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Upgrading of spruce wood with ethanolamine treatment

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Wood is the most abundant non-toxic, recycable and biodegradable natural material, valued as a construction material because of its appearance and its high strength at low density. On the other hand its biodegradability is an important drawback. Improvement of decay resistance of wood with ethanolamine is described in this paper. Ethanolamine chemically reacts with wood (Norway spruce sapwood) and does not evaporate from it. During this reaction, significant amounts of oxygen are consumed. Upgraded specimens show increased fungal activity and increased combustibility. The treatment of the spruce wood samples with aqueous solutions of ethanolamine resulted in colour changes.

Qualitätsverbesserung von Fichtenholz durch Behandlung mit Ethanolamin

Holz ist einerseits der häufigste, nicht-toxische, recycelbare und biologisch abbaubare Wertstoff, insbesondere für Konstruktionszwecke aufgrund seines Erscheinungsbildes, seiner hohen Festigkeit und geringen Dichte. Andererseits ist seine biologische Abbaubarkeit ein großer Nachteil. Verbesserung der Resistenz gegen biologischen Abbau von Holz mittels Ethanolamin wird in dieser Arbeit beschrieben. Ethanolamin reagiert chemisch mit Holz (Fichtensplintholz) und verdampft deswegen nicht mehr. Während dieser Reaktion werden signifikante Mengen an Sauerstoff verbraucht. Die so behandelten Proben zeigen eine erhöhte Resistenz gegen Pilzabbau sowie eine verstärkte Brennbarkeit. Die Behandlung der Fichtenproben mit wässrigem Ethanolamin ruft zudem Farbänderungen hervor.

1
Introduction

For many applications and for various different reasons wood is being preferred over many other materials (Fengel and Wegener 1989; Walker et al. 1993; Tjeerdsma et al. 1998). Its biodegradability is an important drawback for an extensive use of wood. The other significant disadvantage of wood is its low dimensional stability. This has led to intense research for new means to upgrade wood. Chemical modification or upgrading of wood should result in an improvement of its biological and technological properties (Goethals and Stevens 1994) and should not increase the flammability of wood. The theory of the improving resistance of modified wood against fungal deterioration is not completely clarified. The principle of upgrading is to react such chemicals within timber that cannot be utilized as a food source by wood decay organisms, or such that the fiber saturation point of wood is reduced below a level, necessary for biological deterioration (Rowell 1996; Tjeerdsma et al. 1998). Using toxic fungicides (e.g. copper, chromium, PCP) is not regarded suitable for wood modifications. (Suttie et al. 1997) The most applicable methods for the upgrading of wood are acetylation with acetic anhydride, (e.g. Goldstein et al. 1961; Rowell et al. 1986), a combination of oil and heat treatment (Sailer et al. 2000) and thermal treatment (e.g. Hillis 1984; Boonstra et al. 1998). There have been attempts to upgrade wood with ammonia treatment (Graham et al. 1987), but such modified wood has seen limited commercial acceptance due to unpleasant ammonia emissions. Ethanolamine (EA) (2-aminoethanol, C_2H_7NO) seems more suitable for this purpose. EA chemically reacts with wood and most of EA does not evaporate from it (Loconto and Kamdem 1998; Humar and Petrič 2000a). From FTIR spectra it can be seen that EA mainly reacts with C=O groups of hemicelluloses and 1, 3, 4 benzene ring groups in lignin complex (Humar and Petrič 2000b; Zhang and Kamdem 2000a), but the complete reaction is still not completely understood.

In this paper, we want to elucidate the chemical reaction between wood or its components and ethanolamine. We also propose a new possibility of upgrading of wood. The suggested method is very economical, as we do not need an expensive heat treatment. We studied visual appearance and fungicidal properties as well as combustibility of ethanolamine-modified wood.

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2
Material and methods

2.1
Measurements of O_2 concentrations during fixation of ethanolamine in wood

Norway spruce (*Picea abies* Karst) sapwood was ground into sawdust (mesh 40). Afterwards, 20 g of air-dry sawdust was put into a custom designed measuring chamber

(V · 400 ml) and immersed with 40 ml of preservative solution. The measuring chamber was made using metalwork (Metalwork, Italy) valves, Manesmann-Rexroth (Germany) adaptors and Rectus (Germany) connectors. All of the tubes in the system were made of polyurethane plastic. To improve sealing, silicon vacuum paste (Silicon high vacuum grease medium, Merck, Germany) was used on all connections and contact surfaces. The chamber is more precisely described in Tavzes and co-workers (2001). For treatment, 20% aqueous solutions of ethanolamine (Merck 1.00845.1000) were used ($c_{EA} = 20\%$).

Additionally, the same experiment was repeated with brown rotted wood (BRW) as a model for lignin and with Na-cellulose as a model for cellulose. BRW was obtained from untreated wood, which had been exposed to brown rotting fungi (*Coniophora puteana* ZIM L009 and *Gloeophyllum trabeum* ZIM L017 (Raspor et al. 1995)) for 4 month.

With this experiment, we wanted to find out how oxygen concentrations decrease within time of reaction between wood and ethanolamine and which wood components are involved in this reaction. Therefore, measurements were performed immediately after impregnation with preservative solutions. A galvanic sensor was used for the detection of O_2 in the system (1–25%; ±0.01%). The concentrations of O_2 in the closed system were measured every 10 s for at least 7 hours. This method is more precisely described in (Humar et al. 2001). All measurements were performed in three parallels.

2.2
Elemental (CHN) analysis of ethanolamine treated wood

Elemental analysis of ethanolamine treated and untreated Norway spruce sawdust (Mesh 100) was performed. The experiment was repeated in three parallels. 0.5 g of oven dry sawdust was immersed with 0.5 ml of ethanolamine based aqueous solution. The following concentrations of ethanolamine were used: 5, 10 and 20%. The control samples were impregnated with distilled water only. Immediately after this treatment, we placed the treated sawdust into closed tubes for two weeks. Afterwards we let this sawdust dry for additional two weeks. Then we dried these samples in an oven at 103 °C for 24 hours and after that CHN analysis was performed. Because of great variability and various uncontrolled factors, we did not consider carbon and hydrogen content and thus, only nitrogen content is discussed within this paper.

2.3
Fungicidal activity of upgraded wood

The norway spruce sapwood (*Picea abies* Karst) samples were 0.5 cm × 1 cm × 3 cm (longitudinal direction) sized and vacuum impregnated with ethanolamine based aqueous solutions according to the EN 113 procedure (ECS 1989). The concentrations of ethanolamine in preparations were 3.3, 8.3 and 20% m/m. For comparison, some samples were treated only with distilled water. The treatment of small blocks resulted in a solution uptake of about 77% of the oven dry wood mass. After impregnation, the samples were drying for three weeks, the first week in closed chambers, the second week in half closed and the third week in opened chambers. Dry samples were then exposed to wood rotting fungi.

We used four species or strains of fungi. The white rot species was *Trametes versicolor* (L.: Fr.) Pilát (a recently isolated strain). The other fungi were brown rot species. We used *Gloeophyllum trabeum* (Pers. Ex Fr.) Murill (ZIM L017) (Raspor et al. 1995) and two isolates of the same species *Antrodia vaillantii* (DC.: Fr) Ryv. (P163 HUM UH) and ZIM L037) (Raspor et al. 1995). The first strain had been kindly provided by O. Schmidt, University of Hamburg. The cultures were maintained on solid plating media, which contained 3.9% potato dextrose agar (PDA Difco). Petri dishes with PDA medium were inoculated with small pieces of fungal mycelium. Afterwards, we put four samples of treated and of untreated wood into each inoculated petri dish. The samples were exposed to fungal decay (dark, T = 25 °C, RH = 95%) for 8 weeks.

2.4
Anti blue stain efficiency of upgraded wood

For this experiment, pine sapwood (*Pinus sylvestris* L.) was used instead of Norway spruce, as pine sapwood is much more susceptible towards staining than spruce wood (Richardson 1993). The experiment was performed according to the standard EN 152-1 (ECS 1996). The dimensions of samples were 1 cm × 4 cm × 11 cm (longitudinal direction). The samples were brushed with two ethanolamine aqueous solutions ($c_{EA} = 1.6\%$ and 20%). The uptake of each type of a solution was approximately 180 g/m^2. After brushing, we let samples to dry for 3 weeks. Afterwards, the samples were exposed to blue stain fungi for 6 weeks, according to the standard EN 152-1. *Aureobasidium pullulans* (de Barry) Arnaud (ZIM L060) and *Sclerophoma pithyophila* (Corda) Hohn (ZIM L070) (Raspor et al. 1995) were used for this experiment.

2.5
Influence of ethanolamine on wood combustibility

The samples were made of Norway spruce sapwood. The dimensions of longitudinally oriented samples were 1 cm × 3.5 cm × 15 cm. These samples were vacuum impregnated with two ethanolamine based aqueous solutions, according to the EN 113 procedure (ECS 1989). The first solution contained 20% of ethanolamine and the second one contained 40% of ethanolamine. The control samples were impregnated with distilled water only. Vacuum impregnation resulted in a solution uptake of about 30% of the air-dry wood mass. After three weeks of drying in closed, half closed and in opened chambers, the samples were conditioned at 22 °C, 65% RH for 14 days.

The conditioned samples were placed above flame for 0.5 min, 1 min and 2 min. The samples were positioned in a steel tube (r = 2.5 cm, l = 17 cm) directly above the flame. The Bunsen's burner was used as the flame source. After the previously mentioned period of time, the burner was shut down. If a sample was still burning, we waited until flame died out. mass loss of cooled samples was detected gravimetrically. The experiment was performed in five parallels.

2.6
Colour changes of upgraded wood

For the determination of colour-changes as a result of ethanolamine treatment, we performed the method described below. The samples were made of Norway spruce sapwood (1 cm × 3.5 cm × 10 cm) and were vacuum impregnated with ethanolamine aqueous solution. The control samples were impregnated with distilled water. The concentrations of ethanolamine in the treatment solution were; 3.3, 8.3 and 20%. After impregnation, the samples were drying for three weeks, the first week in closed chambers, the second week in half closed and the third week in opened chambers. Afterwards, the colour analyses were performed. Six measurements were carried out per each sample. The experiment was performed in three parallels. For comparison, we measured untreated pine (*Pinus sylvestris* L.) and untreated larch (*Larix decidua* Mill.) hardwood and a more than 10 years old spruce wood which had been kept in dry place and not exposed to the direct sunlight.

The surface colours of dry samples were determined using a Colour Difference measuring Instrument MICRO COLOR (CIELAB system). CIELAB system characterized colours by three parameters: L^*, a^* and b^*. L axis represents the lightness whereas, a^* and b^* are the chromaticity coordinates. In the CIELAB coordinates, $+a^*$ stands for red, $-a^*$ for green, $+b^*$ for yellow, $-b^*$ for blue, and L^* varies from 100 (white) to zero (black). Any colour can thus be characterized (Brock et al. 2000).

L^*, a^* and b^* colour coordinates of treated and untreated specimens were obtained. These values were used to calculate the colour changes ΔE according to Eqs. 1–4 (Zhang and Kamdem 2000b).

$$\Delta L^* = L_1^* - L_2^* \quad (1)$$

$$\Delta a^* = a_1^* - a_2^* \quad (2)$$

$$\Delta b^* = b_1^* - b_2^* \quad (3)$$

$$\Delta E = \sqrt{(\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})} \quad (4)$$

Where ΔL^*, Δa^* and Δb^* stand for the change between the initial (control) and the final (treated) values. L^*, a^* and b^* contribute to the color change ΔE. A low ΔE corresponds to a low colour difference or equal colour. An equal colour is considered when ΔE is lower than 1.0 (Brock et al. 2000).

3
Results and discussion

3.1
Measurements of O_2 concentrations during fixation of ethanolamine in wood

Immediately after the treatment of spruce sawdust with ethanolamine aqueous solution (20%), a significant decrease of O_2 concentrations in the measuring chamber began. The concentration of oxygen was decreasing all the time during our measurements (at least for seven hours). In some cases, the percentage of O_2 in the chamber during the seven hours of reaction between wood and EA, decreased from the atmospheric 20.5% to 14%. This can be well seen from Fig. 1. In some additional experiments, more than 85% of oxygen was consumed in a closed system during the first 72 hours of reaction between wood and the ethanolamine aqueous solution, from normal 20.5% to 3% after 3 days of fixation.

The treatment of cellulose and BRW gave comparable results. Almost the same decreases in oxygen concentration were detected with cellulose as well as with BRW (Table 1). Therefore, we suppose that ethanolamine reacts approximately to the same extent with both major wood components, lignin and cellulose. Thus, we believe that the primary reason for the observed decrease of oxygen concentration lies in the chemical reaction of ethanolamine with wood or its components.

We do not know yet what the reason is for the described consumption of oxygen. FTIR spectra showed that there may be some reactions between ethanolamine and C=O groups in COOH groups of hemicelluloses and 1, 3, 4 benzene ring groups in lignin complex (Humar and Petrič 2000b; Zhang and Kamdem 2000a) or there is some other reason for the observed decrease.

3.2
Elementary (CHN) analysis of ethanolamine treated wood

As already suggested by Humar and Petrič (2000a and 2000b) elementary analyses confirmed, that ethanolamine remains in wood and does not entirely evaporate from it

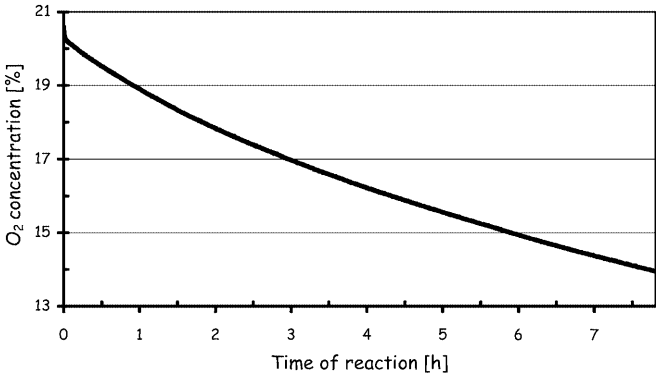

Fig. 1. Changes of oxygen concentration after the treatment of spruce dust with the aqueous solution of ethanolamine (20%)
Bild 1. Änderung der O2-Konzentratrion nach Behandlung von Fichtenholzmehl mit wässrigem Ethanolamin

Table 1. Changes of O_2 concentration in the closed measuring chamber after 7 hours of treatment of wood, brown rotted wood BRW (lignin) or cellulose with ethanolamine (20%)
Tabelle 1. Änderung der O_2-Konzentration in der geschlossenen Messkammer nach 7stündiger Behandlung von Holz, Braunfäuleholz (BRW = Lignin) bzw. Cellulose mit Ethanolamin (20%)

Treatment	Changes in O_2 concentration [%]			
	Wood	BRW	Cellulose	Control/ no substrate
Water	0	0	0	0
Ethanolamine (20%)	−3.2	−3.4	−3.3	0
Control – no treatment	0	0	0	0

(Fig. 2). We presume that the detected nitrogen in wood, originates from unevaporated ethanolamine in wood. As shown in Fig. 2, the percentage of nitrogen in wood correlates well with the concentration of ethanolamine in the treatment solution. However, in sawdust treated with a lower concentration of ethanolamine ($c_{EA} = 5\%$) greater portion of nitrogen/ethanolamine remained in wood, as compared to sawdust treated with a 20% ethanolamine containing solution. For instance, at the lowest concentration of EA in the solutions the concentration of nitrogen in wood was 1.72% whereas in sawdust treated with a four times highest concentration only 2.70% of nitrogen remained, respectively (Fig. 2). This means that some part of EA reacted with wood and remained bounded while the other part evaporated from wood. This is well seen from nitrogen concentrations in wood. The percentage of nitrogen in wood is well correlated with the already published percentage of gravimetrically determined unevaporated ethanolamine based solution in wood (Humar and Petrič 2000a) (Fig. 2).

3.3
Fungicidal activity of upgraded wood

The results of fungicidal test are summarized in Table 2. It can be seen from the data, that even the lowest concentration of EA in the preservative solution reduced decay of both white and brown rot fungi. During the exposure of wood, modified with the lowest concentration of EA (3.3% EA) to brown rotted species *Gloeophyllum trabeum,* the samples lost up to 6% of their initial mass. The controls showed a six times higher mass loss (Table 2). Other fungi *A. vaillantii* 1 and *T. versicolor* caused a similar mass loss as with wood, upgraded with the lowest concentration of EA (3.3%), namely 4% and 6%, respectively. On the other hand, *A. vaillantii* 2 was more effective. This fungus decayed 9% of the initial mass. However, these samples were

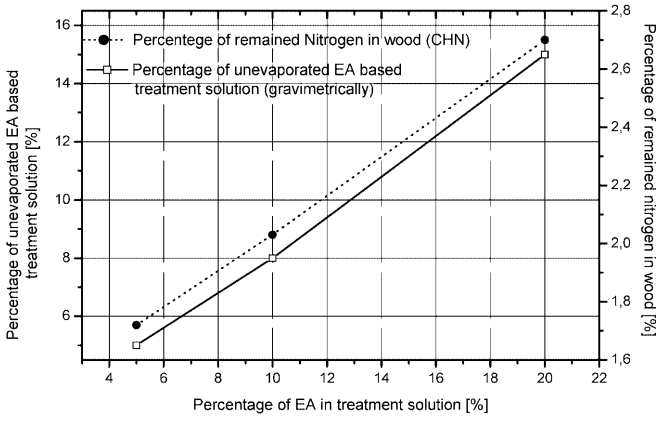

Fig. 2. Percentage of unevaporated nitrogen in wood, treated with ethanolamine water-born solutions, obtained with elemental analysis, compared with percent of unevaporated ethanolamine based treatment solution, obtained gravimetrically (Humar and Petrič 2000b)
Bild 2. Anteil an nichtverdampftem Stickstoff in Holz nach Behandlung mit wässrigem Ethanolamin. Ergebnisse der Elementanalyse im Vergleich mit Ergebnissen der gravimetrischen Analyse (Humar und Petrič 2000b)

Table 2. Mass losses of wood impregnated with ethanolamine based aqueous solutions and exposed to four different wood rotting fungi. (*A. vaillantii* 1 = P163 HUM UH and *A. vaillantii* 2 = ZIM L037)
Tabelle 2. Gewichtsverluste von Holz nach Imprägnieren mit wässrigem Ethanolamin und Behandlung mit 4 abbauenden Pilzen (*A. vaillantii* 1 = P163 HUM UH und *A. vaillantii* 2 = ZIM L037)

Fungus	Conc. of EA in the treatment solution [%]			
	3.3 mass loss [%]	8.3	20	0 – control
Antrodia vaillantii 1	4	5	3	24
Antrodia vaillantii 2	9	3	4	27
Gloeophyllum trabeum	6	5	5	36
Trametes versicolor	6	6	4	19

almost three times less decayed than the control samples. Nevertheless, a higher concentration of EA resulted in a better decay resistance against *A. vaillantii* 2. (Table 2) White rot fungi *T. versicolor* decayed 4% of wood upgraded with the 20% EA solution. A similar decay was caused by *A. vaillantii* 2, while *G. trabeum* caused 5% mass loss, respectively (Table 2).

Decay resistance of EA upgraded wood gave comparable results with other modified wood. The oil treated pine, exposed to *G. trabeum,* lost almost 5% of initial mass (Štern 2000) while heat-treated spruce wood after 16 weeks of exposure in soil block test lost 3% of its mass (Tjeerdsma et al. 1998). Still, some treatments could result in a completely terminated decay (e.g. Sailer et al. 2000).

We believe that one of the reasons for fungicidal resistance of EA modified wood originates in the alkaline pH of modified wood. The pH of spruce wood modified with the highest concentration of EA (20%) is 9.0, which is notably more than pH of untreated spruce 5.1 (Fengel and Wegener 1989; Humar et al. 1999). We think that alkaline pH of wood increases it decay resistance, as especially brown rot fungi require acid environment for their growth (Stephan et al. 1996; Humar and Pohleven 2000). If wood is alkaline enough, acidification may be hinted and decay cannot proceed. Thus, *A. vaillantii* 2, decayed more wood, impregnated with low concentration of EA than the other three wood rotting fungi, as this strain is known by its copious oxalic acid production (Humar et al. 1999).

3.4
Anti blue stain efficiency of upgraded wood

The treatment of wood with ethanolamine did not protect pine samples from blue staining. Samples, brushed with the lowest concentration of ethanolamine (1.6%) blue stained to the same extent as the control ones. Anyway, these results can be regarded as promising, as ethanolamine contains nitrogen and as fungi require nitrogen for their growth (Ruddick and Xie 1995). It is very important that ethanolamine treatment did not result in an even more intensive blue staining. On the other hand, brushing with the highest concentration of ethanolamine in the treatment solution slightly reduced blue staining.

However, it has to be stressed that the samples for this experiment were treated only by brushing. We believe that if samples had been vacuum impregnated, they would have been more blue stain resistant.

3.5
Influence of ethanolamine on wood combustibility

The presence of ethanolamine in wood did not increase the combustibility of wood. On the contrary, wood treated with ethanolamine lost less of its mass than the untreated wood. This difference was significant and well seen after 1/2, 1 and 2 minutes of burning. After 2 minutes of flaming, the untreated wood lost 63% of its mass, while the treated samples lost "only" 39% of their mass respectively. Thus, the treatment of samples with 20% aqueous solution of ethanolamine resulted in a reduced mass loss (33%) as compared to control samples. (Fig. 3). On the other hand, we could not observe any difference between wood treated with a 20% EA solution or the 40% EA solution. These results how that the upgrading of wood with ethanolamine treatment does not protect wood against fire such as, for instance commercial fire retardants, but it does substantially decrease flammability. This feature is especially important for the wood in construction use.

3.6
Colour changes of upgraded wood

The colour parameters of treated and untreated wood and colour changes (ΔL^*, Δa^*, Δb^* and ΔE) are represented in Table 3. From this table we can see that the colour of the treated samples differs from the colour of the untreated samples. This can be particularly well seen from the value of ΔE. This value (difference) is increasing with the concentration of EA in the treatment solution: from initial 14.6 (3.3% EA) to final 17.1 at wood, treated with a 20% ethanolamine containing solution. The upgraded wood becomes slightly darker as seen from the value L^*. The L^* has been changed from 84.6 at control wood to 70.4 at the samples treated with a 20% aqueous solution of EA. Wood also becomes more yellowish and reddish. This can be seen from two values, a^* and b^*. The value a^* has been changed from 5.5 at untreated wood to 10.3 at wood upgraded with a 20% solution of EA (Table 3). The colour of the treated samples is similar to the colour of valuable pine, larch hardwood, or aged untreated spruce wood (Table 3). Therefore, the observed changes showed that the treatment with ethanolamine resulted in acceptable, and in some cases in required colour changes.

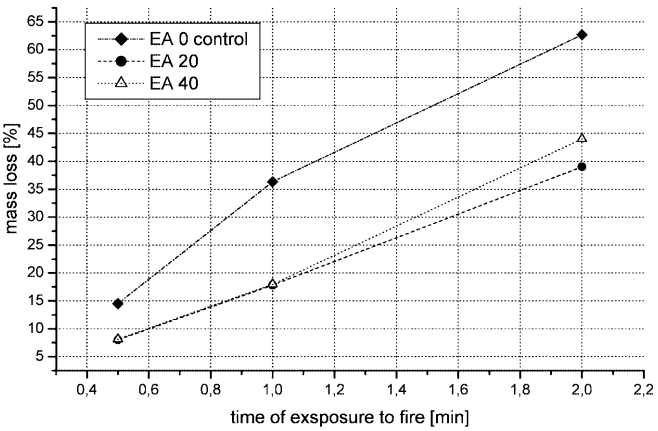

Fig. 3. Combustibility of wood treated with the aqueous solution of ethanolamine. The concentrations of ethanolamine in solutions were 20% (EA 20) and 40% (EA 40). Control samples were impregnated with distilled water (EA 0 control)
Bild 3. Brennbarkeit von Holz nach Behandlung mit wässrigem Ethanolamin. Die Konzentrationen des Ethanolamins waren 20% (EA 20) und 40% (EA 40). Kontrollproben waren mit dest. Wasser behandelt (EA0)

Table 3. Colour changes, caused by treatment with aqueous solutions of ethanolamine (c_{EA} = 0, 3.3%, 8.3% and 20%) and comparisons (larch, pine and aged spruce wood). ΔL, Δa, Δb and ΔE are calculated as shown in Eqns. 1–4
Tabelle 3. Farbänderungen durch Behandlung mit wässrigem Ethanolamin (c_{EA} = 0, 3.3%, 8.3% and 20%) und Vergleichsproben (Lärche, Kiefer und älterer Fichte). ΔL, Δa, Δb und ΔE sind berechnet nach Gleichung 1–4

Treatment	L^*	ΔL	a^*	Δa	b^*	Δb	ΔE
0% EA control	84.6	0.0	5.5	0.0	19.8	0.0	0.0
3.3% EA	75.9	8.7	7.7	−2.2	31.3	−11.5	14.5
8.3% EA	74.7	9.9	8.5	−3.0	30.4	−10.6	14.8
20% EA	70.4	14.2	10.3	−4.8	29.3	−9.5	17.1
Pine	71.6	13.0	11.9	−6.4	26.6	−6.8	16.0
Larch	72.3	12.3	12.1	−6.6	25.6	−5.8	15.1
Aged spruce	72.5	12.1	12.1	−6.6	34.8	−15.0	20.4

4
Conclusions

Ethanolamine chemically reacts with wood. This can be seen from the significant consumption of oxygen that appeared during this reaction. This is supported by some FTIR measurements. more than 85% of oxygen was consumed during the first 72 hours of reaction between wood and the ethanolamine containing preservatives, from normal 20.5% to 3% after 3 days of fixation. The treatment of cellulose and lignin models gave comparable results. The increased nitrogen content in wood is another proof for the reaction between ethanolamine and wood. The amount of bound nitrogen/ethanolamine is strongly correlated with the concentration of ethanolamine in the treating solutions.

Wood, upgraded with ethanolamine, showed increased fungicidal resistance. The specimens treated with the 20% aqueous solution of ethanolamine were at least five times less decayed than the untreated control samples. Additionally, the samples which were upgraded with ethanolamine, performed better combustibility resistance as well. This is particularly important as upgraded wood may be used for construction purposes. The colour of treated wood is slightly changed. The figure of treated specimens seems like the figure of naturally aged surfaces. Therefore, we believe that the ethanolamine treatment results in acceptable and in some cases in required colour changes.

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