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THE IMPACT OF ACCELERATED WEATHERING ON THE MOLD RESISTANCE AND COLOR STABILITY OF THE NORWAY SPRUCE WOOD TREATED WITH NATURALIS OILS

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ABSTRACT

Today, various kinds of natural oils based on fast-drying components are used for the treatment of garden furniture and other wood products exposed to water and sun without ground contact. The anti-mold and the color-stabilization effects of three natural oils, *i.e.* impregnating, teak and tung modified with zinc oxide nanoparticles (nano-ZnO), UV-additive based on benzotriazole and HALS (Tinuvin 5060), and pinia pigment are presented in the paper. The "Naturalis oils" applied by painting on surfaces of the Norway spruce wood were able to inhibit growth of the molds *Asperillus niger* and *Penicillium brevicompactum*, however, their efficiency was a more pronounced only in the presence of nano-ZnO. The accelerated weathering of the oiled spruce samples in Xenotest during 2 or 4 weeks caused: (1) a decrease of their original resistance to molds, and (2) a specific color change – the highest at using unmodified transparent oils and the smallest at using pinia-pigmented oils.

Key words: additives, color, mold, oil coatings, spruce, weathering.

INTRODUCTION

The Norway spruce wood belongs to the most commonly used species for houses, bridges, garden furniture, and other exterior constructions and products in the Central Europe. With the aim to improve the spruce's wood natural resistance against molds (microscopic fungi), staining-fungi, decaying-fungi and insects (EN 350 2016), as well as against UV-light, water and other atmospheric factors (EVANS 2008), this organic material should be treated with suitable coating systems or deeply impregnated with biocides. Today, various natural oils are commonly used for surface treatment of garden furniture and other wooden products without ground contact.

Hydrophobic coatings, including natural oils based on fast-drying components, are effective for wood protection against water absorption as they decrease kinetics of wood soaking and swelling (TURKULIN *et al.* 2006, KÚDELA 2017). However, some kinds of natural oils not always sufficiently protect wood against color changes caused by sun in presence of water, and against mold attacks.

More effective against biodegradations and weathering processes are natural essential oils. For example, a good fungicidal efficiency of these oils documented YANG and CLAUSEN (2006) testing them against the molds *Aspergillus niger*, *Trichoderma viride* and *Penicillium chrysogenum*, as well as PÁNEK *et al.* (2014) who found that the most effective against *Aspergillus niger* and *Penicillium brevicompactum* were thyme, oregano, sweet flag and clove oils.

Protection efficiency and weathering stability of coatings, including natural oils, can be increased by their modification with UV-stabilizers (*e.g.* FORSTHUBER *et al.* 2013, ŠOMŠÁK *et al*. 2015) and fungicides (*e.g.* IŽDINSKÝ *et al*. 2017).

The atmospheric degradation of a defined coating system on the wood's surface differs mainly depending on: (1) the climate conditions (*e.g.* CREEMERS *et al.* 2002, VALVERDE and MOYA 2014), (2) the wood species, its initial roughness, moisture and other surface characteristics (*e.g.* REINPRECHT and PÁNEK 2015), and (3) the technology of coating's application (*e.g.* PÁNEK and REINPRECHT 2016).

With the aim to unify the above three major factors at weathering experiments, this work with twelve combinations of natural oils was performed at the following constant conditions: (1) the accelerated weathering in Xenotest, (2) the Norway spruce wood as underlying material, and (3) the oils painted in two layers.

MATERIALS AND METHODS

Wood

Samples of Norway spruce (*Picea abies* Karst. L.) wood with the dimensions of 40 mm × 20 mm × 5 mm (longitudinal × radial × tangential) were prepared from air-dried and conditioned boards having a moisture content of 12 ± 2%. Top surfaces of boards were firstly grinded gradually with sandpapers No. 60 and No. 120. Samples selected for experiments had no knots, cracks and other growth inhomogeneity or defects.

Natural oils

The basal natural oils (impregnating, teak, tung) and their modified versions with additives – all twelve marked as "Naturalis oils" – were prepared by Renojava s.r.o. Prešov, Slovakia. Their basic composition and selected characteristics are in Table 1.

Tab. 1 The basic composition and characteristics of "Naturalis oils" used in the experiment.

"Naturalis oil"	Mark	Dry mass (%)	Viscosity DIN4/20°C (s)	Drying time (hour)
Impregnating transparent oil	IT	94–96	39–41	12–24
Impregnating transparent oil with 1% nano-ZnO	IT-ZnO			
Teak transparent oil	TT	59–61	21–23	4–6
Teak transparent oil with 1% nano-ZnO	TT-ZnO			
Teak pinia oil with 1% nano-ZnO	TP-ZnO			
Teak transparent oil with 1% UV-additive Tinuvin	TT-UV			
Teak transparent oil with 2% UV-additive Tinuvin	TT-2UV			
Tung transparent oil	TuT	96–98	39–41	24–36
Tung transparent oil with 1% nano-ZnO	TuT-ZnO			
Tung pinia oil with 1% nano-ZnO	TuP-ZnO			
Tung transparent oil with 1% UV-additive Tinuvin	TuT-UV			
Tung transparent oil with 2% UV-additive Tinuvin	TuT-2UV			

- The selected characteristics (dry mass, viscosity, and drying time) of the basal natural oils (IT, TT, TuT) did not change within the defined intervals after their modification with nano-ZnO, Tinuvin 5060, and/or pinia pigment.

Additives for natural oils

The basal natural oils were modified with the following additives (Tabs. 1 and 2):

- nano zinc-oxide suspension (nano-ZnO) in methoxypropylacetate (MPA) with the mean size of 163 nm and the median size of 166 nm (nZ-BOCH 01-MPA 02,

Bochemie Bohumín, Czech Republic),
- light stabilizer Tinuvin 5060, containing a benzotriazole (BTZ) based UV absorber and a non-basic hindered amine light stabiliser (HALS) (BASF Ludwigshafen, Germany),
- pinia pigment.

Treatment of wood with natural oils

The samples from Norway spruce wood were treated with twelve combinations of the "Naturalis oils" (Tabs. 1 and 2). All these natural oils were applied by painting technology in two layers – each one in an amount of 90 ± 10 g per m^2 (Tab. 2). The second upper layer was applied after a drying-time needed for curing of the first base layer.

Tab. 2 The combinations of individual "Naturalis oils" used for treatment of the Norway spruce samples and the initial color coordinates of the oiled samples in CIE-$L^*a^*b^*$ system.

Combination of "Naturalis oils"	1st layer	2nd layer	Color coordinates of samples before weathering		
			L*	a*	b*
A	IT	TT	71.82	9.88	41.77
B	IT	TuT	74.20	8.27	44.77
C	IT	TT-ZnO	73.43	7.77	41.77
D	IT	TuT-ZnO	69.28	11.22	44.22
E	IT	TP-ZnO	61.62	15.93	43.95
F	IT	TuP-ZnO	60.12	17.63	47.95
G	IT-ZnO	TT-ZnO	72.27	8.63	37.90
H	IT-ZnO	TuT-ZnO	69.07	10.50	37.98
I	TT-UV	TT-UV	71.03	7.30	31.75
J	TuT-UV	TuT-UV	71.92	8.67	32.37
K	TT-2UV	TT-2UV	71.37	9.22	33.53
L	TuT-2UV	TuT-2UV	70.90	7.57	32.92

- The basic composition of "Naturalis oils" used for the 1st and 2nd layer is in Tab. 1.
- Combinations E and F (the second upper layers made by pinia pigmented teak or tung oils modified with nano-ZnO) have given the wood a slightly darker and more reddish tint.

Accelerated weathering

The accelerated weathering of the control and oiled samples was carried out in the Q-SUN Xe-1-S Xenotest (Q-Lab Corporation, USA) during 2 and 4 weeks. The weathering took place according to a modified Standard EN 927-6 (2006), at which 1-week of weathering consisted from 24 h conditioning at 45°C and following from 48 subcycles each lasting 3 h, *i.e*. 2.5 h UV-radiation and 0.5 h water spraying. These changes were made to the Standard: Xenon lamps instead of fluorescent UV lamps; irradiance at 340 nm set to 0.55 $W\cdot m^{-2}\cdot nm^{-1}$ instead of 0.89 $W\cdot m^{-2}\cdot nm^{-1}$; temperature on black panel 50°C instead of 60°C.

Mold test

The mold resistance test of the control and oiled samples was carried out against a mixture of two microscopic fungi *Aspergillus niger* Tiegh. and *Penicillium brevicompactum* Dierck. The mold test of samples was based on a modified Standard EN 15457 (2014) with some changes in their shape and mode of sterilization. All surfaces of samples were sterilized with the 30 W germicidal lamp (Chirana, Slovakia) from a distance of 1 m, at a temperature of 22 ± 2°C/0.5 h. The sterilized samples were then placed into Petri dishes with a diameter of 120 mm filled with a 3–4 mm thick layer of the 4.9 % Czapek-Dox agar (HiMedia Ltd., India). Into each Petri dish were placed two oiled pieces of the same type and one control unoiled piece perpendicular to them. Inoculation of the top surfaces of samples was performed directly in Petri dishes with a mixed spore suspension of used molds in sterile water (10^6–10^7 spores/ml). Incubation of the inoculated samples lasted 21 days at a temperature of 24 ± 2°C and a relative air humidity of 90–95%.

The growth activity of molds (GAM) on the top surfaces of samples was evaluated after the 7^{th}. 14^{th} and 21^{th} day by these criteria: 0 $\rightarrow$ no growth; 1 $\rightarrow$ growth $\leq$ 10%. 2 $\rightarrow$ growth > 10% and $\leq$ 30%; 3 $\rightarrow$ growth > 30% and $\leq$ 50%; 4 $\rightarrow$ growth > 50%.

Color analyses

The colors of the top surfaces of samples were analysed according to the CIE-$L^*a^*b^*$ color system using a Color Reader CR-10 (Konica Minolta. Osaka. Japan). This device is based on a D65 light source that simulates daylight. Its sensor head is 8 mm in diameter. Measurements were performed for samples conditioned at a temperature of 20 ± 1°C and a relative air humidity of 60%.

In the initial native state and after accelerated weathering, the color coordinates L^* (darkness: white – black), a^* (red – green), and b^* (yellow – blue) of each sample on its top surface 40 mm × 20 mm were determined on two equal places. The L^*, a^* and b^* values were used to calculate the total color difference ΔE^* according to Eq. 1.

$$\Delta E^* = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \quad (1)$$

where ΔL^* *is* $L^*_{weathered} - L^*_{native}$, Δa^* is $a^*_{weathered} - a^*_{native}$, *and* Δb^* is $b^*_{weathered} - b^*_{native}$.

RESULTS AND DISCUSSION

Activity of molds

Without previous weathering of the oiled spruce samples in Xenotest – in the 7^{th} day of the mold test, there was found that all 12 combinations of “Naturalis oils” were able more or less suppress the growth activity of molds, when the GAM ranged from 1.5 to 2.5 (Fig. 1a). In the 7^{th} day, relatively the highest resistant to molds had spruce samples treated with oils containing nano-ZnO – see combinations from C to H, usually with the GAM equal 1.5. Later, in the 14^{th} day and in the final 21^{st} day of the mold test, only natural oils modified with nano-ZnO secured a mild resistance of spruce samples against molds – see combinations from C to H, usually with the GAM from 2 to 3.

After previous weathering of the oiled spruce samples in Xenotest, there occurred a partial decrease of their original resistance to molds (Figs. 1b and 1c). For example, in the 7^{th} day of the mold test the GAM ranged already between 2 and 3 for 2-weeks weathered samples, and even more from 3 to 4 for 4-weeks weathered samples. Later, in the 14^{th} and 21^{st} days of the mold test, the top surfaces of oiled samples were almost always intensively attacked by molds with the GAM equal 4.

In summary, the growth activity of molds on the top surfaces of spruce samples painted with “Naturalis oils” was partly suppressed in presence of nano-ZnO. However, due to the accelerated weathering, it means at action of visible- and UV-light with stages of water spraying, the initial anti-mold effect of such modified natural oils apparently decreased, as it can be seen in selected photos, as well (Fig. 2).

Generally, the results achieved in the experiment correspond with knowledge that nano zinc oxide nanoparticles are more or less able to suppress activity of molds on surfaces of painted materials (*e.g.* BELLOTTI et al. 2015, REINPRECHT and VIDHOLDOVÁ 2017). Nano-ZnO can interact with various organisms (bacteria, molds, decaying-fungi, insects and other pests) either with the surfaces of living cell, or rather after penetration to the living cell with its core (SEIL and WEBSTER 2012).

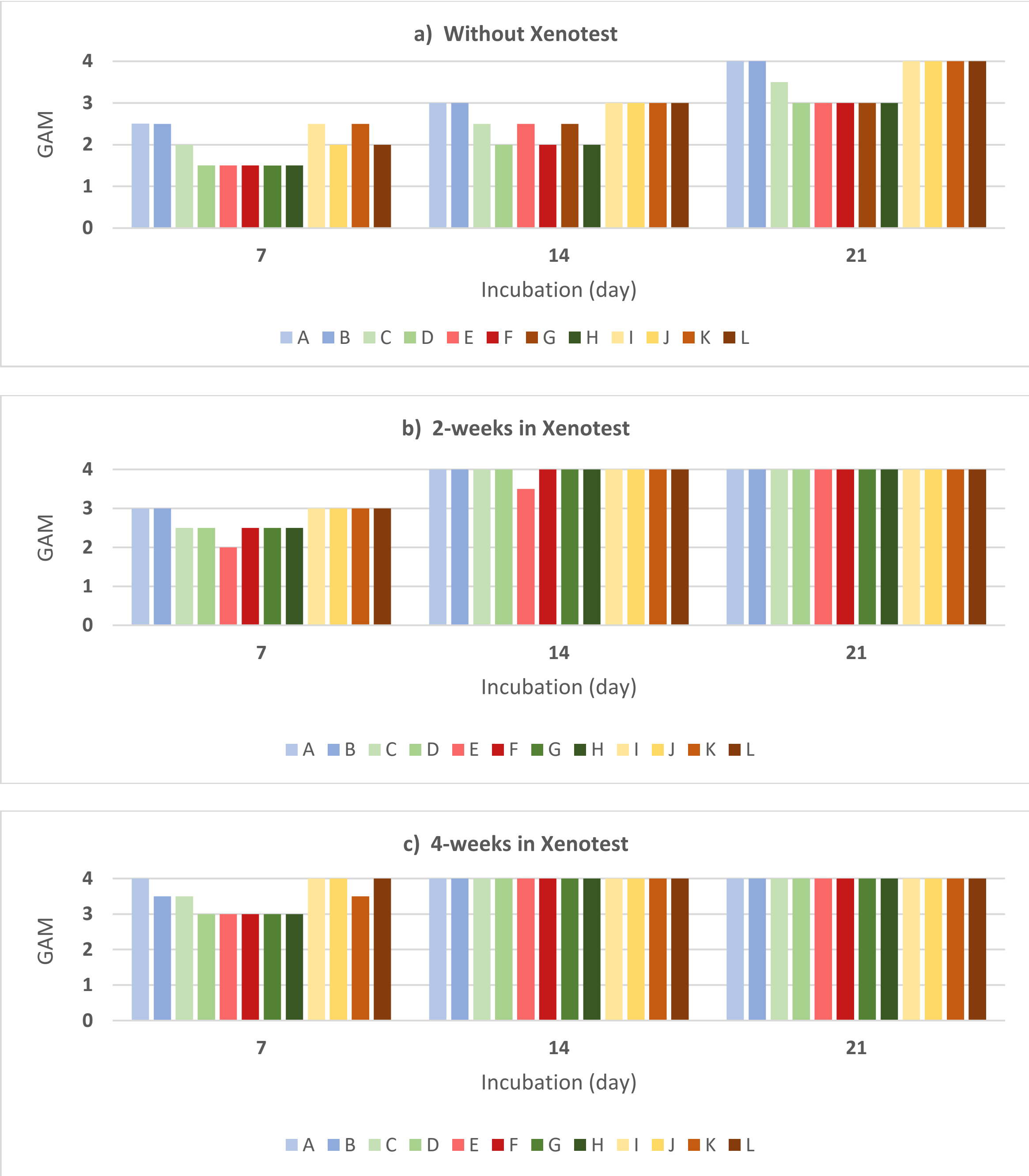


Fig. 1 The growth activity of molds (GAM) on the top surfaces of the oiled Norway spruce samples.

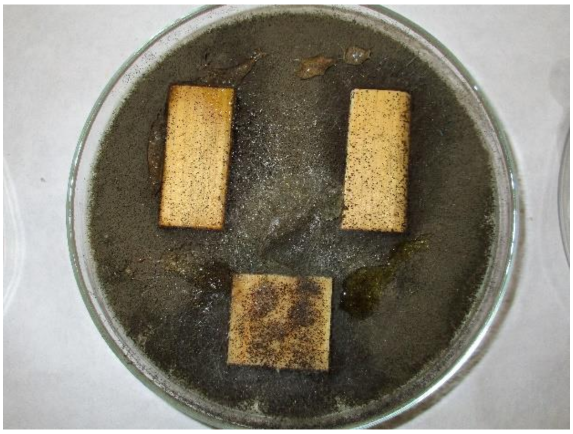
- **12 combinations of "Naturalis oils" with marking from A to L are presented in Tables 1 and 2.**
- **Nano-ZnO having a fungicide effect was used in combinations of natural oils marked from C to H.**
- **Mean values are from 4 replicates.**
- **The control untreated spruce samples had the GAM always 4, *i.e.* for all valued incubation times, with and also without their previous weathering in Xenotest.**

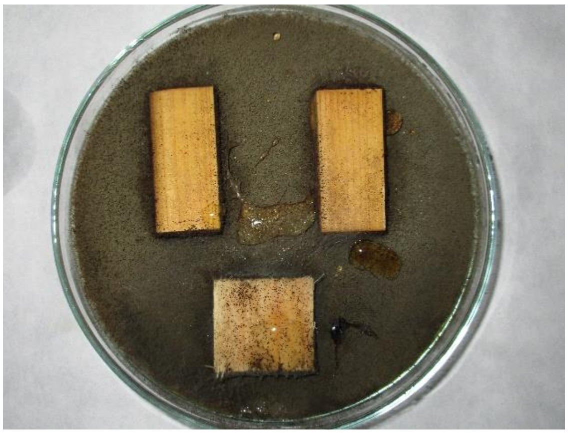
Color changes

More or less changes in the initial color coordinates of the oiled Norway spruce samples occurred at the accelerated weathering in Xenotest (Tab. 2, Figs. 3 and 4).

The top surfaces of spruce samples treated with the basal transparent natural oils (combinations A and B) have become evidently darker with ΔL^* from –11.30 to –14.18 (Fig. 3), and their total color differences ΔE^* achieved high values from 14.78 to 18.43 (Fig. 4). Similar or only slightly smaller values of $-\Delta L^*$ and ΔE^* were determined for the top surfaces of spruce samples treated with the modified transparent natural oils: (1) combinations C and D – upper oil with nano-ZnO, (2) combinations G and H – base and upper oils with nano-ZnO, and (3) combinations I, J, K and L – base and upper oils with UV-additive Tinuvin 5060 at 2 weeks of weathering (Figs. 3 and 4, Tabs. 3 and 4 – see sometimes a smaller or none significance for these modified oil combinations comparing to the basal oil combination "A" or "B").

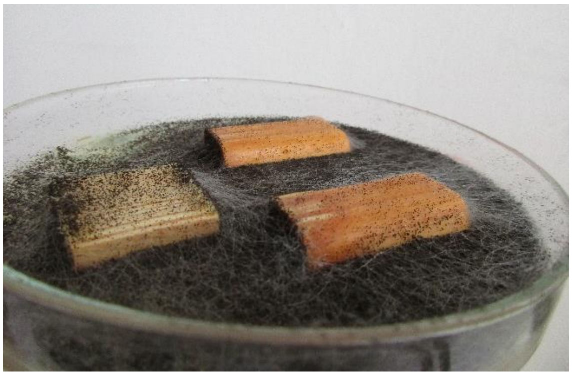
a) Without weathering in Xenotest: 21st day of the mold test

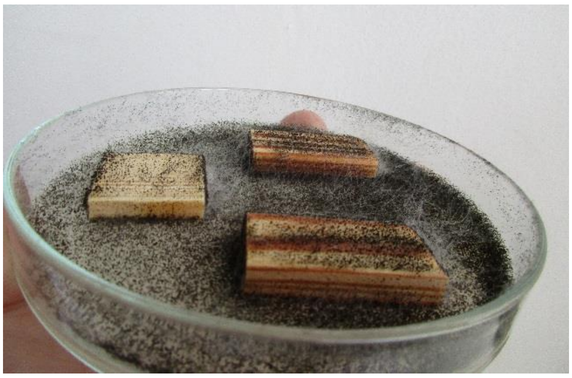




B – combination (IT + TuT): GAM = 4 D – combination (IT + TuT-ZnO): GAM = 3

b) With previous 4-weeks weathering in Xenotest: 7th day of the mold test





D – combination (IT + TuT-ZnO): GAM = 3 L – combination (TuT-2UV + TuT-2UV): GAM = 4

Fig. 2 Examples from the mold's growth on the top surfaces of the oiled Norway spruce samples.

- **In each Petri dish is one control-unoiled spruce sample (smaller sample with the GAM equal 4) placed perpendicular to two tested-oiled samples.**

However, the values of –ΔL* and ΔE* have become apparently smaller for spruce samples treated with: (1) the upper pinia pigmented natural oils – combinations E and F, and (2) also for samples treated with the transparent natural oils modified with UV-additive Tinuvin 5060 at 4 weeks of weathering (Figs. 2 and 3, Tabs. 3 and 4 – see high significance for these modified oil combinations comparing to the basal oil combinations "A" or "B").

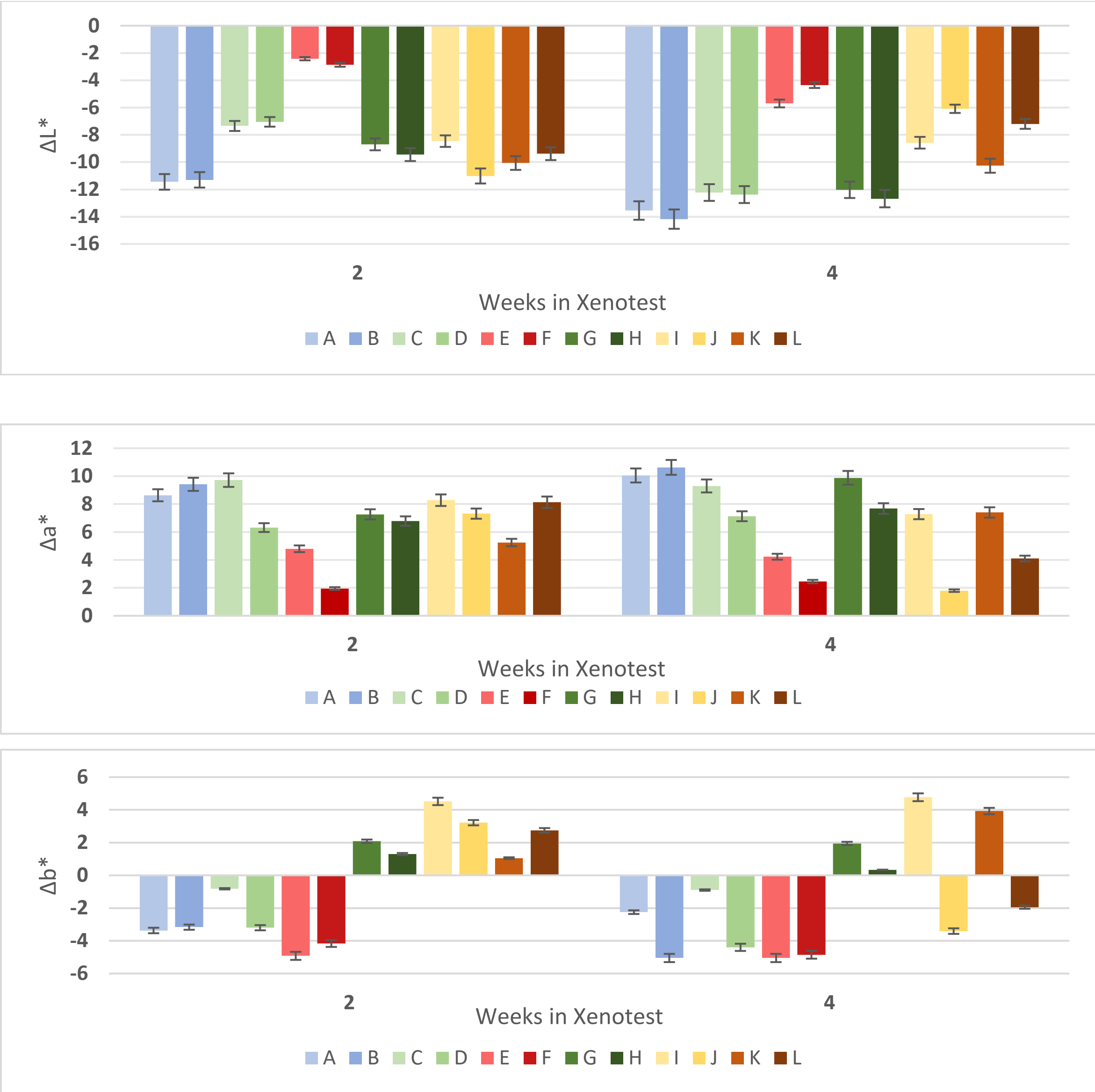


Fig. 3 The color changes ΔL*, Δa*, Δb* determined for the top surfaces of the Norway spruce samples treated with 12 combinations of "Naturalis oils" at their accelerated weathering in Xenotest.

- **12 combinations of "Naturalis oils" with marking from A to L are presented in Tables 1 and 2.**
- **Mean values are from 4 measurements, presented together with standard deviations.**

Due to the accelerated weathering, the oiled surfaces of the Norway spruce samples obtained a reddish shade (Fig. 3). The values of Δa* ranged from 1.80 (combination J – transparent base and upper oils with UV-additive Tinuvin 5060 at 4 weeks of weathering) or from 1.95 (combination F – pinia pigmented upper tung oil at 2 weeks of weathering) to 10.05 or 10.63 (combinations A and B – basal transparent teak or tung upper oils at 4 weeks of weathering). After 4 weeks of weathering in Xenotest the least pronounced redness had spruce samples treated with the pinia pigmented oils (combinations E and F) and with the transparent tung oils modified with UV-additive Tinuvin 5060 (combinations J and L).

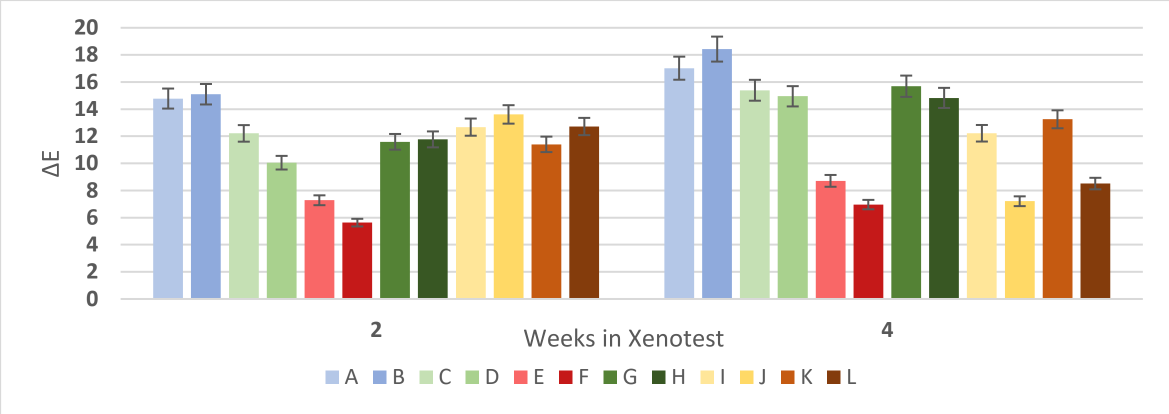


Fig. 4 The total color difference ΔE* determined for the top surfaces of the Norway spruce samples treated with 12 combinations of "Naturalis oils" at their accelerated weathering in Xenotest.

- **12 combinations of "Naturalis oils" with marking from A to L are presented in Tables 1 and 2.**
- **Mean values are from 4 measurements, presented together with standard deviations.**

Tab. 3 Significance levels of the color changes ΔL* and ΔE* at accelerated weathering of the spruce wood painted with TEAK combinations of "Naturalis oils – A, C, E, G, I, K" containing nano-ZnO, UV-additive and pinia pigment.

C	E	G	I	K
2nd 1% ZnO	2nd (pinia + 1% ZnO)	1st 1% ZnO 2nd 1% ZnO	1st 1% UV 2nd 1% UV	1st 2% UV 2nd 2% UV
ΔL* – significance levels for the modified teak oils in comparison to the base teak oil "A"				
2-weeks in Xenotest				
0.00006	0.00005	0.00016	0.00011	0.00219
a	a	a	a	b
4-weeks in Xenotest				
0.05821	0.00005	0.04091	0.00022	0.00159
d	a	c	a	b
ΔE* – significance levels for the modified teak oils in comparison to the base teak oil "A"				
2-weeks in Xenotest				
0.00017	0.00005	0.00009	0.00038	0.00007
a	a	a	a	a
4-weeks in Xenotest				
0.03718	0.00005	0.06498	0.00031	0.00099
c	a	d	a	a

- Duncan test with levels of significance: a > 99.9%, b > 99% ≤ 99.9%, c > 95% ≤ 99%, d < 95%.

Changes of the b* color coordinate were not always clear (Fig. 3). Simultaneously, these two interesting tendencies occurred:

- The top surfaces of spruce samples treated in the first layer with unmodified basal impregnated oils (combinations A, B, C, D, E and F) obtained a more blue shade with Δb* from –0,82 to –5.05, at which this tendency was not influenced by presence of pinia pigment in the second upper layer.
- On the contrary, the top surfaces of spruce samples treated in the first layer with modified natural oils containing nano-ZnO (combinations – G and H) or organic UV-additive Tinuvin 5060 (combinations – I, J, K and L) usually obtained yellower shade with Δb* from 0.33 to 3.93.

From practical point of view, this interesting knowledge was obtained: - the total color differences ΔE* of the oiled spruce samples were not effect by the type of transparent oil, *i.e.* there usually was not a difference between the teak and tung oils (Figs. 3 and 4, Tab. 5).

Tab. 4 Significance levels of the color changes ΔL* and ΔE* at accelerated weathering of the spruce wood painted with TUNG combinations of "Naturalis oils – B, D, F, H, J, L" containing nano-ZnO, UV-additive and pinia pigment.

D	F	H	J	L
2nd 1% ZnO	2nd (pinia + 1% ZnO)	1st 1% ZnO 2nd 1% ZnO	1st 1% UV 2nd 1% UV	1st 2% UV 2nd 2% UV
ΔL* – significance levels for the modified tung oils in comparison to the base tung oil "B"				
2-weeks in Xenotest				
0.00006	0.00005	0.00048	0.30748	0.00041
a	a	a	d	a
4-weeks in Xenotest				
0.00138	0.00005	0.00029	0.00006	0.00008
b	a	a	a	a
ΔE* – significance levels for the modified tung oils in comparison to the base tung oil "B"				
2-weeks in Xenotest				
0.00006	0.00005	0.00018	0.00607	0.00077
a	a	a	b	a
4-weeks in Xenotest				
0.00028	0.00005	0.00014	0.00006	0.00008
a	a	a	a	a

- Duncan test with levels of significance: a > 99.9%, b > 99% ≤ 99.9%, c > 95% ≤ 99%, d < 95%.

Tab. 5 Significance levels of the total color differences ΔE* for the weathered oiled spruce wood at comparing the TEAK versus TUNG combinations of "Naturalis oils" containing nano-ZnO, UV-additive and pinia pigment.

A-B	C-D	E-F	G-H	I-J	K-L
-	2nd 1% ZnO	2nd (pinia + 1% ZnO)	1st 1% ZnO 2nd 1% ZnO	1st 1% UV 2nd 1% UV	1st 2% UV 2nd 2% UV
ΔE* – significance levels at comparing the teak oils versus the tung oils					
2-weeks in Xenotest					
0.23790	0.01391	0.05507	0.583779	0.10327	0.06746
d	c	d	d	d	d
4-weeks in Xenotest					
0.16619	0.25373	0.07682	0.270164	0.00337	0.00907
d	d	d	d	b	b

- Teak oils: A, C, E, G, I, K.
- Tung oils: B, D, F, H, J, L.
- Duncan test with levels of significance: a > 99.9%, b > 99% ≤ 99.9%, c > 95% ≤ 99%, d < 95%.

In summary, the achieved experimental results related to the color stability of the oiled wood surfaces confirmed the knowledge of more researchers (*e.g.* DE MEIER 2001, OZGENC *et al.* 2012, FORSTHUBER *et al.* 2013, PÁNEK *et. al.* 2014, REINPRECHT and PÁNEK 2015, ŠOMŠÁK *et al.* 2015) that the color stability of transparent coatings can be improved by suitable additives, *i.e.* pigments and UV-stabilizing compounds. Presence of these additives in natural oils or in other coating systems can effectively suppress their own weathering and also weathering of the underlying wood materials – because these additives have the ability to decrease absorption of UV-light by lignin, hemicelluloses and extractives with following suppression of their depolymerisation, oxidation and other degradation reactions which usually create various color-different substances.

Organic UV-additives, for example, hindered amine light stabilisers (HALS) and benzotriazoles (BTZ) have been used for decades. However, like most organic materials, they are sensitive to UV light, degrade and cannot offer long-term protection of coated wood in exteriors. Over the last few years, metal oxide nanoparticles, most commonly titanium dioxide – TiO_2 and zinc oxide – ZnO, have been used as UV absorbing materials for a variety of applications (BLANCHET and LANDRY 2015). As more of metal oxide nanoparticles are also bioactive, their application into coatings for treatment of wood products exposed in exteriors could be a prospective.

CONCLUSIONS

- The "Naturalis oils" modified with nano-ZnO better preserved the Norway spruce wood against molds as the original unmodified oils – i.e. impregnating, teak and tung; however, the anti-mold effect of the "Naturalis oils" containing zinc oxide nanoparticles apparently decreased due to the accelerated weathering of the oiled wood in Xenotest lasting 2 or 4 weeks.
- During weathering in Xenotest, the highest color stability of the Norway spruce wood was secured by natural oils modified with pinia pigment; its partially worse protection against color changes was secured by the transparent natural oils modified either with UV-additive Tinuvin 5060 based on benzotriazole and HALS, or with nano-ZnO.
- The color stability of the oiled Norway spruce wood was not at all affected by the type (i.e. teak or tung) of transparent oil used for the second upper layer.

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