Patination of cherry wood and spruce wood with ethanolamine and surface finishes

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Summaries

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The appearance of age or the so-called patina of cherry wood and spruce wood was obtained by treatment with ethanolamine vapours. Chemical changes in the treated wood were investigated by FT-IR spectroscopy, electron paramagnetic resonance and nitrogen content analysis. It was shown that the use of aggressive and toxic ammonia could be replaced by fuming with ethanolamine. Extensive colour stability experiments of patinated cherry wood that was coated with linseed oil varnish, linseed oil, beeswax or nitro-cellulose lacquer indicated that the described antiquing method represents a new possibility for producing various wood products with the appearance of age.

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Introduction

The appearance of age or the so-called patina of wood may be reproduced intentionally for some special needs, such as in antique restoration work[1] and the production of period furniture.

Fuming of wood with ammonia is a well-known and established darkening/patination method. Unfortunately, a concentrated aqueous solution of ammonia is an aggressive and toxic agent with a strong and irritative odour.[2] In comparison with ammonia, ethanolamine (EA) is a less irritant and less toxic chemical and thus more user-friendly. For instance, it may be used as an additive in cosmetic products[3] and, in contrast to some other amines, it is not the subject of the so-called chemical weapons convention.[4] Therefore, the aim of the authors' work was to consider the fumigation of spruce and cherry wood with vapours of ethanolamine (2-aminoethanol, $H_2NC_2H_4OH$). Further, the fumed cherry wood was surface treated with some natural or synthetic resin containing finishes that are commonly applied in the production of antique furniture imitations. Furniture made of patinated wood should retain its appearance during use. Therefore, several experiments were performed to estimate the colour stability of ethanolamine-stained and surface-finished cherry wood.

Material and methods

Wood specimens, amine treatment procedures and surface finishing

Preliminary fuming experiments and the elucidation of chemical processes in ethanolamine-treated wood were performed with different samples of European cherry (*Prunus avium* L) and Norway spruce wood (*Picea abies* (L) Karst). For staining by fuming, a 10%w/w aqueous solution of ethanolamine (Merck) was applied. The specimens were fumed for 8 and 16 hours. For comparison, the authors exposed some samples also to ammonia (Merck) vapours for 2, 8 and 16 hours. The samples of dimensions 10 x 35 x 100mm, with parallel growth rings to the smaller lateral side surface were prepared for colour measurements. Before staining, the upper test sides of the samples were sanded (grain size 180). To record FT-IR spectra, thin microtomed radial slices of 30μm thickness were prepared. The nitrogen content was determined in treated and untreated fine sawdust. Before any treatment, the samples were conditioned for two weeks at the temperature of 23 $\pm$ 2°C and 50 $\pm$ 5% relative humidity. After treatments, all samples were again conditioned for one week in a dark chamber.

To obtain starting-point data for further work on colour stability of antiqued and surface-finished wood, the most commonly used wood species in Slovenia (*Picea abies*) was chosen. The specimens of 230 x 100 x 20mm with sanded (grain size 150) radial surfaces for colour measurement were prepared. Treatment with ethanolamine vapours above a 10% aqueous solution of ethanolamine took place in a large glass chamber of approximately 0.375m^3 with a ventilating fan providing circulation of the treatment gas. About 1.5L of the treatment solution was used so that the bottom of the chamber was completely covered with the liquid and the evaporation surface was about 0.25m^2. Exposure to amine lasted for 42 hours.

For the final colour stability measurements of uncoated and coated samples, cherry wood specimens were prepared as described in the previous paragraph. The treatment procedure with ethanolamine vapours was also as above, but the only exception was in an exposure period that lasted in this case for four days. After a six-day storage in a dark and dry place, various surface finishes were applied. The following surface coatings were chosen: linseed oil varnish (supplied by Mega Commerce, Ljubljana, labelled as LV; retention after the first application: 73.7g/m^2 and after the second application onto the dry first layer: 65.2g/m^2; applied by brushing), linseed oil (Becker Acroma GA820; LO; 122.9 and 60.1g/m^2; brushing), beeswax (Jales, Ljubljana; BW; 21.6 and 14.9g/m^2; applied with a cloth), and a nitro-cellulose lacquer (Becker Acroma; NC; 73.6 and 127.4g/m^2; applied by a conventional compressed air-spraying technique). After application, the surface-coated samples were conditioned for one week, followed by colour stability measurements.

UV irradiation

Some non-treated cherry and spruce wood samples were irradiated by ultraviolet (UV) light in order to simulate aging and to compare its effect on colour with the influence of ethanolamine treatment. The samples were UV irradiated for seven hours. Afterwards, the colour was measured, followed by UV irradiation for an additional 23 hours, when the colour was measured again. UV irradiation was performed with a Philips TUV G30T8 low-pressure mercury-vapour emitting short-UV lamp, with a radiation peak at 253.7nm, in the dark chamber at a temperature of 23 $\pm$ 2°C and 50 $\pm$ 5% relative humidity.

FT-IR spectroscopy

Thin 30 to 40μm slices of untreated and fumed wood were inserted between NaCl plates (see Figure 1). FT-IR spectra of the specimens were recorded between 4000cm^{-1} and 400cm^{-1} (ten scans) with the Perkin Elmer FT-IR 1720X spectrometer. Three parallel measurements were performed on each specimen. The first set of measurements was performed three days after treatment, the second set after one week, and the final set after four weeks of conditioning.
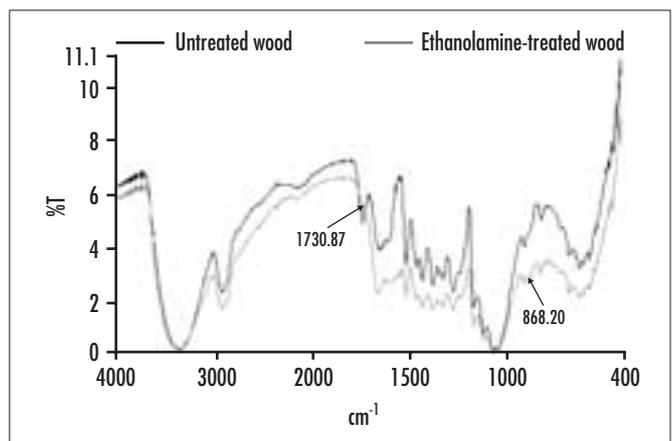

Figure 1: FT-IR spectra of untreated and fumed spruce wood

EPR spectroscopy

For the electron paramagnetic resonance (EPR) measurements, very fine wood sawdust (Mesh 130) was prepared. Approximately 0.1g of wood dust was treated with 0.1ml of an aqueous solution of ethanolamine of a 10%w/w concentration (c_{EA} = 10%) and put into a capillary tube. Afterwards, the EPR measurements started. The first measurement was performed after four minutes of the reaction. In a two-hour period, eight EPR measurements were performed, as seen in Figure 2. The measurements were carried out at room temperature with the Bruker ESP-300 X-band spectrometer (microwave frequency 9.62GHz, microwave power 20mW, modulation frequency 100kHz, modulation amplitude 0.1mT). The intensities of the
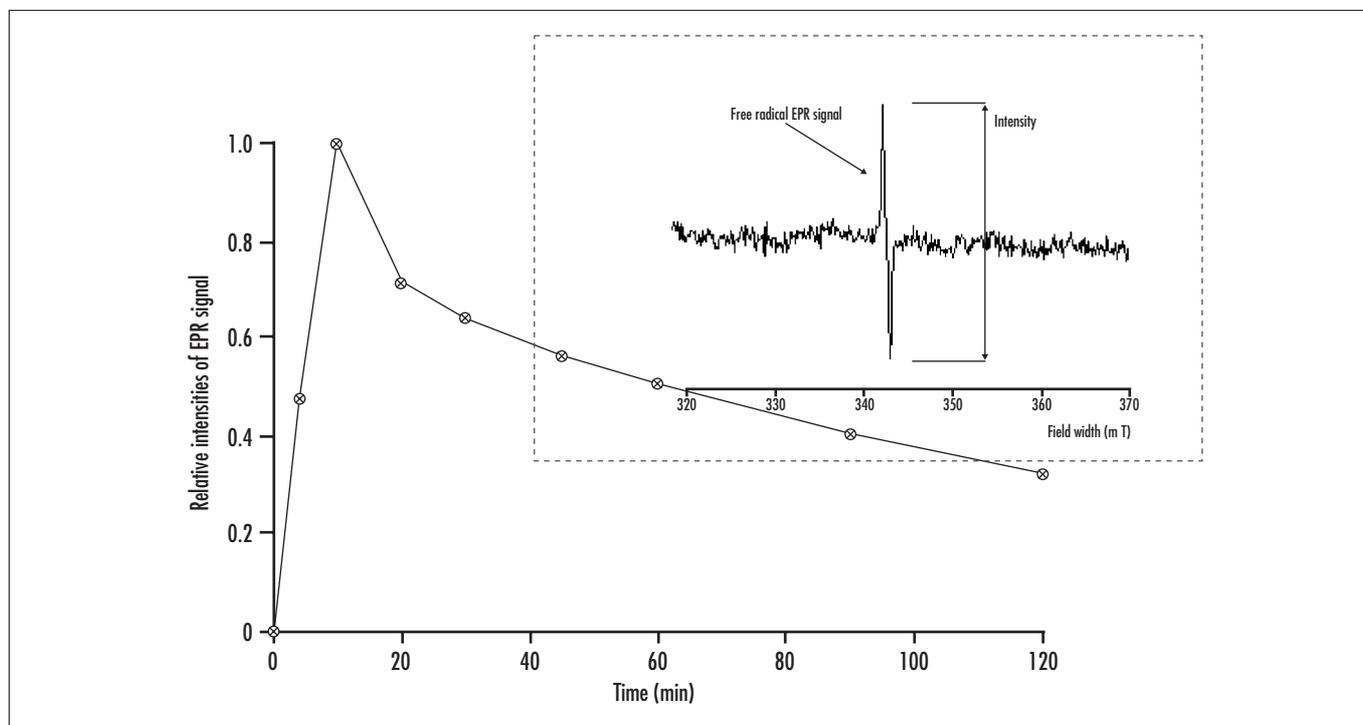
Figure 2: Relative amplitudes of the free radical resonance line in a treated spruce wood, in dependence of the reaction time with a 10% solution of ethanolamine

free radical signal were measured with the Bruker spectrometer software considering the difference between the minimum and maximum of the spectrum (see Figure 2). In such a narrow signal, as found for the free radical, the intensities of the signal corresponded well with the amount of free radicals in the wood.[5]

Nitrogen analysis in the fumed specimens

Fine sawdust of wood (Mesh 50) was exposed to ammonia or ethanolamine vapours. Following two months of conditioning in a dark and dry place, 0.2g of a dry sample was combusted in the oxygen atmosphere at 1350°C in the LECO 2000-CNS analyser. The content of nitrogen was determined in the thermocombustion cell. The experiment was performed in three parallel measurements.

Measurements of colour changes as a result of antiquing and during exposure to various conditions

Surface colours of the samples were determined with the colour difference measuring instrument dr Lange Micro Color (d/8° measuring geometry, 10° standard observer, D65 standard illuminant, xenon flash lamp source, CIELAB system). In the CIELAB system, the L* axis represented lightness (L* varied from 100 (white) to zero (black)), and a* and b* were the chromaticity coordinates. (+a* was for red, –a* for green,

+b* for yellow, –b* for blue). The obtained L*, a* and b* values were used to calculate colour changes ΔE according to equations 1, 2, 3 and 4.[6]

$$\Delta L^* = L^*_2 - L^*_1 \qquad \text{Equation 1}$$

$$\Delta a^* = a^*_2 - a^*_1 \qquad \text{Equation 2}$$

$$\Delta b^* = b^*_2 - b^*_1 \qquad \text{Equation 3}$$

$$\Delta E = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \qquad \text{Equation 4}$$

ΔL*, Δa* and Δb* were the differences between the colour values of the samples after fuming (or after UV irradiation, surface finishing or during storage in dark or exposure to normal light conditions in a laboratory) and of the initial colours, before a specific experiment. The subscript 1 in Equations 1 to 4 refer to original values before treatment or exposure, causing a colour change; the subscript 2 is for the values at the end of the test. A low ΔE value corresponded to a low colour difference. In order to interpret the colour measurement results correctly, a multifactor Anova statistical analysis[7] was performed.

Various colour measurement programs were chosen, according to the expected results, as described below:

Preliminary patination experiments and UV-irradiated specimens

Colours of ammonia and ethanolamine-treated spruce and cherry wood, as well

as UV-irradiated samples, were measured before and after treatments. Three measurements were performed on each sample and the experiments were carried out on three samples.

Changes of colour of fumed spruce wood samples

Three specimens were taken for each case and colour was measured on ten spots on each sample; altogether 30 results were obtained for each set of conditions. ΔE as a result of exposure to ethanolamine vapours was determined one hour after exposure. Untreated and treated samples were then divided into two groups: one group of both types of specimen was kept in the dark and the other group in a laboratory with normal working illumination conditions. Colour measurements were performed after 1, 2, 3, 4, 6, 8 and 12 weeks of exposure.

Changes of colour of fumed and surface-treated cherry wood specimens

Three wooden plates were used for one set of treatment and exposure conditions, with three measurement spots, yielding nine sets of L*, a*, and b* data for each combination. ΔE values were determined firstly six days after fuming. As previously described, one group of specimens was then kept in the dark and the other group exposed to light in a laboratory. In the first week, the authors measured the colours of uncoated samples every day, and then 7, 14, 21, 28,

Table 1: Colours of the amine treated and UV-irradiated cherry wood

Staining method	Treatment time (h)	Cherry wood				Spruce wood			
		L*	a*	b*	ΔE	L*	a*	b*	ΔE
Exposed to ammonia vapours	2	50.8	12.3	18.5	19.5	70.4	5.6	22.6	12.4
	8	52.1	12.4	19.5	18.0	70.8	5.6	21.7	12.1
	16	45.3	11.7	17.2	25.1	69.2	5.6	21.5	13.6
Exposed to ethanolamine vapours	8	65.4	12.7	26.8	4.8	81.3	7.7	24.3	2.0
	16	63.4	13.3	29.7	8.3	78.2	10.1	30.3	9.0
UV-irradiated	7	63.3	14.2	29.3	8.2	77.5	9.1	32.1	10.7
	30	57.1	14.2	29.3	13.4	77.0	9.5	33.5	12.2
Brushed with water	/	67.7	11.4	23.5	2.1	82.0	5.2	30.1	7.5
Untreated	/	69.4	12.2	24.2	0.0	82.6	7.2	23.0	0.0

35, 42, 109, 141, 171, 200, 232 days after the first colour measurement and, in some cases, also after 291, 323 and 353 days. The same measurement programme was applied also to surface-finished samples, with the exception that the first colour measurement took place 13 and not six days after ethanolamine treatment to ensure that the coatings were sufficiently dry.

Results and Discussion

Possibility of wood darkening with ethanolamine vapours

The results for the fumed, stained and UV-irradiated European cherry (*Prunus avium*) and Norway spruce (*Picea abies*) wood are presented in Table 1. The most prominent colour changes of cherry wood were observed after fuming with ammonia. ΔE of 19.5, obtained after two hours of fuming, exceeded the values of all other specimens treated with ethanolamine. After 16 hours of exposure to ammonia vapours, ΔE value of 25.1 was reached. It was presumed that the lower colour differences caused by ethanolamine vapour treatment most likely originated in the much lower vapour pressure of ethanolamine compared with the vapour pressure of ammonia. While above a 25% solution of NH_3, ammonia vapour pressure was 1013kPa (25°C),[8] above 100% liquid, the vapour pressure of ethanolamine had a value of only 0.5kPa (20°C).[9] A comparison with the colours of the UV-irradiated wood showed that the exposure of cherry wood to UV light and a 16-hour exposure of the samples above 10% solution of ethanolamine resulted in practically the same colour. This important result proved the presumption that the aged appearance of wood surfaces may be obtained by fuming with ethanolamine, which is less harmful and more user-friendly than the most established fuming agent, ammonia.

The data in Table 1 clearly shows that treatment with amines induced colour changes also in spruce wood. In general, the colour differences between treated and untreated spruce wood were smaller than those of cherry wood. The relationship between the ΔE values for spruce wood, treated with ammonia or ethanolamine vapours, were the same as in the case of cherry wood.

Chemical changes in antiqued cherry and spruce wood

While reactions between ammonia and wood are well described,[10,11] only some preliminary work on interactions between ethanolamine and wood has been carried out in the authors' laboratories.[12,13] Therefore, they wanted to gather additional information on the mechanisms occurring between wood and ethanolamine by FT-IR investigations of fumed wood.

The representative FT-IR spectrum of ethanolamine-treated spruce wood is presented in Figure 1. The features of FT-IR spectra of ethanolamine-treated cherry wood are very similar. The most prominent change as a consequence of ethanolamine (and also ammonia) treatment was a substantial decrease or even disappearance of the band at 1730cm⁻¹. This band was assigned to C=O stretch in unconjugated ketone, acetyl or carboxyl groups.[11,14,15] It is believed that the band at around 1730cm⁻¹ arises predominantly from –C=O groups in xylanes.[14,16–18] Thus, the observed differences of FT-IR spectra after fuming was most probably evidence of the reaction between ammonia or ethanolamine and –C=O groups in hemicelluloses, presumably xylanes. As suggested by Pawlak and Pawlak,[11] there are three types of chemical reactions that are most likely to occur between ammonia and wood. Firstly, transformation of carboxylic groups into carboxylate anions was proposed. This possibility is strongly supported by Zhang and Kamdem,[19] and Craciun and Kamdem.[20] Secondly, aldehyde and ketonic groups can produce imines and, finally, ester functional groups will react to form amides. The authors believe that these three reactions were also possible when cherry and spruce wood specimens are exposed to ethanolamine.

In contrast to a very significant influence of ammonia or ethanolamine on the FT-IR spectra of treated wood, FT-IR techniques did not provide any information on the processes caused by the UV irradiation of wood. The authors believe that the absence of changes in the vibrational spectra was due to the very short irradiation times during the experiment (only 7 or 30 hours compared with the reported 200 or 1080 hours of artificial weathering[14,18] and three months of outdoor exposure.[21] All the previous studies[11,19,20] reported changes in vibrational spectra because of exposure to UV light.

The data on nitrogen content in fumed cherry and spruce wood (see Table 2) were consistent with the colour difference meas-

Table 2: Nitrogen content in ammonia and ethanolamine treated cherry and spruce wood

Fuming agent	Fuming time (h)	Nitrogen content (%)	
		Cherry wood	Spruce wood
Ammonia	8	0.586	0.251
	16	1.263	0.453
Ethanolamine	8	0.142	0.081
	16	0.151	0.083
Water (control)	8	0.137	0.063
	16	0.133	0.072

urements, illustrating the reaction intensity between ammonia or ethanolamine and wood. Where larger colour difference (ΔE) values were observed, higher amounts of nitrogen were detected. For instance, 16-hour fuming of cherry wood with ammonia resulted in a ΔE of 25.1 and in the largest nitrogen content of 1.263%. On the other hand, concentrations of nitrogen were lower in spruce wood (up to 0.453%), where also minor colour differences were noticed compared with the fumed cherry wood specimens.

It is well known that the carbonyl and azomethine groups (-CH=N-), especially when attached to ring structures, belong to chromophores. The azomethine groups may be found in imines, which are possible reaction products between aldehyde or ketonic groups and amines. The amino and hydroxyl groups are auxochromes.[22] Aldehyde, ketonic and hydroxyl groups are present in wood and the hydroxyl and amino group in ethanolamine. These functionalities are involved in chemical changes during the fuming of wood with ethanolamine and might be thus directly responsible for the observed colour changes. However, at the moment there is no direct proof for such a statement.

It has been proposed that the discoloration of wood, exposed to sunlight, is a consequence of the modification of the chromophore groups in lignin.[23,24] The colour of weathered wood changes towards yellow to brown due to the photo-oxidation of lignin and wood extractives. Coloured quinones are formed during this process.[25] The process is driven by free radicals and their formation is promoted by UV light.[26] It was previously mentioned that in some experiments the colours of patinated and UV-irradiated wood were very similar. So, the possibility was examined of the free radical formation in wood, treated with ethanolamine. EPR spectra of wood very often contains the free radical resonance line at $g_0 = 2.002$ (see Figure 2) supposed to originate from hydroxyl, carbonyl-centred and superoxide radical adducts, promoted by UV irradiation.[24,25] The spruce wood sawdust was placed into a resonator of the EPR spectrometer and then a 10% ethanolamine solution in water was added. Subsequent periodic EPR measurements of a signal amplitude (as shown in Figure 2) exhibited a substantial and fast increase of the free radical resonance line intensity (see Figure 2), followed by a gradual decrease after the first 15 minutes. As the sample in the resonator was in the dark, it is believed that the observed increase originated from the ethanolamine/wood chemical reaction. The tentative presumption is that ethanolamine in wood might in some way induce chemical changes, similar to the ones occurring during photodegradation, including the formation of free radicals. Once the concentration of free radicals was increased, they behaved just the same as if they were promoted by UV light. A 'free radical mechanism' oxidation may result in a formation of quinones that are responsible for darkening the wood. To check this speculation, additional and extensive research work is needed.

Colour changes of fumed spruce wood

40 hours of exposure of spruce wood to ethanolamine vapours resulted in the following colour changes: $\Delta L^* = -5.8$, $\Delta a^* = 3.9$, $\Delta b^* = 8.0$, $\Delta E = 10.6$. Afterwards, during storage in the dark or when exposed to light, the most intensive subsequent colour changes occurred in the first two weeks after treatment (see Figure 3). Further changes of colour, up to the 12th week after treatment, were almost negligible. In total, during storage
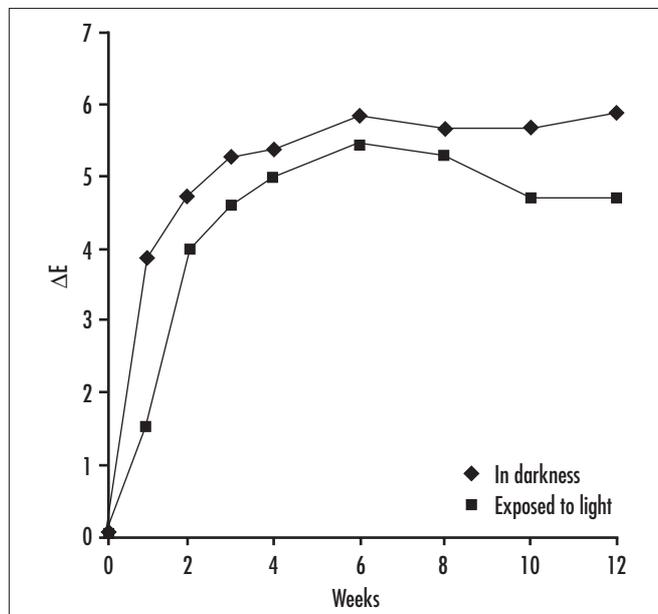

Figure 3: Colour changes of ethanolamine treated spruce wood during storage in the dark and during exposure to light in laboratory

in the dark, the ethanolamine treated specimens became slightly lighter, less red and more yellow ($\Delta L^* = 0.6$, $\Delta a^* = -2.6$, $\Delta b^* = 5.2$, the final $\Delta E = 5.9$) On the other hand, the samples exposed to light in the laboratory became darker and again less red and more yellow ($\Delta L^* = -1.3$, $\Delta a^* = -0.9$, $\Delta b^* = 4.5$, $\Delta E = 4.7$). To conclude, the colour of the fumed spruce wood was still changing intensively two weeks after treatment with ethanolamine, and later, the rate of changes decreased. The authors believe that this fact reflects some chemical processes between wood and ethanolamine that last quite some time after treatment. It has to be noted that during storage in the dark or during exposure to light, the untreated wood exhibited some colour changes (after 12 weeks: $\Delta E = 2.7$ and $\Delta E = 3.3$, respectively).

Colour properties of ethanolamine treated and surface-finished cherry wood

For this set of experiments, cherry wood specimens were exposed to ethanolamine vapours for four days. Fuming resulted in a substantial colour difference of $\Delta E = 14.7$ ($\Delta L^* = -12.9$, $\Delta a^* = 3.3$, $\Delta b^* = 1.1$). As expected, the application of various surface coatings onto controlled non-stained wood as well as on ethanolamine treated samples changed the appearance of the surfaces (see Table 3). Very similar effects were caused by linseed oil varnish and by linseed oil, both on untreated and treated samples, with ΔE values even exceeding those obtained by fuming. Beeswax and a nitro-cellulose lacquer had less prominent effects but again approximately the

Table 3: Colours of ethanolamine untreated and ethanolamine treated cherry wood and colour changes after application of surface coatings

Surface finish	Untreated cherry wood				Treated cherry wood			
	L^* 73.8	a^* 10.9	b^* 22.3		L^* 60.9	a^* 14.2	b^* 23.4	ΔE 14.7
	ΔL^*	Δa^*	Δb^*	ΔE	ΔL^*	Δa^*	Δb^*	ΔE
Linseed oil varnish	−12.0	6.6	10.2	17.1	−11.7	8.0	4.8	15.3
Linseed oil	−9.7	5.6	10.4	15.2	−9.2	7.3	7.1	13.9
Beeswax	−4.6	1.5	3.8	6.1	−4.1	2.7	2.1	5.4
Nitro-cellulose lacquer	−5.6	2.0	5.7	8.2	−4.7	3.5	3.4	6.8

same effect on untreated and fumed wood. An important conclusion is that previous fuming of the cherry wood substrate does not influence the colour changes caused by the application of coatings.

The graph in Figure 4 represents a summary of extensive work in order to determine colour changes of the untreated (unexposed to ethanolamine) and uncoated or surface-coated specimens during exposure to light in the laboratory. Three statistically significant different results were obtained. Almost the same and very low colour changes after 232 days were observed in linseed oil varnish (LV) and beeswax (BW) (ΔE = 4.7 and 4.3, respectively). Somewhat higher differences were observed during exposure of ethanolamine untreated samples, coated with nitro-cellulose (NC) lacquer (ΔE = 6.1), or linseed oil (ΔE = 5.9). Not surprisingly, the uncoated specimens exhibited the highest change of colour (ΔE = 7.5). The curves of uncoated and NC specimens for additional exposure periods of up to 353 days showed that at least for these two treatment types, the colour became stable.
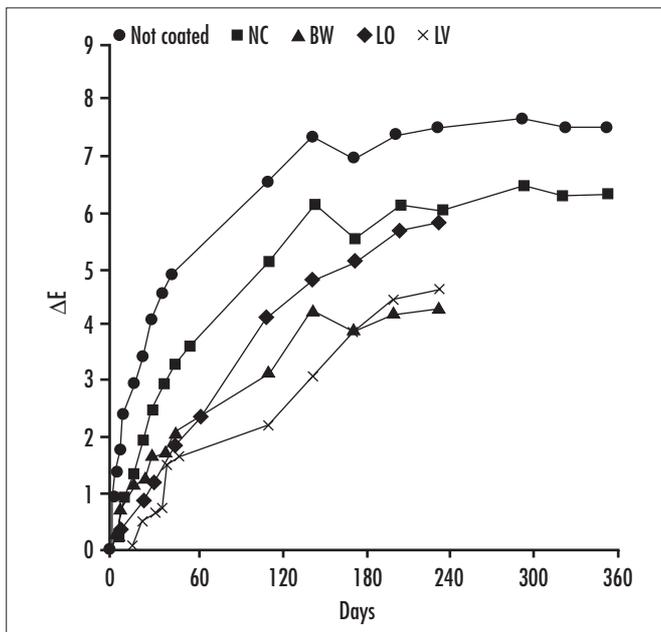

Figure 4. Changes of colour of untreated cherry wood, coated with linseed oil varnish (LV), linseed oil (LO), beeswax (BW) and nitro-cellulose lacquer (NC) during exposure to normal illuminating rhythm in a laboratory

Similarly as described above, we also investigated the antiqued (fumed) coated and uncoated specimens (see Figure 5). The first conclusion was that in 232 days of exposure to light, the colours of the fumed finished samples changed less then the unfumed ones. Among the fumed and coated samples, the smallest colour differences were observed at nitro-cellulose lacquer coated wood (NC). In contrast to unfumed and linseed oil varnish-treated cherry wood specimens (LV), ethanolamine antiqued wood, coated with this finish, exhibited the largest colour change (ΔE = 6.2). It is interesting that the colour of uncoated ethanolamine treated wood after 353 days of exposure exhibited the lowest colour change (ΔE = 3.8). All ΔE values were calculated with the starting state before 232-353 days of exposure. That means that once wood changed its colour because of exposure to
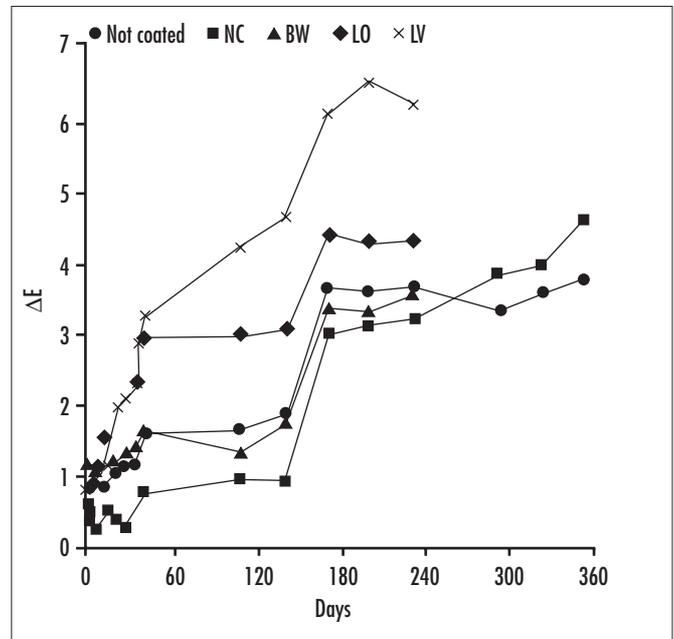

Figure 5. Changes of colour of fumed cherry wood, coated with linseed oil varnish (LV), linseed oil (LO), beeswax (BW) and nitro-cellulose lacquer (NC) during exposure to normal illuminating rhythm in a laboratory

ethanolamine, this colour remained relatively stable over a longer period.

Finally, an analysis of the colour changes of the same systems that were kept in the dark during the colour measurement period was performed. A comparison of ΔE values of fumed and coated cherry wood samples after 232 days of storage in the dark (see Table 4) was made with those obtained of the samples that were exposed to the light and already described above, which showed some interesting points. While the colour change of ethanolamine untreated and uncoated samples was very low in the dark, much higher differences were observed after exposure to light (ΔE = 7.5), which was an expected result. On the other hand, the antiqued cherry wood exhibited very low and similar ΔE values both after exposure in the dark or in the light (ΔE = 3.3 and 3.7, respectively). Ethanolamine treatment of wood also significantly improved the colour stability of nitro-cellulose lacquer-coated wood (ΔE = 3.2 on light, compared with ΔE = 6.0 at NC coated unfumed wood).

Conclusions

The appearance of age or the so-called 'patina' of cherry and spruce wood may be obtained by fuming with ethanolamine. Therefore, for wood darkening, aggressive and toxic ammonia could be replaced by this more user-friendly chemical. FT-IR measurements of the treated wood and the determination of its

Table 4: Comparison of ΔE values of ethanolamine untreated or ethanolamine treated and coated cherry wood samples after 232-day storage in dark or exposure to light

Surface finish	Untreated cherry wood		Treated cherry wood	
	ΔE (in dark)	ΔE (on light)	ΔE (in dark)	ΔE (in light)
Without surface finish	2.7	7.5	3.3	3.7
Linseed oil varnish	5.6	4.7	4.6	6.2
Linseed oil	3.9	5.9	5.3	4.3
Beeswax	2.5	4.3	2.8	3.6
Nitro-cellulose lacquer	2.0	6.0	3.4	3.2

nitrogen content brought the conclusion that the important reaction between amines and wood components must involve hemicellulose carbonyl groups. There are some indications that the mechanisms of photo- and amine-induced wood discoloration might have similar mechanisms, involving free radicals. Extensive colour stability experiments of the fumed cherry wood that was surface finished showed that sometimes, staining with ethanolamine may improve the colour stability; a typical example is wood coated with a nitrocellulose lacquer. The authors believe that they have shown that fuming with ethanolamine vapour is a new possibility for producing various wood products and furniture with the appearance of age.

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