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A Study on the Use of Vinegar and Carbonate Mixtures as Color Modifiers on Mulberry (*Morus alba*) Wood

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ABSTRACT

This study investigates changes in some surface properties (color, glossiness, and whiteness index: *WI**) following the application of vinegar (grape and hawthorn) and carbonate mixtures, which are chemicals used in the food industry as color modifiers, on mulberry (*Morus alba*) wood. Following the preparation of the solutions, they were applied separately onto the wooden surfaces using a brush. The untreated and treated surfaces were compared with each other. Variance analyses revealed that the type of solution was found to be significant across all tests. With both prepared solutions, an increase in the *a** values was observed, while decreases were detected in *WI**, *L**, *b**, *C**, and *h*°, as well as in the glossiness values measured at all degrees and directions. The Δ*E** values were determined to be 12.31 for grape vinegar + carbonate solution and 12.92 for hawthorn vinegar + carbonate solution. With the application of both solutions, negative Δ*L**, Δ*b**, and Δ*C** values were obtained, while Δ*a** values were found to be positive. It was observed that the prepared solutions had a color-modifying effect on the wooden material surfaces.

Keywords: Mulberry, carbonate, color, glossiness, vinegar, whiteness index

Dut (*Morus alba*) Ahşabında Renk Değiştirici Olarak Sirke Ve Karbonat Karışımlarından Hazırlanan Çözeltilerin Kullanılması Üzerine Bir Araştırma

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ÖZ

Bu çalışma, dut (*Morus alba*) ahşabında renk değiştirici olarak gıda sektöründe kullanılan kimyasallardan olan sirke (üzüm ve alıç) ve karbonat karışımlarının kullanılması sonrasında meydana gelen bazı yüzey özelliklerine (renk parametreleri, parlaklık değerleri ve beyazlık indeksi: *WI**) ait değişimleri araştırılmıştır. Çözeltiler hazırlandıktan sonra ahşap malzeme yüzeylerine tek olarak fırça ile uygulanmıştır. Çözelti uygulanmamış ve uygulanmış olan yüzeyler birbirleri ile kıyaslanmıştır. Varyans analizlerine bakıldığında bütün testler üzerinde çözelti türünü anlamlı olarak tespit edildiği görülmüştür. Hazırlanmış olan her iki çözeltiler ile *a** değerleri artarken, *WI**, *L**, *b**, *C**, *h*° ile bütün dereceler ve yönlerde yapılan parlaklık değerlinde azalışlar tespit edilmiştir. Δ*E** değerleri üzüm sirke + karbonat çözeltisi ile 12.31 ve alıç sirkesi + karbonat çözeltisi ile 12.92 olarak elde edilmiştir. Her iki çözelti uygulamaları ile Δ*L**, Δ*a** ve Δ*C** değerleri negatif olarak elde edilirken, Δ*b** değerleri ise pozitif olarak bulunmuştur. Hazırlanan çözeltilerin ahşap malzeme yüzeylerinde renk değiştirici etkide bulunduğu görülmüştür.

Anahtar Kelimeler: Dut, karbonat, renk, parlaklık, sirke, beyazlık indeksi

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Introduction

Given the low process energy requirements and the consequent reduction in carbon emissions linked with wooden constructions, the incorporation of wood and wood products in building construction has emerged as a notable aspect of this table [1].

Color plays a crucial role, particularly in the selection of species for diverse engineering applications such as plywood, furniture, and flooring material production. Nonetheless, it's worth noting that the color of wood may undergo changes upon exposure to light [2].

The color appearance of wood surfaces holds significant importance, particularly in indoor settings where wood is extensively utilized. This necessitates a precise method of measurement to discern color disparities (Δ*E**) across wooden surfaces. One widely adopted approach for color assessment is through the CIELAB system. Within this framework, each distinct color is defined and can be represented as a vector within a three-dimensional geometric space. The *L** axis in the CIELAB system signifies luminosity, with values ranging from 0 for black to 100 for white, while the *a** and *b** coordinates denote chromaticity (+*a* for red, -*a* for green, +*b* for yellow, -*b* for blue). By employing *a**, *L**, and *b** values, the comprehensive color deviation Δ*E* can be determined. Consequently, the disparity in color between two samples can be readily evaluated using the DIN 6174 [3] equation [4].

Vinegar, wine, sorghum, grape, pear, molasses, fruit, coconut, apple, honey, maple syrup, cantaloupe, beer, potato, beet, malt, grains, and cottage cheese are examples of products that can be derived from almost any carbohydrate source capable of fermentation. Initially, yeasts transform natural sugars found in food into alcohol. Following this, acetic acid bacteria convert the alcohol into acetic acid [5].

Carbonate minerals include common types such as calcite and aragonite (both $CaCO_3$), metal ores (e.g., siderite: $FeCO_3$, malachite: $Cu_2CO_3(OH)_2$), industrial minerals (e.g., magnesite: $MgCO_3$, huntite: $CaMg_3(CO_3)_4$), and rare minerals (e.g., McGuinnessite: $(Mg, Cu)_2(CO_3)(OH)_2$) [6,7].

Wood has always been perceived as a versatile material with multifunctional properties. Each of its versatile characteristics makes it suitable for specific ultimate uses [8].

The hue of contemporary trees is dictated by the existence of lignin and assorted organic substances. Nonetheless, during the process of wood mineralization, despite the preservation of cellular attributes, the innate color of the wood tends to diminish. Should trees undergo mineralization involving iron pyrite, iron oxide, or copper minerals, the tint of fossilized wood is influenced by the hues of these minerals. Silica mineralization is prevalent in wood [9].

To change the color of wood or replicate its texture, chemical stains that interact with wood are often employed, a technique referred to as wood staining. Nevertheless, these stains can present several issues, including limited resistance to light, inadequate color longevity, and chemical instability. Moreover, some stains might contain toxic heavy metal elements like lead and chromium. Furthermore, conventional wood staining methods entail significant water usage, and the rate of stain loss is notably higher when compared to textile dyeing processes [10,11].

Morus alba L., a member of the Moraceae family, is one of the most valuable plants in terms of natural ingredients [12-14]. *Morus alba* originates from India, Japan, and China, with sporadic cultivation observed in certain parts of Europe, North America, and Africa. Known widely as white mulberry, it is cultivated globally in areas where silkworms are reared. The primary nourishment for silkworms is sourced from the leaves of the white mulberry tree [15]. The branches are collected either at the end of spring or the beginning of summer, and subsequently dried for future utilization. Roots, however, are gathered during the winter season [16].

The leaves contain 10% tannins [17]. White mulberry, or its species reported from the China-Japan Diversity Center, has been reported to tolerate diseases, low pH, hydrogen fluoride, drought, frost, shade, salt, slope, poor soil, and weeds [18].

Plants of many species grow white in color. However, they later turn pale yellow with pink edges, and upon ripening, they become red. When fully ripe, their colors turn from dark purple to black [19]. Mulberry roots are fragile and should be handled with care when planting. Any pruning should only be done during the winter months when the plant is completely dormant [16]. Plants are quite resistant to wind [20].

They tolerate annual average minimum temperatures between -23.3 to -28.9°C. White mulberry grows well in a wide variety of soils. It is drought-resistant when thoroughly established but can be damaged by wind. It is grown up to 3300 m altitude in India but can also grow at sea level [21]. It provides medium-grade firewood. Branches are used as binding material and in basket making. The bark is fibrous and is used in papermaking in China and Europe [22]. The leaves of the plant have been used as medicine since ancient times [12-14].

In mulberry wood, the Janka hardness values were found to be 73.24 N/mm^2 on the radial surface, 77.69 N/mm^2 on the tangential surface, and 93.71 N/mm^2 on the transverse surface [23], and the thermal conductivity coefficient was found to be 0.155 W/m.K [24].

This study investigates changes in some surface properties (color, glossiness, and whiteness index) of mulberry (*Morus alba*) wood after the use of vinegar and carbonate mixtures, which are used as colorants in the food industry.

Materials and Methods

Dut (*Morus alba*) wood samples were prepared in dimensions of 10 x 10 x 2 cm. Subsequently, conditioning treatments were applied to these samples at 20±2°C and 65% relative humidity [25].

Chemicals used in this study include carbonate and two different types of vinegar [grape (with sodium metabisulfite additive) and hawthorn (saturated sugar %0.17, carbohydrate %1.00, fat %0.05, salt %0.07, saturated fat %0.02, and protein %0.30)]. Solutions prepared in two different types [50 ml vinegar + 5 g carbonate] were applied to wooden surfaces as a single layer using a brush.

The whiteness index (*WI**) values were determined using the Whiteness Meter BDY-1 device [26]. Glossiness tests were conducted using the ETB-0833 model gloss meter device, following the ISO 2813 [27] standard. Color changes were measured using the CS-10 (CHN Spec, China) device [28]. The results for total color differences were determined using the following formulas.

$$h^o = \arctan [b^*/a^*] \quad (1)$$

$$\Delta a^* = [a^*_{\text{vinegar + carbonate applied}}] - [a^*_{\text{control}}] \quad (2)$$

$$\Delta b^* = [b^*_{\text{vinegar + carbonate applied}}] - [b^*_{\text{control}}] \quad (3)$$

$$\Delta L^* = [L^*_{vinegar + carbonate\ applied}] - [L^*_{control}] \quad (4)$$
$$\Delta E^* = [(\Delta L^*)^2 + (\Delta b^*)^2 + (\Delta a^*)^2]^{1/2} \quad (5)$$
$$\Delta C^* = [C^*_{vinegar + carbonate\ applied}] - [C^*_{control}] \quad (6)$$
$$C^* = [(a^*)^2 + (b^*)^2]^{1/2} \quad (7)$$
$$\Delta H^* = [(\Delta E^*)^2 - (\Delta L^*)^2 - (\Delta C^*)^2]^{1/2} \quad (8)$$

In the literature, Δ*C** is defined as chroma difference or saturation difference and Δ*H** as hue difference or shade difference, and definitions for other parameters are presented in Table 1.

Standard deviations, maximum and minimum values, mean values, homogeneity groups, variance analyses, and percentage (%) change rates were calculated using a statistical program.

Findings and Discussion

The results for total color differences are given in Table 3. Δ*E** values are determined as 12.31 with grape vinegar + carbonate solution and 12.92 with hawthorn vinegar + carbonate solution. It is observed that these results are very close to each other. Δ*H** values are obtained as 4.47 with grape vinegar + carbonate solution and 5.24 with hawthorn vinegar + carbonate solution. While Δ*L**, Δ*b**, and Δ*C** values are negative (darker than the reference, bluer than the reference, and duller than the reference, respectively) with both solution applications, Δ*a** values are obtained as positive (redder than the reference). According to the color change criteria, both prepared solutions give "very strong (> 12.0)" results as shown in Table 3.

Table 1. Definitions of Δb, ΔL*, Δa*, and ΔC* values [29]*

Parameter	Positive condition	Negative condition
Δ*L**	Lighter than the reference	Darker than the reference
Δ*a**	Redder than the reference	Greener than the reference
Δ*b**	Yellower than the reference	Bluer than the reference
Δ*C**	Clearer, brighter than the reference	Dull, hazier than the reference

Table 2. Comparison criteria for ΔE assessment [30]*

Δ*E**	Visual color score difference	Δ*E**	Visual color score difference
<0.20	Imperceptible	3.00 - 6.00	Very noticeable
0.20 - 0.50	Very slight	6.00 - 12.00	Strong
0.50 - 1.50	Slight	> 12.00	Very strong
1.50 - 3.00	Noticeable		

Table 3. Results of total color differences

Solution Type	Δ*L**	Δ*a**	Δ*b**	Δ*C**	Δ*H**	Δ*E**	Color Change Criterion (DIN 5033, 1979)
Grape vinegar + carbonate	-10.73	2.72	-5.40	-4.07	4.47	12.31	Very strong (> 12.0)
Hawthorn vinegar + carbonate	-10.78	2.98	-6.47	-4.83	5.24	12.92	

Table 4. The measurement results determined for color parameters, whiteness index (WI) values, and glossiness values*

Test	Solution Type	Mean	Change (%)	Homogeneity Group	Standard Deviation	Mini-mum	Maxi-mum	Coefficient of Variation
*L**	Control	57.06	-	A*	0.41	56.62	58.05	0.72
	Grape vinegar + carbonate	46.33	↓18.80	B	0.37	45.72	46.88	0.81
	Hawthorn vinegar + carbonate	46.28	↓18.89	B**	0.85	44.98	47.76	1.83
*a**	Control	9.30	-	B**	0.42	8.22	9.84	4.50
	Grape vinegar + carbonate	12.01	↑29.14	A	0.35	11.50	12.51	2.91
	Hawthorn vinegar + carbonate	12.27	↑31.94	A*	0.65	11.07	13.09	5.29
*b**	Control	29.43	-	A*	0.77	28.52	30.71	2.62
	Grape vinegar + carbonate	24.03	↓18.35	B	0.49	23.18	24.79	2.02
	Hawthorn vinegar + carbonate	22.96	↓21.98	C**	0.88	21.75	24.59	3.85
*C**	Control	30.86	-	A*	0.71	30.06	32.13	2.29
	Grape vinegar + carbonate	26.79	↓13.19	B	0.46	26.12	27.51	1.73
	Hawthorn vinegar + carbonate	26.04	↓15.62	C**	1.01	24.74	27.75	3.88
h°	Control	72.45	-	A*	0.96	71.59	74.81	1.33
	Grape vinegar + carbonate	63.43	↓12.45	B	0.49	62.59	64.08	0.78
	Hawthorn vinegar + carbonate	61.87	↓14.60	C**	0.96	61.01	63.71	1.55
⊥20°	Control	0.28	-	A*	0.04	0.20	0.30	15.06
	Grape vinegar + carbonate	0.12	↓57.14	B	0.04	0.10	0.20	35.14
	Hawthorn vinegar + carbonate	0.10	↓64.29	B**	0.00	0.10	0.10	0.00
⊥60°	Control	2.62	-	A*	0.39	1.90	2.90	14.71
	Grape vinegar + carbonate	2.12	↓19.08	B	0.26	1.80	2.50	12.34
	Hawthorn vinegar + carbonate	1.52	↓41.98	C**	0.12	1.30	1.60	8.09
⊥85°	Control	3.22	-	A*	0.32	2.70	3.60	10.02
	Grape vinegar + carbonate	2.10	↓34.78	B	0.30	1.80	2.60	14.20

	Hawthorn vinegar + carbonate	0.78	↓75.78	C**	0.10	0.60	0.90	13.24
‖20°	Control	0.40	-	A*	0.00	0.40	0.40	0.00
	Grape vinegar + carbonate	0.25	↓37.50	B	0.05	0.20	0.30	21.08
	Hawthorn vinegar + carbonate	0.12	↓70.00	C**	0.04	0.10	0.20	35.14
‖60°	Control	2.87	-	A*	0.14	2.70	3.00	4.94
	Grape vinegar + carbonate	2.22	↓22.65	B	0.13	2.10	2.40	5.93
	Hawthorn vinegar + carbonate	1.56	↓45.64	C**	0.05	1.50	1.60	3.31
‖85°	Control	6.48	-	A*	0.54	5.90	7.30	8.38
	Grape vinegar + carbonate	2.96	↓54.32	B	0.21	2.70	3.20	6.98
	Hawthorn vinegar + carbonate	1.28	↓80.25	C**	0.17	1.10	1.50	13.18
*WI**	Control	11.46	-	A*	0.20	11.10	11.60	1.71
⊥	Grape vinegar + carbonate	6.38	↓44.33	C**	0.08	6.30	6.50	1.24
	Hawthorn vinegar + carbonate	7.36	↓35.78	B	0.32	6.90	7.70	4.40
*WI**	Control	8.42	-	A*	0.51	7.50	8.90	6.02
‖	Grape vinegar + carbonate	3.46	↓58.91	C**	0.27	3.00	3.70	7.85
	Hawthorn vinegar + carbonate	4.80	↓42.99	B	0.11	4.70	4.90	2.20

Number of Measurements: 10, *: Highest result, **: Lowest result

Table 5. Analysis of variance results for color parameters, whiteness index (WI) values, and glossiness values*

Source	Test	Mean Square	Sum of squares	Degrees of freedom	F	α≤0.05
	*L**	770.573	2	385.286	1129.469	0.000*
	*a**	54.374	2	27.187	113.443	0.000*
	*b**	240.711	2	120.355	223.674	0.000*
	*C**	134.889	2	67.445	116.559	0.000*
	h°	653.038	2	326.519	469.413	0.000*
	⊥20° glossiness	0.195	2	0.097	82.125	0.000*
Solution Type	⊥60° glossiness	6.067	2	3.033	39.224	0.000*
	⊥85° glossiness	29.835	2	14.917	219.852	0.000*
	‖20° glossiness	0.393	2	0.196	129.293	0.000*
	‖60° glossiness	8.581	2	4.290	320.884	0.000*
	‖85° glossiness	140.843	2	70.421	576.874	0.000*
	*WI** (⊥)	145.256	2	72.628	1459.045	0.000*
	*WI** (‖)	131.672	2	65.836	577.134	0.000*
	*L**	9.210	27	0.341		
	*a**	6.471	27	0.240		
	*b**	14.528	27	0.538		
	*C**	15.623	27	0.579		
	h°	18.781	27	0.696		
	⊥20° glossiness	0.032	27	0.001		
Error	⊥60° glossiness	2.088	27	0.077		
	⊥85° glossiness	1.832	27	0.068		
	‖20° glossiness	0.041	27	0.002		
	‖60° glossiness	0.361	27	0.013		
	‖85° glossiness	3.296	27	0.122		
	*WI** (⊥)	1.344	27	0.050		
	*WI** (‖)	3.080	27	0.114		
	*L**	75458.128	30			
	*a**	3820.014	30			
	*b**	19715.338	30			
	*C**	23498.349	30			
	h°	131025.983	30			
	⊥20° glossiness	1.060	30			
Total	⊥60° glossiness	138.780	30			
	⊥85° glossiness	155.700	30			
	‖20° glossiness	2.410	30			
	‖60° glossiness	156.350	30			
	‖85° glossiness	527.200	30			
	*WI** (⊥)	2263.400	30			
	*WI** (‖)	1062.160	30			
orr cte d	*L**	779.783	29			

*a**	60.845	29
*b**	255.239	29
*C**	150.513	29
h°	671.819	29
⊥20° glossiness	0.227	29
⊥60° glossiness	8.155	29
⊥85° glossiness	31.667	29
‖20° glossiness	0.434	29
‖60° glossiness	8.942	29
‖85° glossiness	144.139	29
*WI** (⊥)	146.600	29
*WI** (‖)	134.752	29

*: Significant

The measurement results determined for the color parameters, whiteness index (*WI**) values, and glossiness values are presented in Table 4. The results of the variance analysis are presented in Table 5.

According to these results, it is observed that the factor of solution type applied for all tests is significantly obtained (Table 5).

For the *L** value, the highest result is found in the control samples (57.06), while the lowest result is determined by the application of the solution composed of hawthorn vinegar + carbonate mixture to the experimental samples (46.28). The highest decrease rate in the *L** parameter is achieved with hawthorn vinegar + carbonate solution at 18.89%, while the lowest decrease rate is found with grape vinegar + carbonate solution at 18.80% (Table 4).

In terms of the parameter *a**, the control samples exhibit the lowest value at 9.30, while the experimental samples treated with a solution containing hawthorn vinegar + carbonate mixture display the highest value at 12.27. Notably, the application of hawthorn vinegar + carbonate solution shows the most significant increase in the *a** value, reaching 31.94%, whereas the lowest increase is observed with grape vinegar + carbonate solution, registering at 29.14% (Table 4).

Regarding the *b** value, the control samples yield the highest result at 29.43, whereas the lowest result is seen following the application of the solution containing hawthorn vinegar + carbonate mixture, which measures 22.96. Notably, the most significant decrease in the *b** parameter occurs with the application of hawthorn vinegar + carbonate solution, showing a decrease rate of 21.98%, whereas the lowest decrease rate is associated with grape vinegar + carbonate solution, standing at 18.35% (Table 4).

In terms of the *C** value, the control samples exhibit the highest result, measuring 30.86, while the lowest value is found after applying the solution containing hawthorn vinegar + carbonate mixture to the wood samples, resulting in 26.04. Notably, the most substantial decrease in the *C** parameter is associated with hawthorn vinegar + carbonate solution, showing a decrease rate of 15.62%, whereas the lowest decrease rate is observed with grape vinegar + carbonate solution, registering at 13.19% (Table 4).

Regarding the *h*° parameter, the control samples display the highest measurement, reaching 72.45, whereas the lowest reading is obtained when applying the solution comprising hawthorn vinegar + carbonate mixture to the experimental samples, yielding 61.87. Notably, the most pronounced decrease in the *h*° parameter is observed with hawthorn vinegar + carbonate solution, showing a reduction of 14.60%, while the smallest decrease rate is attributed to grape vinegar + carbonate solution, with a decrease of 12.45% (Table 4).

Looking at the glossiness tests, decreases are obtained in all grades and directions. The highest glossiness results are determined in the control experimental samples (⊥20°: 0.28, ⊥60°: 2.12, ⊥85°: 3.22, ‖20°: 0.40, ‖60°: 2.87, and ‖85°: 6.48). The lowest glossiness results are found on the samples treated with hawthorn vinegar + carbonate solution (⊥20°: 0.10, ⊥60°: 1.52, ⊥85°: 0.78, ‖20°: 0.12, ‖60°: 1.56, and ‖85°: 1.28), and the highest decrease rates are also obtained with this solution application (Table 4).

After the application of solutions, decreases are noted in both directions of *WI** values. The highest *WI** values are observed in the control samples (⊥: 11.46 and ‖: 8.42), whereas the lowest values are recorded in samples treated with grape vinegar + carbonate solution (⊥: 16.38 and ‖: 3.46). The decrease rates for *WI** values are %44.33 to %35.78 in the ⊥ direction and %58.91 to %42.99 in the ‖ direction for grape vinegar + carbonate and hawthorn vinegar + carbonate solutions, respectively (Table 4).

Studies in the literature have reported changes in color parameters, glossiness values, and whiteness index of wood surfaces when solutions prepared from vinegar and carbonate chemicals are applied [31-33]. It can be inferred that the solution interacts with the wood material's structure, causing alterations.

Conclusion

With the application of both prepared solutions, there was an increase in the *a** values, while reductions were noted in *WI** (in both directions), *L**, *b**, *C**, *h*°, and across all degrees and directions of gloss values. The Δ*E** values were recorded as 12.31 for the grape vinegar + carbonate solution and 12.92 for the hawthorn vinegar + carbonate solution. Prepared solutions have been observed to have a color-changing effect on wooden surfaces. With the application of both solutions, negative values were obtained for ΔL^*, Δb^*, and ΔC^*, while positive values were observed for Δa^*.

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