally durable extractives that alter fixation and precipitation (Schultz and Nicholas 2002; Taylor et al. 2002; Venkatasamy 2005; Hwang et al. 2007). Structural anomalies like compression wood also influence behavior; for example, compression wood accelerates chromium reduction and enhances copper binding in CCA-treated wood due to a higher availability of lignin-associated reactive sites (Kartal and Lebow 2001).

Despite these advances, limited research has systematically integrated species-specific wood chemistry, particularly for commercially important Japanese species with preservative formulation and post-treatment conditions. While native species like Sugi (*Cryptomeria japonica*) and Hinoki (*Chamaecyparis obtusa*) are heavily utilized or exported due to their abundance and heartwood durability, structural limitations like Sugi's relatively low modulus of elasticity necessitate the concurrent use of imported species like Douglas-fir, especially in laminated structural products (Forestry Agency 2024; Japan Forestry Agency 2025). Consequently, understanding how these distinct wood materials affect preservative fixation remains critical. To mitigate leaching, researchers have explored reformulation and post-treatment conditioning, including adjusting copper proportions, modifying Cu–amine ratios, and applying thermal post-treatments (Pankras et al. 2009; Yu et al. 2009, 2010; Ye and Morrell 2015). Processes such as hot-air curing, steaming, microwave heating, and hot-water treatments significantly enhance copper fixation (Barnes 1988; Cao and Kamdem 2004; Ung and Cooper 2005; Cao and Yu 2007; Kang et al. 2008; Yu et al. 2009a). However, conditions must be carefully controlled, as excessive temperatures can promote lignin depolymerization and counteract fixation, particularly in copper–ethanolamine systems (Humar and Žlindra 2007). Optimized hot-air post-treatments near 100°C shorten stabilization time and reduce copper leaching in ACQ- and CuAz-treated softwoods without compromising biological efficacy (Oh and Kim 2020; Oh et al. 2025).

The present study evaluates the effects of wood species and post-treatment temperature-humidity-time schedules on copper fixation and leaching from CuAz-treated wood. By linking leaching performance to wood material properties and processing variables, this work aims to support the development of species-adapted CuAz treatment and conditioning strategies that enhance durability while minimizing copper leaching.

MATERIAL AND METHODS

Preparation of wood specimens

Wood specimens measuring 20 mm (T) x 20 mm (R) x 10 mm (L) were cut from sapwood and heartwood portions of Sugi wood (*Cryptomeria japonica* D. Don). The study employed hinoki heartwood (*Chamaecyparis obtusa* (Siebold and Zucc.) Endl.), Western red cedar heartwood (*Thuja plicata* Donn ex D. Don), and Douglas fir heartwood (*Pseudotsuga menziesii* (Mirbel) Franco) test specimens as well. Before copper treatments, all wood specimens were conditioned at 20°C and 65% relative humidity (RH) for two weeks. The specimens were free of knots, and visible concentration of resins, and showed no visible evidence of infection by mold, stain, or wood-destroying fungi. All specimens were numbered and weighed to the nearest 0.001 g before preservative treatments. Air-dry densities of wood specimens were determined according to slightly modified ASTM D 2395-07 standard test method by employing twelve test specimens.

Procedures for preservative treatment, conditioning and leaching

CuAz wood preservative (Tanalith® CY, Arxada, Tokyo, Japan) (Tab. 1) was obtained from Dainihon Wood Preserving Co., Ltd., Nagoya City, Japan. CuAz solution was diluted 89-fold with distilled water for the treatments of Sugi sapwood, and 45-fold for the other wood species used to achieve comparable target copper retentions, accounting for species-dependent permeability differences. Sugi sapwood exhibits significantly higher permeability; thus, a higher dilution was required to avoid excessive uptake. For comparisons, anhydrous $CuSO_4$ (Copper (II) sulphate (Code: 09607-55) - Nacalai Tesque, Inc., Kyoto, Japan) (Tab. 1) was used and 0.26% and 0.52% solution concentrations were prepared with distilled water for Sugi sapwood and the other wood species used, respectively. These concentrations were selected based on preliminary trials and literature values to produce retention levels within a comparable range to CuAz-treated specimens.

Tab. 1: Information on CuAz and $CuSO_4.5H_2O$ wood preservatives.

Preservative	Commercial name	Producer	Composition (%) (w/w), properties
Cu-Azole	Tanalith® CY	Arch Wood Protection (M) Sdn Bhd Kuala Lumpur, Malaysia	11.6 copper oxide (CuO) (Cupric carbonate ethanolamine complex); 0.136 cyproconazole. Tanalith® CY concentrate is a dark blue viscous liquid with an amine odor. Diluted solution is an opaque blue liquid with a faint amine odor. Treated timber has an aesthetically pleasing green coloration, the surface of the treated timber is clean, non-oily, and non-greasy.
$CuSO_4.5H_2O$	Copper(II) sulfate pentahydrate	Nacalai Teqque Inc., Kyoto, Japan	Copper (Cu): 25.45%. It is blue crystallin solid, odorless. It imparts the wood blue to greenish-blue tint. Over time the color can fade to green or brown as copper reacts with wood components or oxides.

The wood specimens were treated with the prepared copper solutions stated above under vacuum. In the treatments, low-pressure cycle consisted of a 60 min vacuum (88 kPa absolute pressure) in a treatment desiccator followed by soaking the specimens in the same treatment solution at ambient conditions for 16 h. After the treatments, specimens were re-weighed to determine the uptake solution retention in the specimens. Ten test specimens were employed for each wood species, treatment concentrations, and post-treatment conditions.

Treated wood specimens were immediately subjected to three different post-treatment conditions as shown below: *(1)* No fixation, *(2)* Conditioning at 60ºC for 3 days, *(3)* Conditioning at ambient conditions, 22ºC and 65% relative humidity (RH) for 2 weeks.

Leaching process was similar to Japanese Industrial Standard (JIS) K 1571 (2010) to reproduce the effects that would occur over time in service by using more aggressive conditions. The process involved immersing three wood specimens in 120 mL distilled water, stirring with a magnetic stirrer (400 to 450 rpm) at 27ºC for 8 h followed by drying at 60°C for 16 h. This cycle was repeated 5 times. After each weathering cycle, leachate was collected and leaching water was renewed. All leachates for each treatment and replicate were cumulatively collected in the same jar for further analyses. Six test specimens for each wood species, treatment solutions and post-treatments conditions were leached in two separate groups, in triplicate. Remaining four untreated test specimens out of ten treated ones were used for background Cu levels in the specimens.

Determination of copper content in leachates and wood

Copper content was determined by ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy, Optima 8300 ICP-OES, PerkinElmer) directly in the leachates collected in the leaching tests. All leachates were passed through a hydrophilic PTFE filter (ϕ13 mm/ ϕ0.45 µm, ADVANTEC) before the ICP analysis.

For ICP analyses of treated wood specimens, three leached and unleached specimens from each treatment group were ground to pass a US standard 40-mesh screen. Ground wood specimens were microwave-digested using the AWPA A7-19 (2021) standard method with slight modifications. The specimens (0.5 g) were weighed into a microwave digestion tube and nitric acid (15 mL) was added. A digestion blank was prepared along with the specimens as a control. The digestion tubes were sealed into digestion vessels and placed into a carousel in a microwave oven for a pre-programmed digestion sequence. After the digestion was complete (20 min), each vessel was brought up to 50 mL volume with DI water.

The digested specimens were analyzed using a Perkin Elmer ICP-OES according to the AWPA A21-16 (2021) standard method to determine the percentage of copper release, and the retention levels of copper solutions after leaching. The percent reduction of copper element in the treated and leached wood specimens was calculated based on the initial amount of the elements in the specimens before leaching.

RESULTS AND DISCUSSION

The air-dry density of the five investigated wood types, representing indigenous Japanese softwoods and imported North American species, varied substantially, ranging from 348 kg/m³ for Sugi heartwood to 521 kg/m³ for Douglas fir heartwood (Tab. 2).

Tab. 2: Air-dry densities of wood species used in the study.

Wood species	Air-dry density (kg/m^3)
Sugi sapwood	371 (6)
Sugi heartwood	348 (12)
Hinoki heartwood	469 (18)
Western red cedar heartwood	353 (8)
Douglas fir heartwood	521 (24)

Values in parentheses are standard deviations; n: 12.

Douglas fir and hinoki exhibited notably higher densities than Sugi and Western red cedar, reflecting fundamental differences in anatomical structure and cell-wall composition. Sugi sapwood showed slightly higher density than Sugi heartwood, although both remained within the low-density range typical of lightweight softwoods. These density differences are relevant for preservative performance, as density and associated permeability strongly influence preservative uptake, diffusion, and subsequent fixation reactions. Species with higher density and lower permeability, such as Douglas fir heartwood, often exhibit reduced penetration and less uniform copper distribution, which can ultimately affect fixation efficiency and leaching behavior, as reported previously for several softwood species (Lin et al. 2009). They found differences between ACQ and CuAz wood preservatives among wood species were pronounced. Japanese cedar (*Cryptomeria japonica*), Taiwania (*Taiwania cryptomerioides*), and China fir (*Cunninghamia lanceolata*) generally exhibited higher copper retention than Western hemlock and Japanese larch for both ACQ and CuAz treatments, before and after leaching. Japanese larch consistently showed the lowest copper retention, reflecting species-related differences in permeability, density, and wood chemistry that limit copper uptake and fixation. When comparing preservative systems, ACQ-treated wood generally showed slightly higher copper retention than CuAz-treated wood at equivalent concentrations, both before and after leaching, across most species (Lin et al. 2009).

Tab. 3 gives the copper content in unleached and leached specimens treated with CuAz and $CuSO_4$ wood preservatives, respectively. Differences in initial copper content among post-treatment conditions are attributed to variability in solution uptake and test specimens, redistribution during fixation, and experimental variability due to limited replicates. Figs. 1 and 2 illustrate Cu element content in the leachates collected in the leaching tests of Cu Azole (CuAz) and $CuSO_4$-treated wood and percentage leaching of copper element from treated wood with either Cu Azole (CuAz) or $CuSO_4$ solutions, respectively. Copper leaching from treated wood was strongly influenced by preservative formulation, wood species, tissue type, and post-treatment fixation conditions. Across all species and fixation regimes, CuAz-treated specimens consistently exhibited lower copper leaching than those treated with $CuSO_4$, indicating superior fixation stability of azole-containing systems. This finding is consistent with previous studies showing that modern copper-based formulations with organic co-biocides and amine complexes enhance copper–wood interactions relative to simple inorganic copper salts (Lupsea et al. 2013; Oh and Kim 2020). Lupsea et al. (2013) found that preservative treatment alters the wood matrix, particularly in systems containing copper and amines, where fixation involves not only the binding of Cu–amine complexes but also chemical modification of wood components through reactions with amines. Wood treated with copper–boron–azole (CBA) formulations was found to release various nitrogen-containing compounds, likely arising from excess amines and their interactions with wood polymers. These reactions are influenced by the type of amine, the Cu-to-amine ratio, and the pH conditions of both the preservative solution and the treated wood. Despite fixation, copper release remained relatively high compared with its initial content. Copper was shown to bind primarily to carboxyl and phenolic functional groups and to form soluble complexes with wood extractives.

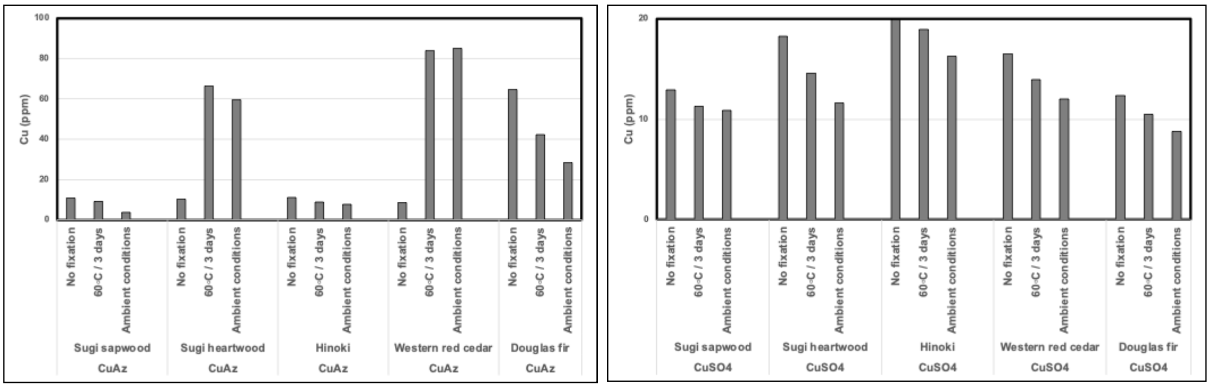
Tab. 3: Cu content (kg/m³) in wood specimens before and after leaching.

Wood type	Fixation conditions	CuAz treatments		$CuSO_4$ treatments	
		Before leaching	After leaching	Before leaching	After leaching
Sugi sapwood	No fixation	3.230	2.077	1.557	0.762
	60∘C / 3 days	3.662	2.691	1.471	0.815
	Ambient conditions	3.264	2.986	1.435	0.852
Sugi heartwood	No fixation	2.389	2.188	1.574	1.089
	60∘C / 3 days	2.937	2.620	1.983	1.403
	Ambient conditions	3.208	3.087	2.320	1.587
Hinoki heartwood	No fixation	3.391	1.855	1.211	0.432
	60∘C / 3 days	3.135	2.777	1.197	0.478
	Ambient conditions	3.068	2.997	1.166	0.651
Western red cedar heartwood	No fixation	3.184	1.767	1.119	0.701
	60∘C / 3 days	3.319	3.044	1.085	0.738
	Ambient conditions	3.654	3.385	1.073	0.724
Douglas fir heartwood	No fixation	2.543	2.086	1.120	0.506
	60∘C / 3 days	3.769	3.436	1.111	0.629
	Ambient conditions	3.031	2.840	1.088	0.777

Optimized hot-air post-treatments near 100°C have been reported to reduce copper leaching (Oh and Kim 2020; Oh et al. 2025); however, excessive temperatures may also degrade wood polymers or alter fixation chemistry. Therefore, moderate-temperature post-treatment conditions were selected in this study to balance fixation enhancement and material stability. Post-treatment conditions had a pronounced effect on copper stabilization. Heat post-treatment at 60°C for 3 days considerably reduced copper leaching compared with unfixed or ambient-cured specimens, particularly for $CuSO_4$-treated wood. This observation confirms that freshly treated wood contains a substantial fraction of loosely bound water-soluble copper that requires time and energy input to become immobilized. Thermal fixation accelerates diffusion of copper ions into the cell wall and promotes reactions with lignin, hemicelluloses, and extractives, leading to the formation of less soluble copper complexes. Similar kinetically controlled fixation behavior has been reported for non-chromium copper systems, where copper immobilization continues over time but proceeds slowly at ambient temperatures (Ung and Cooper 2005; Lupsea et al. 2013).

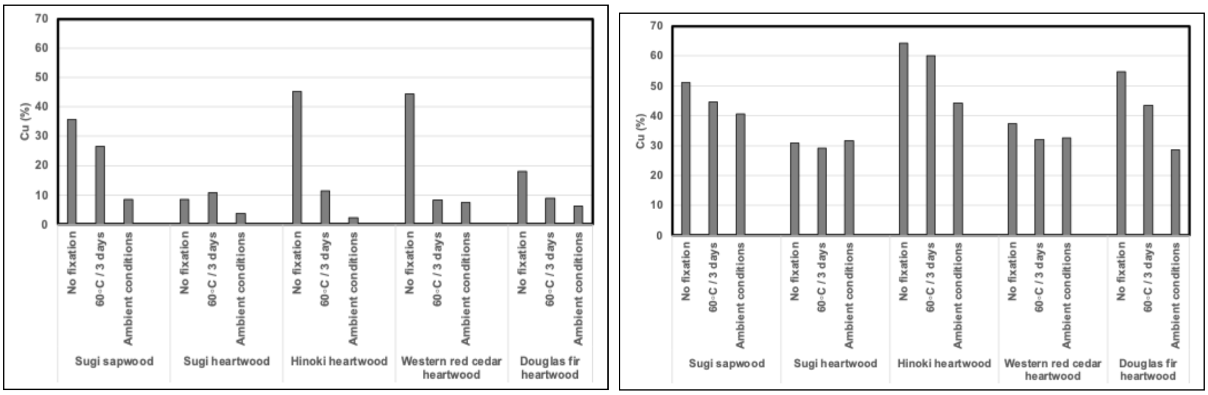
Wood species and tissue type exerted a dominant influence on copper leaching in the study. Sugi sapwood consistently showed the highest copper release for both CuAz and $CuSO_4$ treatments, regardless of fixation condition. This behavior can be attributed to its higher permeability, possible lower extractive content, and limited availability of strong copper-complexing sites, resulting in weaker fixation. In contrast, extractive-rich heartwoods, particularly hinoki and Western red cedar, exhibited substantially lower copper leaching, while Douglas fir heartwood showed intermediate behavior. These results align with previous findings that heartwoods rich in phenolic compounds and lignin-associated functional groups immobilize copper more effectively through complexation and precipitation reactions (Guo et al. 2008; Lin et al. 2009). The beneficial effect of elevated-temperature fixation was most pronounced in species with inherently high copper leaching, such as Sugi sapwood. Although thermal fixation remarkably reduced copper losses in this tissue, leaching levels remained higher than those observed for extractive-rich heartwoods, indicating that intrinsic wood chemistry ultimately constrains fixation efficiency. This interaction between wood species and fixation conditions highlights the need to consider species-specific characteristics when developing predictive models for copper leaching, rather than relying solely on preservative formulation or retention level.

The observed differences between CuAz and $CuSO_4$ treatments can be explained by their distinct fixation mechanisms. Copper fixation in wood is generally described as a combination of chemisorption and physical precipitation, with the dominant pathway dependent on preservative chemistry and environmental conditions (Lupsea et al. 2013). In MEA-based systems, copper exists as mono-, di-, and neutral Cu–MEA complexes, which interact with carboxyl and phenolic hydroxyl groups in wood polymers to form surface complexes (Jiang 2000; Ravat et al. 2000; Zhang and Kamdem 2000a,b; Lee 2011). The fixation of these complexes is strongly pH dependent, with ligand-exchange reactions dominating at alkaline pH and cation-exchange mechanisms prevailing under moderately alkaline conditions (Craciun et al. 1997; Thomason and Pasek 1997; Lee and Cooper 2010a, 2010b). Azole-containing systems further benefit from the formation of relatively stable copper–azole associations, which reduce copper solubility and leachability compared with simple copper salts. Although direct complexation between tebuconazole and copper in wood remains poorly documented, *in vitro* studies have demonstrated the formation of copper–azole complexes with defined stoichiometries (Dytrtova et al. 2011; Evans and Schmalzl 2007; Evans and Schmalzl 2008). These interactions likely contribute to the lower leaching observed for CuAz-treated wood in the present study.



(a) (b)

Fig. 1: Content in the leachates collected in the leaching tests of Cu Azole (CuAz) (a) and $CuSO_4$-treated wood (b) (average of two replicate specimens).



(a) (b)

Fig. 2: Percentage leaching of copper element from treated wood with either Cu Azole (CuAz) (a) or $CuSO_4$ solutions (b) (average of two replicate specimens).

Recent literature provides additional context for the present findings. Başkal et al. (2023) reported that CuAz-treated wood exhibited higher copper leaching than ACQ and CCB under soil-contact conditions, particularly at higher concentrations and longer leaching periods, highlighting the relatively greater mobility of copper in chromium-free systems. Similarly, Oh and Kim (2020) demonstrated that copper leaching from CuAz and ACQ is strongly pH dependent and substantially higher under acidic conditions, whereas chromium-containing preservatives, such as CCA, exhibit superior fixation stability. These studies reinforce the conclusion that, although CuAz performs better than simple copper salts, chromium-free systems remain more sensitive to environmental conditions and post-treatment protocols.

Reduced copper leaching is essential for both long-term biological efficacy and environmental risk mitigation. Excessive copper loss diminishes decay resistance and increases copper release into surrounding soils and aquatic systems. The present results demonstrate that appropriate selection of wood species, combined with optimized post-treatment fixation, particularly moderate thermal curing, can substantially reduce copper leaching from treated wood. While CuAz formulations clearly outperform $CuSO_4$ in terms of fixation stability, effective post-treatment remains critical to minimize copper release, especially for permeable sapwoods and refractory species. Overall, these findings support the continued use of CuAz as an environmentally preferable alternative to chromium-containing preservatives, provided that fixation is carefully optimized and species-specific characteristics are taken into account.

CONCLUSIONS

A substantial proportion of copper in freshly treated wood remained weakly bound and water-soluble, underscoring the necessity of effective post-treatment fixation. Among the evaluated regimes, moderate heat treatment at 60°C for 3 days was most effective in enhancing copper fixation and reducing leaching, demonstrating that fixation in non-chromium copper systems is largely kinetically controlled. Sapwood, particularly Sugi sapwood, consistently showed lower fixation and higher leaching than corresponding heartwood. In contrast, hinoki heartwood exhibited superior fixation and minimal leaching. Western red cedar and Douglas fir showed comparatively greater copper release. Preservative formulation was a key determinant of leaching behavior. CuAz-treated wood consistently outperformed $CuSO_4$-treated wood, exhibiting higher fixation and lower copper release under all conditions. Overall, reduced copper leaching is essential for maintaining long-term preservative efficacy and minimizing environmental risks. Optimized post-treatment strategies, particularly controlled heat fixation, are critical for improving the environmental performance of copper-based preservatives.

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