

INVESTIGATION ON BLEACHING APPLICATIONS WITH $C_2H_2O_4$ AND $H_2O_2 + NaOH$ CHEMICALS ON IROKO (*Milicia excelsa* Welw. C.C. Berg) WOOD

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Abstract:

*In this study, selected surface characteristics (color parameters, glossiness, and whiteness index: WI^*) of iroko (*Milicia excelsa* Welw. C.C. Berg) wood were investigated following bleaching treatments using two different chemicals: oxalic acid ($C_2H_2O_4$) and hydrogen peroxide + sodium hydroxide ($H_2O_2 + NaOH$). The surfaces that underwent bleaching treatment were compared with surfaces that did not undergo bleaching. The obtained results were analyzed using an SPSS program. All tests yielded significant findings in their variance analyses. The application of both bleaching chemicals to wooden surfaces at 60 and 85 degrees led to decreased glossiness levels in both perpendicular and parallel fiber orientations. Furthermore, decreases were noted in the measurements of WI^* , a^* , C^* , and b^* values. Furthermore, decreases were noted in the measurements of a^* , C^* , and b^* values. When subjected to the $C_2H_2O_4$ chemical, the ΔE^* values were determined as 2.20, whereas they measured 4.09 under exposure to the $H_2O_2 + NaOH$ chemical. The Δa^* , Δb^* , and ΔC^* values were identified as negative. The utilization of chemicals led to changes in the surface characteristics of the wooden material.*

Key words: NaOH, bleaching, color, $C_2H_2O_4$, glossiness, iroko, whiteness index, H_2O_2 .

INTRODUCTION

All types of wood are made up of cellulose, lignin, ash-forming minerals, and extractives within a cellular structure. Variations in the properties and amounts of these four components, along with differences in cellular structure, result in some woods being heavy while others are light, some being stiff while others are flexible, and some being hard while others are soft. Although the properties are generally consistent within certain limits for a particular species, selecting wood based solely on the species can sometimes be sufficient. However, to fully utilize the benefits of wood and effectively use it in engineering applications, the impact of specific characteristics or physical properties must be carefully considered (Forest Products Laboratory 1987).

Bleaching and removing color can happen through various processes, like breaking apart conjugated chains, removing double bonds in the chain, or oxidizing different parts of the chain. These reactions lead to an energy gap increase between the molecule's ground and excited states, enabling it to absorb UV light and not generate color. Additionally, bleaching can enhance the water solubility of organic compounds post-reaction (Farr et al. 2003).

Bleach concentration plays a crucial role in evaluating bleaching effectiveness. Excessive concentration results in wastage, whereas insufficient concentration impacts bleaching results. Moreover, the brightness of bleaching is directly affected by bleach concentration (Hemmasi 2017).

Bleaching procedures typically utilize a range of chemicals, such as oxalic acid, hydrogen peroxide, sodium hydroxide, and others. Oxalic acid, a toxic crystalline organic compound, is frequently employed as a purifying agent in pharmaceuticals, a precipitating agent in the processing of rare earth metals, and serves as a bleaching agent in the textile and wood sectors (Osoka et al. 2019). Hydrogen peroxide is a frequently employed eco-friendly bleaching substance for whitening stains on textiles, wood pulp, raw cotton, paper, and hard surfaces (Kirk et al. 1991).

In the literature, various bleaching studies have been conducted on different types of wood using different bleaching chemicals [bulletwood (*Manilkara bidentata* (A.DC.) A. Chev.) (Peker et al. 2023), ayous (*Triplochiton scleroxylon*), linden (*Tilia* spp.), poplar (*Populus* L.) (Lu et al. 2023), birch (*Betula pendula*) (Yamamoto et al. 2017), Maritime pine (*Pinus pinaster*) (Mehats et al. 2021), Japanese larch (*Larix kaempferi*), Mongolia oak (*Quercus mongolica*) (Park et al. 2022), birch (*Betula platyphylla* Suk.) (Liu et al. 2015), linden (*Tilia tomentosa* - Moench.) (Çamlıbel and Ayata 2023), bamboo (*Indosasa angustata*) (Nguyen et al. 2019), silver birch (*Betula pendula* Roth.) (Mononen et al. 2005)]. These studies have indicated that the color characteristics (b^* , h^0 , L^* , C^* , and a^*) of wooden materials were altered following bleaching treatments.

This research was conducted to examine the changes in surface properties between iroko wood and bleaching applications.

OBJECTIVE

This study examined the surface attributes of iroko (*Milicia excelsa* Welw. C.C. Berg) wood after undergoing bleaching procedures with two distinct chemicals: oxalic acid and a combination of hydrogen peroxide and sodium hydroxide. The aim is to analyze how the obtained results interact with wood materials during the process of color alteration after bleaching, aiming to offer fresh perspectives to producers and consumers alike.

MATERIALS

Samples of iroko wood (*Milicia excelsa* Welw. C.C. Berg) were obtained with dimensions of 100 x 200 x 20mm. The preparation of these samples followed the TS ISO 13061-1 (2021) standard. During the research, both single-component and dual-component chemicals were employed. The single-component consisted of oxalic acid ($C_2H_2O_4$), which is a colorless and odorless liquid with a pH value of 2.0 ± 0.5 . On the other hand, the double-component solution had a pH value of 7 and was also liquid, colorless, and odorless. This solution was soluble in water and comprised hydrogen peroxide (H_2O_2) as component A and sodium hydroxide (NaOH) as component B, with a ratio of 2:1. Ten samples were used per group.

METHODS

The chemicals were applied to the wooden material surfaces using a sponge with the smear technique, applying a single layer. The experimental samples that underwent bleaching treatments were compared with the control samples that did not undergo bleaching. Different tests were conducted in this comparison. Glossiness assessments were carried out on wooden surfaces utilizing an ETB-0833 model gloss meter device, measuring at three distinct angles (20°, 60°, and 85°), both perpendicular and parallel to the wood fibers, adhering to the ISO 2813 (1994) standard. Whiteness index (W^*) values were determined in both parallel and perpendicular orientations to the wood fibers, following the guidelines outlined in ASTM E313-15e1 (2015). Color alterations were evaluated employing a CS-10 Portable Colorimeter manufactured by CHN Spec, China, adhering to the ASTM D 2244-3 (2007) standard. The comparison criteria for the visual evaluation of the calculated ΔE^* color difference are provided in Table 1 according to DIN 5033 (1979).

Table 1

Comparison criteria for ΔE^* evaluation (DIN 5033 1979)

Visual	Total Color Difference	Visual	Total Color Difference
Undetectable	<0.20	Very Distinct	3.00 - 6.00
Very Weak	0.20 - 0.50	Strong	6.00 - 12.00
Weak	0.50 - 1.50	Very Strong	> 12.00
Distinct	1.50 - 3.00		

The definitions of Δb^* , Δa^* , ΔC^* , and ΔL^* are presented in Table 2 based on Lange (1999). In the existing literature, ΔC^* denotes the difference in chroma or saturation, whereas ΔH^* signifies the disparity in hue or shade, as established by Lange in 1999.

Table 2

The definitions of Δb^*, Δa^*, ΔC^*, and ΔL^* (Lange 1999)		
Test	Positive Description	Negative Description
Δa^*	More red than the reference	More green than the reference
ΔL^*	Lighter than the reference	Darker than the reference
Δb^*	More yellow than the reference	More blue than the reference
ΔC^*	Clearer, brighter than the reference	More dull, matte than the reference

The results of total color differences were determined using the following formulas.

$$\begin{aligned} \Delta H^* &= [(\Delta E^*)^2 - (\Delta L^*)^2 - (\Delta C^*)^2]^{1/2} & (1) \\ \Delta L^* &= [L^*_{\text{treated with bleach solution}}] - [L^*_{\text{not treated with bleach solution}}] & (2) \\ \Delta b^* &= [b^*_{\text{treated with bleach solution}}] - [b^*_{\text{not treated with bleach solution}}] & (3) \\ \Delta a^* &= [a^*_{\text{treated with bleach solution}}] - [a^*_{\text{not treated with bleach solution}}] & (4) \\ \Delta E^* &= [(\Delta L^*)^2 + (\Delta b^*)^2 + (\Delta a^*)^2]^{1/2} & (5) \\ \Delta C^* &= [C^*_{\text{treated with bleach solution}}] - [C^*_{\text{not treated with bleach solution}}] & (6) \\ C^* &= [(a^*)^2 + (b^*)^2]^{1/2} & (7) \\ h^0 &= \arctan [b^*/a^*] & (8) \end{aligned}$$

Homogeneity groups, standard deviations, mean values, variance analyses, maximum and minimum values, and percentage (%) change rates were computed utilizing a statistical software package.

RESULTS AND DISCUSSION

According to the results of the multivariate analysis of variance presented in Table 3, it is observed that colour parametres are found to be significant for the type of bleaching chemical (Table 3).

Table 3

Results of the variance analysis for color parameters					
Bleaching Chemical Type					
Test	Sum of Squares	df	Mean Square	F value	Sig.
L^*	98.637	2	49.318	335.377	0.000*
a^*	9.491	2	4.746	142.876	0.000*
b^*	46.392	2	23.196	31.420	0.000*
C^*	30.769	2	15.384	176.265	0.000*
h^0	77.532	2	38.766	225.565	0.000*
Error					
Test	Sum of Squares	df	Mean Square		
L^*	3.970	27	0.147		
a^*	0.897	27	0.033		
b^*	19.933	27	0.738		
C^*	2.357	27	0.087		
h^0	4.640	27	0.172		
Total			Corrected Total		
Test	Sum of Squares	df	Test	Sum of Squares	df
L^*	86449.063	30	86449.063	102.607	29
a^*	2187.930	30	2187.930	10.388	29
b^*	16734.086	30	16734.086	66.325	29
C^*	18657.835	30	18657.835	33.125	29
h^0	146893.233	30	146893.233	82.173	29

*: Significant

The measurement outcomes for color parameters are presented in Table 4.

Table 4

Results obtained for color parameters

Test	Bleaching Chemical Type	Mean	Change Ratio (%)	HG	SD	Minimum	Maximum	COV
L^*	Control (no treatment)	54.06	-	B	0.24	53.65	54.40	0.45
	$C_2H_2O_4$	55.63	↑2.90	A*	0.36	54.99	56.02	0.66
	$H_2O_2 + NaOH$	51.25	↓5.20	C**	0.50	50.54	52.05	0.98
a^*	Control (no treatment)	9.08	-	A*	0.23	8.78	9.49	2.58
	$C_2H_2O_4$	7.75	↓14.65	C**	0.14	7.42	7.89	1.80
	$H_2O_2 + NaOH$	8.72	↓3.96	B	0.16	8.52	9.01	1.81
b^*	Control (no treatment)	24.81	-	A*	1.44	23.94	28.83	5.80
	$C_2H_2O_4$	24.03	↓3.14	A	0.27	23.56	24.38	1.11
	$H_2O_2 + NaOH$	21.87	↓11.85	B**	0.27	21.40	22.30	1.23
C^*	Control (no treatment)	25.96	-	A*	0.37	25.40	26.64	1.44
	$C_2H_2O_4$	25.25	↓2.73	B	0.26	24.79	25.62	1.01
	$H_2O_2 + NaOH$	23.54	↓9.32	C**	0.24	23.14	23.96	1.01
h^0	Control (no treatment)	69.51	-	B	0.33	68.97	70.09	0.47
	$C_2H_2O_4$	72.11	↑3.74	A*	0.36	71.79	72.98	0.50
	$H_2O_2 + NaOH$	68.25	↓1.81	C**	0.53	67.24	68.91	0.78
COV: Coefficient of Variation, *: Highest Result, **: Lowest Result, SD: Standard Deviation, HG: Homogeneity Group, Number of Measurements: 10								

The highest result for the L^* parameter is observed in samples treated with $C_2H_2O_4$ bleaching chemical (55.63), while the lowest result is obtained on samples with $H_2O_2 + NaOH$ bleaching chemical (51.25). Regarding the L^* value, there is an increase of 2.90% in samples treated with $C_2H_2O_4$ bleaching chemical, whereas a decrease of 5.20% is observed in samples subjected to $H_2O_2 + NaOH$ application (Table 4).

The control experiment samples yielded the highest result for the a^* parameter (9.08), whereas the lowest result was recorded in experiment samples treated with $C_2H_2O_4$ bleaching agent (7.75). Regarding the a^* value, there were reductions of 14.65% with $C_2H_2O_4$ bleaching agent and 3.96% with $H_2O_2 + NaOH$ bleaching agent (as shown in Table 4).

In their findings, the authors noted reductions in the a^* parameter after applying bleaching treatments with oxalic acid and hydrogen peroxide + sodium hydroxide chemicals on black locust (*Robinia pseudoacacia* L.) (Peker and Ulusoy 2023) wood species.

The b^* parameter exhibited its highest value in the control experiment samples (24.81), whereas the lowest value was observed in samples treated with $H_2O_2 + NaOH$ bleaching chemical (21.87). In terms of the b^* value, reductions of 11.85% were recorded with $H_2O_2 + NaOH$ bleaching agent, while a decrease of 3.14% was noted with $C_2H_2O_4$ bleaching agent (as indicated in Table 4).

The lowest result for the C^* value is obtained in experiment samples exposed to $H_2O_2 + NaOH$ bleaching chemical (23.54), while the highest result is determined in control experiment group samples (25.96). Furthermore, reductions of 2.73% with $C_2H_2O_4$ bleaching agent and 9.32% with $H_2O_2 + NaOH$ bleaching agent were observed in terms of the C^* value (Table 4).

The authors reported decreases in the C^* parameter following bleaching treatments using oxalic acid and hydrogen peroxide + sodium hydroxide chemicals on black locust (*Robinia pseudoacacia* L.) (Peker and Ulusoy 2023) wood species.

For the h^0 value, an increase of 3.74% is determined with $C_2H_2O_4$ bleaching agent, whereas a decrease of 1.81% is found with $H_2O_2 + NaOH$ agent. When examining the h^0 value, the highest result is found on samples treated with $C_2H_2O_4$ bleaching chemical (72.11), while the lowest result is observed on samples with $H_2O_2 + NaOH$ application (68.25) (Table 4).

Following bleaching treatments on black locust (*Robinia pseudoacacia* L.) wood, an increase was observed in the h^0 parameter when treated with oxalic acid, whereas a decrease was noted when treated with hydrogen peroxide + sodium hydroxide chemicals (Peker and Ulusoy 2023).

The outcomes derived from employing color formulas to calculate the total color differences are presented in Table 5.

The ΔE^* values were calculated as 2.20 when exposed to $C_2H_2O_4$ chemical and 4.09 when exposed to $H_2O_2 + NaOH$ chemical. The Δb^* , Δa^* , and ΔC^* values were determined as negative (more blue than the reference, more green than the reference, and more dull, matte than the reference, respectively). The ΔH^* values were obtained as 1.37 for the $C_2H_2O_4$ chemical and 1.72 for the $H_2O_2 + NaOH$ chemical, respectively.

The ΔL^* value was found to be positive (lighter than the reference) with the $C_2H_2O_4$ chemical and negative (darker than the reference) with the $H_2O_2 + NaOH$ chemical (Table 5).

Table 5

Results for total color differences

Bleaching Chemical Type	ΔL^*	Δb^*	Δa^*	ΔC^*	ΔH^*	ΔE^*	Color Change Criterion (DIN 5033, 1979)
$C_2H_2O_4$	1.57	-1.33	-0.78	-0.70	1.37	2.20	Distinct (1.5 - 3.0)
$H_2O_2 + NaOH$	-2.81	-0.36	-2.94	-2.41	1.72	4.09	Very Distinct (3.0 - 6.0)

Based on the multivariate analysis of variance results illustrated in Table 6, it can be noted that the glossiness values demonstrate statistical significance concerning the type of bleaching chemical utilized (Table 6).

Table 6

Results of the variance analysis for glossiness values

Bleaching Chemical Type					
Test	Sum of Squares	df	Mean Square	F value	Sig.
$\angle 20^\circ$ glossiness	0.035	2	0.017	11.700	0.000*
$\angle 60^\circ$ glossiness	3.059	2	1.529	98.314	0.000*
$\angle 85^\circ$ glossiness	3.851	2	1.925	73.841	0.000*
$\parallel 20^\circ$ glossiness	0.056	2	0.028	13.500	0.000*
$\parallel 60^\circ$ glossiness	2.150	2	1.075	106.319	0.000*
$\parallel 85^\circ$ glossiness	9.683	2	4.841	193.367	0.000*
Error					
Test	Sum of Squares	df	Mean Square		
$\angle 20^\circ$ glossiness	0.040	27	0.001		
$\angle 60^\circ$ glossiness	0.420	27	0.016		
$\angle 85^\circ$ glossiness	0.704	27	0.026		
$\parallel 20^\circ$ glossiness	0.056	27	0.002		
$\parallel 60^\circ$ glossiness	0.273	27	0.010		
$\parallel 85^\circ$ glossiness	0.676	27	0.025		
Total			Corrected Total		
Test	Sum of Squares	df	Test	Sum of Squares	df
$\angle 20^\circ$ glossiness	0.720	30	0.720	0.075	29
$\angle 60^\circ$ glossiness	35.100	30	35.100	3.479	29
$\angle 85^\circ$ glossiness	8.300	30	8.300	4.555	29
$\parallel 20^\circ$ glossiness	0.880	30	0.880	0.112	29
$\parallel 60^\circ$ glossiness	58.730	30	58.730	2.423	29
$\parallel 85^\circ$ glossiness	21.400	30	21.400	10.359	29

*: Significant

Table 7 displays the measurement outcomes for glossiness values. It was observed that glossiness values decreased in all directions at 60 and 85 degrees with both bleaching chemicals. The lowest glossiness values in all degrees and directions were found on experimental samples treated with $H_2O_2 + NaOH$ chemical (Table 7).

Table 7

Results obtained for glossiness values

Test	Bleaching Chemical Type	N	Mean	Change Ratio (%)	HG	SD	Minimum	Maximum	COV
$\angle 20^\circ$	Control (no treatment)	10	0.16	-	A	0.05	0.10	0.20	32.27
	$C_2H_2O_4$	10	0.18	$\uparrow 12.50$	A*	0.04	0.10	0.20	23.42
	$H_2O_2 + NaOH$	10	0.10	$\downarrow 37.50$	B**	0.00	0.10	0.10	0.00
$\angle 60^\circ$	Control (no treatment)	10	1.40	-	A*	0.15	1.20	1.60	10.65
	$C_2H_2O_4$	10	1.06	$\downarrow 24.29$	B	0.08	1.00	1.20	7.96
	$H_2O_2 + NaOH$	10	0.62	$\downarrow 55.71$	C**	0.13	0.50	0.80	21.23

⊥85°	Control (no treatment)	10	0.86	-	A*	0.28	0.40	1.20	32.52
	C ₂ H ₂ O ₄	10	0.10	↓88.37	B**	0.00	0.10	0.10	0.00
	H ₂ O ₂ + NaOH	10	0.10	↓88.37	B**	0.00	0.10	0.10	0.00
20°	Control (no treatment)	10	0.20	-	A*	0.07	0.10	0.30	33.33
	C ₂ H ₂ O ₄	10	0.18	↓10.00	A	0.04	0.10	0.20	23.42
	H ₂ O ₂ + NaOH	10	0.10	↓50.00	B**	0.00	0.10	0.10	0.00
60°	Control (no treatment)	10	1.72	-	A*	0.12	1.60	1.90	7.15
	C ₂ H ₂ O ₄	10	1.32	↓23.26	B	0.04	1.30	1.40	3.19
	H ₂ O ₂ + NaOH	10	1.07	↓37.79	C**	0.12	0.90	1.20	10.84
85°	Control (no treatment)	10	1.40	-	A*	0.24	1.00	1.60	17.17
	C ₂ H ₂ O ₄	10	0.32	↓77.14	B	0.13	0.20	0.50	41.14
	H ₂ O ₂ + NaOH	10	0.10	↓92.86	C**	0.00	0.10	0.10	0.00
C ₂ H ₂ O ₄ : Oxalic Acid, H ₂ O ₂ + NaOH: Hydrogen Peroxide + Sodium Hydroxide, COV: Coefficient of Variation, *: Highest Result, **: Lowest Result, SD: Standard Deviation, HG: Homogeneity Group, N: Number of Measurements									

Considering the outcomes of the multivariate analysis of variance as presented in Table 8, it becomes evident that there is a statistically significant relationship between the whiteness index (WI^*) values and the type of bleaching chemical employed (Table 8).

Table 8

Bleaching Chemical Type					
Test	Sum of Squares	df	Mean Square	F value	Sig.
Whiteness index ($WI^* \perp$)	37.331	2	18.665	329.819	0.000*
Whiteness index ($WI^* $)	24.645	2	12.322	214.786	0.000*
Error					
Test	Sum of Squares	df	Mean Square		
Whiteness index ($WI^* \perp$)	1.528	27	0.057		
Whiteness index ($WI^* $)	1.549	27	0.057		
Total			Corrected Total		
Test	Sum of Squares	df	Test	Sum of Squares	df
Whiteness index ($WI^* \perp$)	3933.100	30	3933.100	38.859	29
Whiteness index ($WI^* $)	2379.410	30	2379.410	26.194	29

*: Significant

Table 9 presents the measurement outcomes for whiteness index (WI^*) values. WI^* values showed increases on wood surfaces in both directions ($\perp$: 12.28% and $||$: 8.87%) when using C₂H₂O₄ chemical, while decreases were observed ($\perp$: 18.71% and $||$: 12.74%) with the use of H₂O₂ + NaOH chemical. The highest WI^* results were determined in samples exposed to C₂H₂O₄ chemical ($\perp$: 12.28% and $||$: 9.96%), while the lowest results were found on samples treated with H₂O₂ + NaOH chemical ($\perp$: 9.82% and $||$: 7.74%) (Table 9).

Table 9

Results obtained for whiteness index (WI^*) values									
Test	Bleaching Chemical Type	N	Mean	Change Ratio (%)	HG	SD	Minimum	Maximum	COV
$WI^* \perp$	Control (no treatment)	10	12.08	-	A	0.20	11.80	12.40	1.69
	C ₂ H ₂ O ₄	10	12.28	↑1.66	A*	0.27	11.90	12.70	2.20
	H ₂ O ₂ + NaOH	10	9.82	↓18.71	B**	0.23	9.40	10.00	2.39
$WI^* $	Control (no treatment)	10	8.87	-	B	0.26	8.40	9.20	2.96
	C ₂ H ₂ O ₄	10	9.96	↑12.29	A*	0.30	9.40	10.20	3.04
	H ₂ O ₂ + NaOH	10	7.74	↓12.74	C**	0.11	7.60	7.90	1.39
C ₂ H ₂ O ₄ : Oxalic Acid, H ₂ O ₂ + NaOH: Hydrogen Peroxide + Sodium Hydroxide, COV: Coefficient of Variation, *: Highest Result, **: Lowest Result, SD: Standard Deviation, HG: Homogeneity Group, N: Number of Measurements									

CONCLUSION

Statistically significant results were observed in all variance analyses conducted across all tests. The ΔE^* values were determined as 2.20 under the influence of the $C_2H_2O_4$ chemical and 4.09 under the influence of the $H_2O_2 + NaOH$ chemical. Additionally, reductions were observed in the values of a^* , C^* , and b^* . The use of both bleaching chemicals on wooden surfaces at temperatures of 60 and 85 degrees resulted in diminished shine levels in both perpendicular and parallel fiber directions. The use of chemicals resulted in alterations in the surface properties of the wooden material. It is recommended to conduct aging performance tests on both bleached and unbleached materials.

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