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Influence of UV radiation on the color of furfurylated wood protected with refining coatings

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Abstract:*Influence of UV radiation on the color of furfurylated wood protected with refining coatings*. Furfurylation is a method of chemical modification of wood, with significant effects on its strength parameters, resistance to degradation and color. The purpose of this study was to evaluate the color changes of furfurylated wood under UV radiation in an accelerated aging test. The wood was refined with selected protection agents - polyurethane varnish, linseed oil and wax; in addition, the stabilizing agent Tinuvin® 292 was used as an additive to the wax and polyurethane varnish. The most favorable results in terms of UV light protection of furfurylated wood were obtained with polyurethane varnish, while among the two natural coatings, linseed oil showed significantly more favorable properties than wax. The UV light stabilizing agent was much more effective in protecting the color of unmodified wood than furfurylated wood.

Keywords: chemical modification, furfurylated wood, UV radiation, color, refining coatings, protection

INTRODUCTION

Wood - a material that has accompanied the man for centuries, is invariably one of the key natural raw materials due to its availability, renewability, ease of acquisition and processing, versatility of use and favourable physical and chemical properties. The fact that, despite all its advantages, it remains an organic constructionmaterial, inherently susceptible to degradation by atmospheric agents and organisms adapted to its decomposition, has led us to seek methods directed at counteracting these factors. The dynamic development of technology and science of the last two centuries, and the wealth of knowledge about the structure and chemistry of wood that has been expanded as a result, has enabled us to develop effective methods of refining this material to protect its properties, as well as to introduce methods of modification that are capable not only of protecting them, but also of modifying them.

The aforementioned methods include chemical modification methods, which involve stable covalent bonding of a given functional group of the reactant to the structural components of the wood cell wall, i.e. cellulose, hemicelluloses and lignin. These methods, due to the fact that they are directed at changing the chemical structure of the structural components of wood, also interfere in a very significant and irreversible way with its physicochemical characteristics, which is manifested, *inter alia*, in enhanced resistance to degradation and dimensional stability, higher density and hardness, as well as changes in color (Rowell 1983). Among the mentioned characteristics changes, reduced water sorption and hygroscopicity of the material, related to the conversion of hydroxyl groups into larger, more hydrophobic groups (Thybring, Fredriksson 2021), with the cell wall of the wood becoming permanently swollen and the equivalent moisture content reduced, are responsible for the enhanced resistance to degradation and dimensional stability - but these are factors that are part of more complex mechanisms (Homan, Jorissen 2004; Mantanis 2017).

Furfurylation of wood is a method of modifying it with furfuryl alcohol ($C_5H_6O_2$) - a reagent obtained industrially through the hydrogenation of furfural ($C_5H_4O_2$), which in turn is a widely used product of lignocellulosic biomass processing (Malinowski, Wardzińska 2012; Gómez Millán, Sixta 2020; An, Li 2022). In simple terms, the modification process involves vacuum-pressure impregnation of the wood with furfuryl alcohol with the addition of an acid catalyst, followed by *in situ* polycondensation at elevated temperature, followed by product drying. Chemically, the polycondensation of furfuryl alcohol in wood is a complex series of chemical reactions, and the conditions of the process (time, temperature, chemical structure of the catalyst, pH, presence of water) highly determine the final effects of the modification: the weight percentage gain (WPG) as the main parameter determining the effects of the process, the type of chemical bonds formed with the structural components of the wood, and the degree of wood degradation - mainly depolymerization through acid hydrolysis (Lande*et al.* 2004; Lande*et al.* 2008*;* Eikenes*et al*. 2008; Dumarçay*et al*. 2017).

Furfurylation has a significant impact on the wood physical and strength characteristics and its resistance to biodegradation, with the changes depending on the WPG parameter achieved in the process. The wood's equivalent moisture content (Esteves *et al*. 2011) and water absorption are reduced. Wood density and dimensional stability are increased. Changes in mechanical properties are manifested in an increase in modulus of elasticity and hardness (Abdillah*et al*. 2022), however, the brittleness of the material is also enhanced (Eikenes*et al*. 2008). Resistance to degradation caused by fungi and insects goes up, while the biological and ecological harm of modified wood is low, similarly to that of polymerized furfuryl alcohol (ibid.).

Specific to furfurylated wood are also changes in color, which, as a result of the modification, are sometimes compared to the colors of exotic wood species, which is also caused by the chemical changes that have occurred in the wood structure. The color changes - again - are bigger, the higher the WPG index obtained (Li *et al.* 2016*;* Abdillah*et al*. 2022).

The protective effect of conventional wood refining agents - such as drying vegetable oils and waxes - is based primarily on their water repellency. Wood dried and coated or impregnated with these agents becomes more or less resistant to the penetration of water into the cells (Humar, Lesar 2013), so it can more easily be maintained in a moisture range that is suboptimal for the development of wood decay fungi, i.e. a dry protective condition - with a moisture content equal to or lower than 20% (in the case of particular insect species, this limit is accordingly lower) (Witomski 2015). More broadly, the physical and chemical properties of agents belonging to the category of oils and waxes, as well as their mode of action, are quite individual for a given agent, depending on the chemical composition - which also applies to the question of wood protection against UV radiation impact.

The purpose of this study is to evaluate the effectiveness of selected refining agents in fixing the color obtained by furfurylation through exposure to UV radiation in the accelerated wood aging test.

MATERIALS AND METHODS

The study was carried out on 120 samples of black poplar (*Populus Nigra* L.) wood with 20×20×50 mm dimensions. The samples were subjected to furfurylation followed by refinement. Popular wood preservatives of natural (wax and linseed oil) and synthetic origin - polyurethane (PUR) varnish - were used in the study. The use of a commercially available UV-dispersing agent (Tinuvin® 292), which is a mixture of bis(1,2,2,6,6-pentamethyl-4-piperidinyl) sebacate and 1-(methyl)-8-(1,2,2,6,6-pentamethyl-4-piperidinyl) sebacate, as an additive to wax and varnish in separate trials with a concentration of 1%, was also included in the test programme. The list of additives is detailed in Table 1. The final step was an accelerated material UVageing test. Color measurements were taken witha SP60 spectrophotometer X-Rite Europe GmbH (Regensdorf, Switzerland) expressing the results in the CIELabcolor space.

Table 1. Division of test samples by refining agents used

Coating	Number of samples in the trial		Manufacturer	Product code
	Modified	**Non-modified**		
Wax	10	10	BormaWachs	012008
Linseed oil	10	10	Flexol	5LB_CP3
PUR varnish	10	10	Tikkurila	005 0090
Wax	10	10	BormaWachs	012008
+ Tinuvin® 292			Kremer Pigmente	78152
PUR varnish	10	10	Tikkurila	005 0090
+ Tinuvin® 292			Kremer Pigmente	78152
Without coating	10	10	-	-

Furfurylation

The modification process was carried out using a 50% aqueous solution of furfuryl alcohol (furfuryl alcohol 98%; Sigma Aldrich) with 1% citric acid (monohydrate p.a.; Chempur) as catalyst. In the first step of the process, the samples were placed in a vacuum-pressure impregnator for a several hours; the temperature of the solution was not raised at that time. Before and after furfurylation, the samples were weighed to calculate the WPG index.

The next step was the polycondensation of furfuryl alcohol in the modified samples, which for this purpose were put into a laboratory dryer - initially wrapped in aluminium foil for 72 hours at 120°C and then without foil for 24 hours at 105°C.

After furfurylation, the average WPG index was calculated, indicating the weight percentage gain obtained in the process, using the following formula:

$$\boldsymbol{WPG = \frac{W_2 - W_1}{W_1} \times 100\%}$$

For: W_1 – dry mass of the sample before modification
W_2 – dry mass of the sample after modification

UV accelerated ageing test

The device used in the test was a Solarbox ageing chamber. The test was carried out using four operation cycles of the device and the programmes of the individual cycles (duration and irradiance) were as follows:

a) Cycle I: 24 h; 280 W/m^2
b) Cycle II – IV: 95 h; 500 W/m^2

The total radiation values were summed up for the above cycles:

$$\boldsymbol{H_e = 280\frac{J}{s \cdot m^2} \times (60 \times 60 \times 24)s + 3 \times 500\frac{J}{s \cdot m^2} \times (60 \times 60 \times 95)s}$$
$$\boldsymbol{= 537192000\frac{J}{m^2} = 537.19\frac{MJ}{m^2}}$$

Colortesting

Color measurements were taken using a portable spectrophotometer (SP60 spectrophotometer X-Rite Europe GmbH; Regensdorf, Switzerland).

Measurement results were expressed in the three components of the CIELabcolor space:

L - lightness (luminance) from black (0) to white (100), a - color from green (-60) to magenta (+60), b - color from blue (-60) to yellow (+60).
Based on the further results, the values of total color difference ΔE and color intensity change ΔC were calculated according to the following formulas:

$$\boldsymbol{\Delta E = \sqrt{(L_2 - L_1)^2 + (a_2 - a_1)^2 + (b_2 - b_1)^2} = \sqrt{(\Delta L_1)^2 + (\Delta a_1)^2 + (\Delta b_1)^2}}$$

$$\boldsymbol{\Delta C = \sqrt{{a_2}^2 + {b_2}^2} - \sqrt{{a_1}^2 + {b_1}^2}}$$

The total color difference ΔE corresponds to the distance between two color-points in the three-dimensional CIELabcolor space.

The classification of ΔE intervals in terms of perceptibility of color changes from the perspective of a standard observer is as follows (Mokrzycki, Tatol 2011)

- $0 < \Delta E < 1$ - observer perceives no difference
- $1 < \Delta E < 2$ - only an experienced observer notices the difference
- $2 < \Delta E < 3.5$ - difference is also perceived by an inexperienced observer
- $3.5 < \Delta E < 5$ - observes a clear color difference
- $5 < \Delta E$ - has the impression of two different colors

RESULTS AND DISCUSSION

The average WPG of the modified samples was 84.33% with a standard deviation of s = 9.82%. The furfurylation process significantly affected the L, a and b parameters, where the luminance (L) was reduced by an average value of 51.29 points, the *a* parameter was increased by 4.77, and the b parameter was reduced by a value of 7.61. These changes are in line with literature data (Stamm 1977; Dong *et al*. 2016; Liu *et al*. 2021).

Table 2. shows the values of the *L*, *a* and *b* parameters after the refinement process, before the UV test and Table 3. shows the values of these parameters after the test.

It is worth noting the differences in color resulting from the use of individual enhancers (Table 2.), where, for example, linseed oil produced the highest "deepening" of wood color toward black. The values of the parameters *a* and (especially) *b* suggest an opposing effect of the enhancing agents on both parameters in the case of unmodified wood, where the values are increased, and furfurylated, where they are decreased relative to the sample without coating, which may also be related to a decrease in luminance values.

The average luminance of all samples of the control group decreased after the UV test relative to the initial condition - defined as the condition after refinement and before the UV test; the largest change in luminance was observed in the linseed oil-protected wood sample, where the parameter decreased by a value slightly higher than in the uncoated sample.

Table 2. Color measurements of samples before UV test. Standard deviation in parentheses.

		L	*a*	*b*
Control	Wax	**78.08** (2.85)	**5.17** (1.39)	**22.63** (2.09)
	Linseedoil	**77.06** (3.31)	**6.05** (1.76)	**27.63** (2.93)
	PUR varnish	**78.22** (3.12)	**4.46** (1.21)	**24.02** (1.49)

	Wax + *Tinuvin® 292*	**78.18** (3.21)	**5.27** (1.26)	**23.33** (1.22)
	PUR varnish + *Tinuvin®292*	**76.80** (2.23)	**4.71**(1.36)	**24.91** (1.13)
	Withoutcoating	**82.85** (3.12)	**3.69** (1.10)	**18.12** (1.44)
Furfurylated	Wax	**28.58** (1.64)	**6.75** (1.62)	**6.03**(2.23)
	Linseedoil	**25.10** (1.20)	**6.00** (1.37)	**5.39** (1.45)
	PUR varnish	**29.06** (1.32)	**6.26** (1.15)	**5.45** (1.41)
	Wax +*Tinuvin® 292*	**26.99** (1.07)	**7.57** (1.47)	**6.93** (1.71)
	PUR varnish + *Tinuvin® 292*	**28.29** (1.15)	**5.99** (0.95)	**5.10** (1.36)
	Withoutcoating	**30.38** (1.81)	**8.12** (1.18)	**11.19** (1.99)

Table 3. Color measurements of the samples after the UV test. Standard deviation in parentheses.

		L	*a*	*b*
Control	Wax	**75.53** (0.98)	**6,77**(0,50)	**28.94** (1.30)
	Linseedoil	**71.44** (1.87)	**8.58** (0.79)	**35.04** (1.40)
	PUR varnish	**74.94** (1.74)	**7.27** (0.64)	**33.05** (1.02)
	Wax + *Tinuvin® 292*	**76.64** (0.94)	**6.13** (0.44)	**27.09** (1.37)
	PUR varnish + *Tinuvin®292*	**74.47** (1.14)	**7.01**(0.61)	**32.75** (1.18)
	Withoutcoating	**77.35** (1.59)	**6.45** (0.64)	**27.71** (1.08)
Furfurylated	Wax	**45.90** (3.19)	**3.98** (0.58)	**13.25**(0.97)
	Linseedoil	**32.69** (3.08)	**4.80** (0.81)	**10.03** (2.51)
	PURvarnish	**33.61** (2.32)	**4.97** (1.00)	**8.31** (2.28)
	Wax +*Tinuvin® 292*	**49.64** (2.30)	**4.02** (0.82)	**13.34** (2.33)
	PURvarnish + *Tinuvin® 292*	**33.40** (1.71)	**5.10** (0.50)	**9.19** (1.25)
	Withoutcoating	**41.12** (1.98)	**4.49** (0.76)	**14.22** (1.97)

Parameters *a* and *b* increased in all samples of the control group, with parameter *b* by significantly higher values. The highest changes in these parameters were recorded for the uncoated sample and - similar - for the PUR-finished wood sample. The lowest changes in all parameters were recorded for the sample of wood treated with wax with the addition of an agent*Tinuvin® 292.*

In the group including furfurylated wood, the average luminance and the *b* parameter increased in all samples, also in all samples the *a* parameter decreased. The highest changes in the *L* and *b* parameters were recorded for the sample of wood treated with wax with the *Tinuvin*® *292* agent - where the changes in the parameters were twice as high as for the sample of wood without the coating - and for the sample treated with wax without the agent, where the value of the change in the *L* parameter was slightly lower than for the wax with the agent. The lowest changes in the *L* and *b* parameters were observed for the wood refined with pure PUR varnish, followed by the sample with PUR varnish and *Tinuvin*® *292* agent, and the sample with linseed oil.

The values of the estimators *ΔE* and *ΔC* after successive test cycles in relation to the initial condition are shown in Table 4.

Table 4. Values of total color difference (*ΔE*) and color intensity change (*ΔC*) after individual UV test cycles. Standard deviation in parentheses.

		ΔE				***ΔC***			
	Cyclenumber	I	II	III	IV	I	II	III	IV
Control	wax	2.39 (1.60)	6.07 (1.82)	7.14 (2.33)	7.48 (2.45)	-0.21 (2.16)	5.10 (2.50)	6.03 (2.69)	6.48 (2.43)
	Linseedoil	2.95 (1.08)	5.96 (2.59)	8.25 (3.20)	9.72 (3.23)	-0.32 (1.74)	4.56 (2.53)	6.67 (2.76)	7.77 (2.66)
	PURvarnish	1.23 (0.39)	5.83 (2.39)	8.32 (2.82)	10.17 (2.72)	0.05 (1.00)	5.46 (2.16)	7.73 (2.35)	9.39 (2.11)
	wax + *Tinuvin® 292*	3.43 (2.35)	4.15 (1.86)	4.63 (1.70)	5.22 (1.83)	-2.32 (2.06)	2.48 (2.38)	3.28 (2.08)	3.84 (2.15)
	PURvarnish + *Tinuvin® 292*	1.40 (0.85)	4.61 (1.52)	6.53 (1.68)	8.68 (1.82)	-0.44 (1.13)	4.12 (1.83)	6.19 (1.65)	8.11 (1.65)
	Withoutcoatin g	4.66 (1.20)	9.78 (2.06)	10.99 (2.32)	11.47 (2.38)	3.96 (1.10)	8.84 (1.69)	9.65 (1.73)	9.95 (1.66)
Furfurylated	wax	11.78 (1.96)	15.14 (2.26)	17.46 (2.79)	19.11 (2.89)	4.14 (1.41)	4.40 (1.62)	5.09 (1.37)	4.77 (1.90)
	Linseedoil	2.65 (1.42)	5.57 (2.14)	7.31 (2.14)	9.02 (2.85)	1.27 (1.03)	2.09 (1.05)	2.48 (0.83)	3.06 (1.08)
	PURvarnish	1.59 (0.41)	3.47 (0.89)	4.66 (1.15)	5.61 (1.74)	-0.61 (0.53)	0.25 (0.82)	0.96 (0.86)	1.40 (1.28)
	wax +*Tinuvin® 292*	14.19 (2.66)	19.33 (2.40)	22.15 (2.34)	23.92 (2.50)	3.30 (2.45)	4.03 (2.49)	3.77 (2.23)	3.67 (2.26)
	PURvarnish + *Tinuvin® 292*	1.77 (0.58)	4.23 (0.80)	5.55 (0.86)	6.70 (1.07)	0.32 (0.65)	1.46 (1.07)	1.97 (0.83)	2.64 (1.09)
	Withoutcoatin g	5.36 (1.96)	9.01 (2.37)	11.01 (2.37)	11.85 (2.53)	1.17 (1.06)	1.06 (1.32)	0.97 (1.57)	1.09 (1.56)

The total color difference (*ΔE*)after all UV aging cycles for all samples exceeded the value of 5, which, in Mokrzycki and Tatol's classification, means that the standard observer gets the impression of two different colors in these cases. The lowest value of the estimator in the test group was shown for the PUR varnish coating, and in the control group for the samples with a wax coating with 1% addition of the UV-scattering agent *Tinuvin® 292*.

In general, the highest values of total color difference were obtained for the wax coating trial with the addition of *Tinuvin® 292* agent in the test group. However, it should be mentioned that the reason for these changes was primarily the precipitation of the mixture in the form of a light gray opaque precipitate on the surface of the samples, the visibility of which increased with successive test cycles. To a lesser extent, this problem also applied to the pure wax test.

A much more favorable result among the two natural agents in this combination was obtained by impregnation with linseed oil, which was characterized by more effective penetration of the material than wax. After each successive cycle, the total change in color was found to be lower compared to the uncoated sample, both in the modified wood group and the control group, but the difference between the results decreased with successive cycles - after the first cycle, the result for furfurylated wood impregnated with linseed oil was 50% lower than for the uncoated sample, and after the fourth cycle - by 24%. Changes in color intensity, on the other hand, exceeded the results for the uncoated sample by increasingly higher values in this comparison, reaching almost three times the reference result after the last cycle.

As mentioned, the most favorable results were obtained with PUR varnish without the addition of the UV-scattering agent, but this is true only for the furfurylated wood group - in the control group, the lowest values of ΔC and ΔE estimators were shown for wax with the addition of the said agent. It is possible that the modified wood is less chemically compatible with the agent, which may also be evidenced by the higher effectiveness of the polyurethane varnish with the addition of *Tinuvin® 292* in the control group than in the pure varnish, a trend that is opposite to that of the modified group, where the addition of the agent reduces the effectiveness of the coating in protecting against color changes.

CONCLUSIONS

The study found that:

I. Furfurylation significantly changes the color of the wood, lowering the luminance (*L*) by an average of 51 and the *b* parameter by a value of 7.61, and increasing the *a* parameter by 4.77.

II. All of the applied protections for unmodified wood resulted in a smaller UV color change, as defined by ΔC and ΔE, compared to unmodified and unprotected wood.

III. Polyurethane varnish has the most favorable protective properties of furfurylated wood against UV radiation.

IV. Of the natural coatings used, linseed oil is more effective than wax in protecting furfurylated wood from UV exposure.

V. Tinuvin® 292 UV scattering agent, which is a mixture of bis(1,2,2,6,6-pentamethyl-4-piperidinyl) sebacate and 1-(methyl)-8-(1,2,2,6,6-pentamethyl-4-piperidinyl) sebacate, shows significantly more favorable properties for protecting unmodified wood against UV radiation than for modified wood, where its addition reduced the protective properties of the agents used - wax and polyurethane varnish.

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Streszczenie: *Wpływ promieniowania UV na barwę drewna furfurylowanego zabezpieczonego powłokami uszlachetniającymi*. Furfurylacja jest metodą chemicznej modyfikacji drewna, mającą istotny wpływ na jego parametry wytrzymałościowe, odporność na degradację oraz barwę. Celem badania była ocena zmian barwy drewna furfurylowanego pod wpływem promieniowania UV w teście przyspieszonego starzenia. Drewno uszlachetniono wybranymi środkami ochrony – lakierem poliuretanowym, olejem lnianym i woskiem, ponadto wykorzystano środek stabilizujący Tinuvin® 292 jako dodatek do wosku i lakieru poliuretanowego. Najkorzystniejsze wyniki pod względem ochrony barwy drewna furfurylowanego przed promieniowaniem UV uzyskano przy użyciu lakieru poliuretanowego, natomiast spośród dwóch naturalnych powłok, olej lniany wykazał znacznie korzystniejsze właściwości niż wosk. Środek stabilizujący światło UV dużo skuteczniej chronił barwę drewna niepoddanego modyfikacji niż furfurylowanego.

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