

# Weathering of silane treated wood

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**Abstract** Outdoor and artificial weathering tests were performed on specimens of Scots pine sapwood treated with silanes to achieve water repellent properties. During outdoor weathering, the treatment caused a reduction in (liquid) water uptake but the sorption of moisture (air humidity) was not affected by silane treatment. The water repellent effect of vacuum-pressure treated specimens remained stable over the tested exposure period of one year, as was shown in dipping tests.

Evaluation of silane treated specimens did not reveal any reduction of crack formation during exposure to weathering. The application of silanes in combination with UV light stabilizers enhanced the effectiveness of these protectants and led to higher colour stability during artificial weathering in a QUV device.

## Bewitterung von mit Silanen behandeltem Holz

**Zusammenfassung** Zur Erlangung Wasser abweisender Eigenschaften wurden Kiefernspinnholzproben mit Silanen behandelt und einer natürlichen und künstlichen Bewitterung ausgesetzt. Die Behandlungen bewirkten eine verringerte Aufnahme von (flüssigem) Wasser bei der Außenbewitterung; jedoch wurde die Feuchtesorption (durch Interaktion mit der Luftfeuchte) nicht beeinflusst. Im Laufe der Bewitterungszeit von einem Jahr erwies sich der Wasser abweisende Effekt bei Vakuum-Druck-behandelten Proben in Tauchversuchen als stabil. Es wurde kein Einfluss

auf die Bildung von Oberflächenrissen während der Bewitterung festgestellt. Die Kombination von Silanen mit UV-Stabilisatoren verstärkte die Wirkung dieser Mittel und führte zu verbesserter Farbstabilität bei künstlicher Bewitterung.

## 1 Introduction

Water repellents are supposed to protect wood products exposed to weathering over long periods. The prevention of liquid water uptake from rain and dew can lead to higher resistance against surface erosion and leaching of wood components. In addition, water repellent treatment lowers the moisture level of wood in water submersion tests (Donath et al. 2005), and thus decreases the tendency of crack formation and the risk of biological degradation. Combinations of active substances such as fire retardants, biocides or UV light protectants with water repellents reportedly prevented these agents from leaching (Rowell and Banks 1985, Zahora 1991, Zahora 1992, Zahora 2000, Williams and Feist 1999, Evans et al. 2003, Schulte et al. 2004, Mai and Militz 2004a).

Alkoxysilanes are a group of chemicals which are unstable in water and undergo the so called sol-gel-process upon hydrolysis. This generates the formation of silanols which subsequently condense to three-dimensional units. Condensation already starts while the hydrolysis is still in process leading first to the formation of colloid oligomeric particles called sols. The further reaction causes cross-linking of these sol particles so that highly condensed gels are formed (Brinker and Scherer 1990, Mai and Militz 2004b).

Silanes treatment has been shown to increase the water repellence of wood (Tshabalala and Gangstad 2003, Tshabalala et al. 2003, Donath et al. 2005) and to enhance the efficiency of biocides and fire retardants (Saka and Tanno

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1996, Saka and Ueno 1997, Tanno et al. 1998, Saka et al. 2001). In contrast to other water repellents, such as oils or waxes, silanes do not strongly influence the sorption behaviour of wood. Treated wood remains open to moisture exchange and, therefore, the equilibrium moisture content and the moisture flow rates are comparable to untreated wood (Donath et al. 2004).

In this study, water repellent treatments based on monomeric alkoxy silanes and multifunctional oligomeric silane systems were tested for their ability to enhance the performance of wood during outdoor and artificial weathering.

## 2 Experimental

### 2.1 Wood specimens

Tests were performed on pine sapwood (*Pinus sylvestris* L.) specimens, free of knots and straight of grain, with a size of  $15 \times 75 \times 270 \text{ mm}^3$  (rad  $\times$  tang  $\times$  long). Five replicates were used for each treatment variation. After silane application and curing, the specimens were end-grain-sealed with a commercial silicone sealant.

### 2.2 Silane treatment

Three monomeric silanes, methyltriethoxysilane (MTES), propyltriethoxysilane (PTEO) and isobutyltriethoxysilane (IBTEO), and two oligomeric silane systems (Dynasytan<sup>®</sup> HS 2909 and Dynasytan<sup>®</sup> F 8815) were chosen for the treatments. Dynasytan<sup>®</sup> HS 2909 is a water borne, solvent free amino-alkyl-functional siloxane cooligomer. Dynasytan<sup>®</sup> F 8815 is an aqueous modified fluoroalkylsiloxane.

For preparation of silane-based sols, MTES, PTEO and IBTEO were hydrolysed prior to the treatment of wood. Water (2.5 mol) acidified to pH 4 with HCl (37%) was mixed with 1 mol silane and 1 wt % ethanol (related to silane) as a solvent mediator. The mixture was stirred at room temperature until it became transparent and kept without stirring for 1 h (Table 1).

Specimens for outdoor weathering tests were treated with a mixture of alkoxy silanes from MTES and PTEO, or with the aqueous functional silanes HS 2909 or F 8815, respectively. Mixtures of alkoxy silanes from MTES and PTEO (mass ratio 20 : 80) were prepared after separate pre-

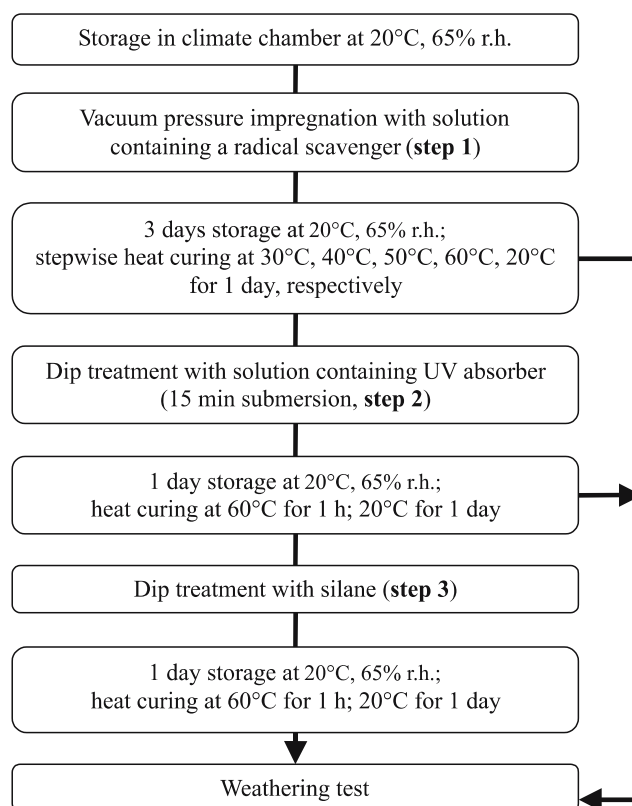
**Table 1** Preparation of silane sols

**Tabelle 1** Herstellung der Silan-Sole

Base agent	pH of water	Stirring time (min)
MTES	2.0	15
PTEO	1.5	50
IBTEO	1.5	120

hydrolysis and then diluted with ethanol to a concentration of 50 wt %.

The aqueous functional silanes HS 2909 and F 8815 were diluted with tap water to obtain active ingredient concentra-



**Fig. 1** Schedule for silane treatments. Principle of process for wood treatments with radical scavengers, UV-absorbers and silanes. Treatments were performed stepwise; the indicated steps (1–3) are related to Table 3

**Abb. 1** Schema der Silanbehandlung. Verfahrensprinzip der Holzbehandlung mit Radikalfängern, UV-Absorbern und Silanen. Die Behandlung erfolgte in einzelnen Schritten; die angegebenen Schritte (step 1–3) beziehen sich auf Tabelle 3

**Table 2** UV-light protectors applied for combination with silanes

**Tabelle 2** Zur Kombination mit Silanen eingesetzte UV-Lichtschutzmittel

Product	Function	Active ingredient	Supplier
CGL 1198	radical scavenger	hindered amine	CIBA, Basle (Ch)
Tinuvin 384-2	UV-absorber	hydroxyphenyl-benzotriazole	CIBA, Basle (Ch)
Aerosil	UV-absorber	pyrogenic silica	Degussa, Hanau (D)
RM 300 WP	UV-absorber	doped TiO <sub>2</sub> based	Sachtleben, Duisburg (D)
UVASil	radical scavenger	4(3-triethoxy-silyl-n-propoxy)-2.2.6.6-tetramethylpiperidin	Degussa, Hanau (D)

tions of 20% and 15%, respectively. The active ingredient content of both stock solutions amounted to 60% (Donath et al. 2005). All silanes were supplied by Degussa AG, Rhein-  
felden, Germany.

For artificial weathering tests, the silanes were combined with radical scavengers and UV-absorbers (Table 2). Therefore, the treatments were performed in several steps (Fig. 1, Table 3). Specimens for outdoor weathering were treated solely with silanes in a one step process. Silane treatments were carried out by vacuum pressure impregnation (vac; 100 mbar for 1 h followed by a pressure of 12 bar for 2 h), dipping for 30 min (dip) or combinations of both. After impregnation, the specimens were weighed and subsequently cured in an oven.

For determination of weight percent gains (WPG), 5 reference specimens per treatment variation were cured in an oven for 24 h at 103 °C and subsequently weighed.

### 2.3 Weathering

Prior to weathering, water up-take of the specimens was determined in a dipping test over 24 h, followed by re-conditioning for two weeks at 65% relative humidity (r.h.)

and 20 °C. Outdoor weathering was performed according to the European standard EN 927-3. The specimens were fixed on racks with a rubber band, exposing the side of the specimens to weathering which was originally facing the bark. The exposure area was positioned south-west and 45° horizontally tilted, arranged free of shadow, leaves and scrubs at the test field in Göttingen. Weathering was performed from May 2003 to August 2004. All specimens were weighed monthly. After 6 and 12 months, the specimens were stored for one week in a climate of 65% r.h. and 20 °C. The weathered surfaces were visually evaluated for cracks and subsequently water uptake was determined.

Artificial weathering was performed following EN 927-6 using a UV cabinet with spray option (QUV, QPanel, Lab Products, Cleveland, USA). The exposed specimens were alternately stressed with UV (A)-irradiation and water spraying for a total of two weeks (parameters see Table 4). Evaluation was done after each week by surface crack characterisation and water uptake testing as described above. Digital images of the specimen surfaces were recorded with a Canon CanoScan 3000 scanner (image solution: 300 dpi) and analyzed using Adobe Photoshop 7.0 software. The surface colour was determined according to the Commission

**Table 3** Schedule for silane treatments of specimens which were exposed to artificial weathering. Treatment steps for combining silanes with UV-light protectors

**Tabelle 3** Schema der Silan-Behandlung von Proben, die einer künstlichen Bewitterung ausgesetzt waren. Behandlungsschritte der Kombination von Silanen mit UV-Lichtschutzmitteln

Treatment	1. Step vacuum-pressure impregnation			2. Step dip-treatment						3. Step dip-treatment		
	Water	Medium HS 2909 20%	Agent CGL 1198	PTEO/MTES 50%	Medium IBTEO 100%	Ethanol	Tinuvin 384-2	Agent Aerosil	RM 300 WP	UVAasil	Medium HS2909 20%	F8815 15%
1	97%		3%									
2	97%		3%			97%	3%					
3	97%		3%	100%								
4	97%		3%	97%			3%					
5	97%		3%	90%				10%				
6	97%		3%	95.5%				4.5%				
7	97%		3%	90%					10%			
8	97%		3%		97%		3%					100%
9		97%	3%									
10		97%	3%	97%			3%					
11		97%	3%			97%	3%				100%	
12		97%	3%	90%				10%				
13		97%	3%	95.5%				4.5%				
14		97%	3%	90%					10%			
15		97%	3%		97%		3%					100%
16	100%											

**Table 4** Schedule for artificial weathering in a QUV weathering device. Chosen parameters realize UV-, leaching- and moisture change stresses  
**Tabelle 4** Schema der künstlichen Bewitterung in einem QUV-Gerät. Die gewählten Parameter realisieren eine UV- und Auswaschungsbeanspruchung, sowie eine Beanspruchung durch Feuchtewechsel

Reiteration	Step	Function	Temperature	Duration	Condition
1×	1	Condensation (100% r.h.)	(45 ± 3) °C	24h	
48×	2	UV (A)	(60 ± 3) °C	2.5h	wave band 300–400 nm, max. 0.77 Wm <sup>-2</sup> nm <sup>-1</sup> at 340 nm
	3	Spray		0.5 h	6 l/min to 7 l/min; UV off

International de l'Eclairage (CIE) on basis of the Lab colour space. Lightness is represented by the L axis running from black to white, chromaticity is described by the coordinates a and b running from red to green and yellow to blue, respectively. The chroma change  $\Delta C$  was calculated from the data before  $(a_1, b_1)$  and after  $(a_2, b_2)$  weathering according to the following equation:

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

### 3 Results and discussion

#### 3.1 Weight percent gain (WPG)

Treatment with PTEO/MTES caused a WPG of 22.3% (vacuum pressure impregnation) and 2.3% (dip treatment), that with HS 2909 of 11.2% (vac) and 0.2% (dip). Dip treatment with F 8815 resulted in a WPG of 3.2%.

#### 3.2 Outdoor weathering

Monthly weighing of outdoor exposed specimens revealed a mass variation for untreated specimens of more than 25 g per specimen, which made up about 35% of their initial mass (Fig. 2).

The mass variation is heavily dependent on the weather conditions before the days of weighing which are listed in Table 5. The initial state of exposure was a long period of hot days in August, with the first intense rain after several weeks falling just the night before the measurement. The mass of the untreated specimens immediately rose, while the treated specimens were protected by the silanes and did not show this sensitivity (Fig. 2(1)). In autumn, the relative

**Table 5** Weather conditions during outside exposure

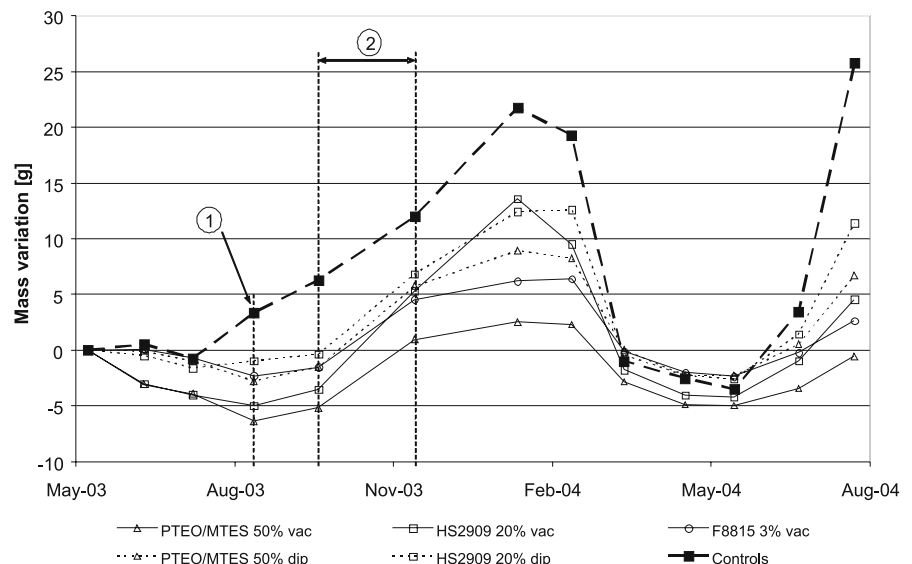
**Table 5** Wetterbedingungen während der Außenbewitterung

Date of measurement	Weather conditions before weighing
16-Jun-03	mild spring days
14-Jul-03	hot summer days
18-Aug-03	hot summer, heavy rainfall in the night before
24-Sep-03	mild autumn, sunny but also moist phases
18-Nov-03	mild wet days
16-Jan-04	rainy days, without freeze
16-Feb-04	dry in the daytime
17-Mar-04	dry in the daytime
21-Apr-04	dry in the daytime, shower the day before
19-May-04	sunny days
25-Jun-04	wet days
27-Jul-04	wet days

humidity was higher and, in this phase, all curves showed similar rise (Fig. 2(2)). These two phases illustrate that the silanes protected the wood against liquid water (rain) but almost not against moisture (air humidity), because the influence on sorption properties of wood had only been low. Over the whole exposure time, the PTEO/MTES and F 8815 had the best regulatory effect, resulting in mass variations of less than 10 g per specimen (equivalent to about 12% of the initial wood dry mass before treatment). The lower efficiency of HS 2909 in reducing moisture could be due to the absolutely unchanged sorption behaviour of the treated wood, while other treatments could have changed the equilibrium moisture content (EMC) slightly.

Water uptake tests performed before and during the time of exposure showed a high stability of the water repellent effect (Fig. 3) caused by vacuum-pressure treatments with silanes. The increase in water uptake of dip-treated specimens was preceded by the formation of cracks breaking the water repellent siloxane layer at the surface and expos-

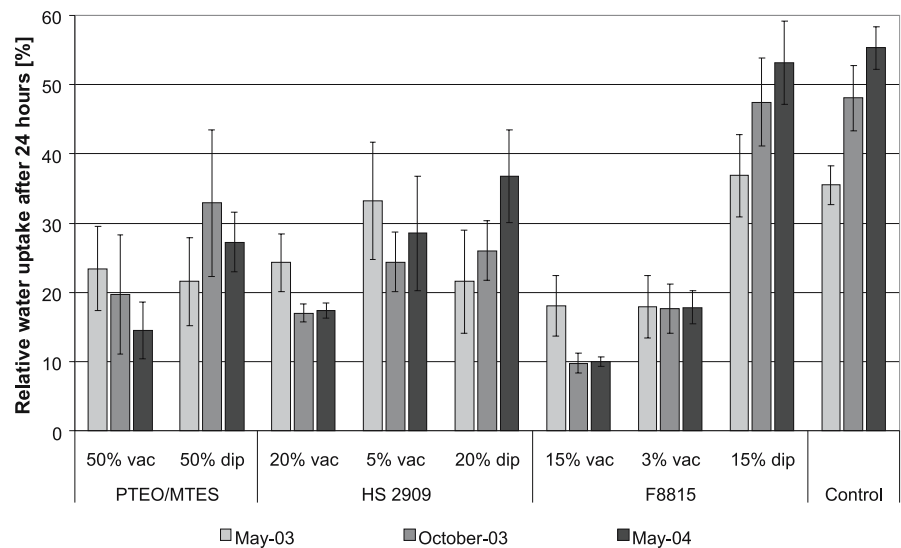
**Fig. 2** Weight variation during outdoor exposure. (1): treated specimens were protected against water uptake after one-time rainfall. (2): longer moist phase led to enhanced moisture content of untreated and treated specimens  
**Abb. 2** Gewichtsschwankungen während der Außenbewitterung. (1): Behandelte Proben sind gegen einmaligen Niederschlag geschützt. (2): längere Feuchtephasen führen zu einer höheren Holzfeuchte der unbehandelten und behandelten Proben



**Fig. 3** Relative water uptake before (May-03), after 5 months (October-03) and after one year of weathering (May-04).

Vac: vacuum-pressure treatment, dip: dip-treatment

**Abb. 3** Relative Wasseraufnahme vor (May-03), nach 5-monatiger (October-03) und nach einjähriger Bewitterung (May-04). Vac: Vakuum-Druck-Behandlung, dip: Tauch-Behandlung



ing uncovered inner parts of wood. Surface evaluation after one month of exposure revealed that all treatments, without exception, failed to prevent the specimens from cracking. A main reason for cracking is a high moisture gradient between the surface and the inner part of wood; this is caused by fast surface drying and comparably slow moisture exchange within the wood. As a consequence, faster shrinkage at the surface induces tensions, which lead to the formation of cracks. Thus, effective protection against cracking not only requires a reduction of wood moisture but also an attenuation of this moisture gradient. The reduction of wood moisture through a particular treatment is expressed as the water repellent efficiency (WRE) which relates the water uptake of treated specimens to that of the untreated controls. Kabir et al. (1992) reported that if the exclusion of water due to the treatment (WRE) correlates well with the rate of moisture fluctuation and the rate of swelling, the WRE can be seen as an indicator for the potential to prevent cracking.

However, the anti-shrink-(swell) efficiency (ASE) obtained at moisture equilibrium has only a minor effect. Oils and wax-based water repellents are known to considerably decelerate the velocity of moisture sorption, shrinkage and swelling, even though they actually do not cause a change of the EMC (Rowell and Banks 1985, Zahora 1991, Treu et al. 2003). Inclusion of water repellent additives (wax emulsions) into CCA treatment solutions led to stabilization of wood conditions during outdoor weathering and reduced checking of pine boards (Evans et al. 2003, Zahora 1992, Zahora 2000). Silane treatment systems applied in this study did not achieve a reduction in cracking, because they had solely influenced the water repellent characteristic but left the sorption behaviour of wood (almost) unchanged.

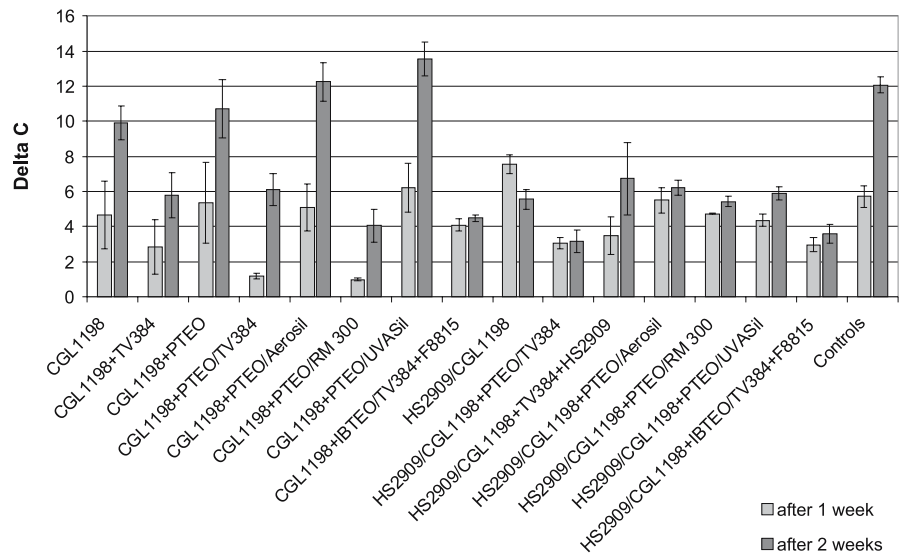
### 3.3 Artificial weathering

In this approach, colour measurements were carried out before weathering, after one week (including 120 h UV irradiation) and after two weeks (including 240 h UV irradiation). Evaluation of the chroma values revealed that the used radical scavenger (CGL 1198, hindered amine light stabilizer) alone did not significantly improve the light stability of the wood surface (Fig. 4). Therefore, the producer recommended applying the radical scavenger in combination with a topcoat containing UV absorbers. Enhanced colour stability was observed when the surface had been additionally treated with the UV absorbers TV 384 (hydroxyphenylbenzotriazole class) or RM 300 WP (doped TiO<sub>2</sub> based). In addition, one-step vacuum pressure impregnation with a solution of HS 2909 and CGL 1198 led to comparable results. No additional effects were observed after secondary treatment with Aerosil (pyrogenic silica), UVASil (hindered amine) and pure sols of PTEO/MTES.

Evaluation of lightness values (Fig. 5) revealed that all treated specimens displayed a darker surface colour than the untreated controls at the onset of the weathering test. After the first week, untreated controls had darkened; this showed the predominance of lignin oxidation and chromophore formation in this state of exposure. After continued weathering, the colour lightened because of the wash-out of chromophoric lignin degradation products. Comparable behaviour was observed with those specimens which were initially vacuum-pressure impregnated without silanes. Lightening was considerably reduced when the treatment solution was based on HS 2909; this reveals effective prevention against the wash-out of wood surface constituents. The use of ethanolic solutions in the second treatment step apparently led to a reduction of this protective effect un-

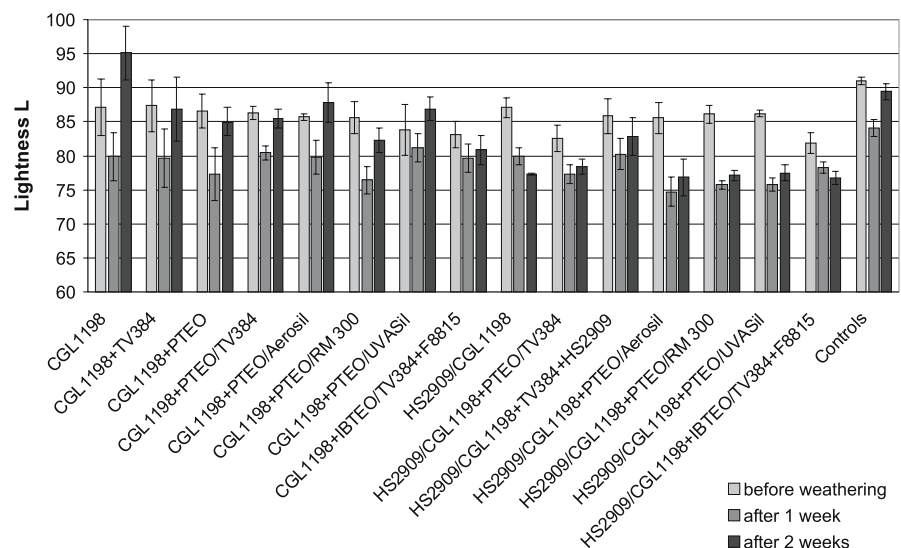
**Fig. 4** Change in chroma according to the CIE-Lab system after artificial weathering in a QUV device. Treatments were performed either in one, two or three steps. First step: vacuum-pressure impregnation; second and third step: dip-treatment

**Abb. 4** Veränderung der Chroma nach dem CIE-Lab-System nach künstlicher Bewitterung in einem QUV-Gerät. Die Behandlungen wurden in einem, zwei oder drei Schritten durchgeführt. Erster Schritt: Vakuum-Druck-Imprägnierung; zweiter und dritter Schritt: Tauchbehandlung



**Fig. 5** Lightness according to CIE-Lab before and after artificial weathering of pine sapwood surfaces in a QUV device

**Abb. 5** Helligkeit nach dem CIE-Lab-System vor und nach künstlicher Bewitterung der Oberfläche von Kiefernspilnholz in einem QUV-Gerät



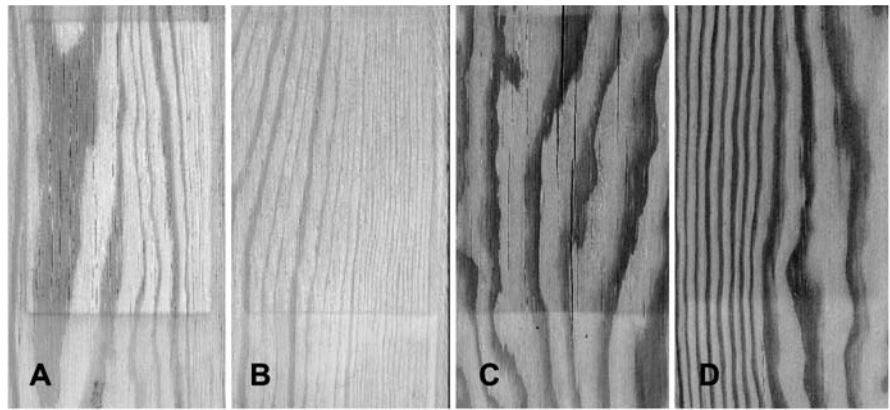
less in a third step the aqueous silane system F 8815 was applied. This silane system forms a thin layer on the surface with an exceptionally high hydrophobicity (Donath et al. 2005). A selection of photographs from the artificial weathering test (after 2 weeks) is given in Fig. 6. About one fourth of the surface area of the specimens were exposed; the lower part shows the initial colour prior to weathering. The untreated control specimens (Fig. 6A) displayed a strong bleaching of the exposed area after weathering. Bleaching was significantly reduced when the specimens were treated with a UV absorber (Tinuvin 384) and a radical scavenger (CGL 1198). Lightening could, however, be observed due to lignin removal (Fig. 6B). All wood specimens which were treated with a solution that contained the oligomeric silane system HS 2909 were darker after treatment. Those specimens which additionally contained a rad-

ical scavenger (CGL 1198), an alkyl silane (PTEO) and RM 300 (TiO<sub>2</sub>) darkened after weathering (Fig. 6C) as a result of the formation of lignin chromophors. Very low changes were observed after weathering of specimens which were treated with a combination of an oligomeric silane system (HS 2909), a radical scavenger (CGL 1198), an alkyl silane (IBTEO), a UV absorber (Tinuvin 384) and a fluoro-alkyl silane (F 8815). Only a minor degree of darkening was observed after treatment (Fig. 6D); this revealed a very low degree of lignin removal and chromophor formation.

Surface degradation during weathering is caused to a major extent by the UV degradation of lignin followed by wash-out of the water soluble reaction products (Feist and Hon 1984). UV irradiation with wavelengths shorter than 350 nm generates free radicals at the exposed surface which trigger lignin degrading oxidative reactions. Light

**Fig. 6** Pine sapwood specimens after 2 weeks artificial weathering in QUV device. Treatments from left to right: (A) untreated control, (B) CGL 1198 + Tinuvin 384, (C) HS 2909/CGL 1198 + PTEO/RM 300, (D) HS 2909/CGL 1198 + IBTEO/Tinuvin 384 + F 8815

**Abb. 6** Kiefernspiltholzproben nach 2-wöchiger künstlicher Bewitterung in einem QUV-Gerät. Behandlungen von rechts nach links: (A) unbehandelte Kontrolle, (B) CGL 1198 + Tinuvin 384, (C) HS 2909/CGL 1198 + PTEO/RM 300, (D) HS 2909/CGL 1198 + IBTEO/Tinuvin 384 + F 8815



with wavelengths longer than 400 nm causes photobleaching and whitening, although lignin is not degraded by light with wavelengths longer than 350 nm. Polysaccharides are degraded to a much lower extent, since they absorb significantly less light in the UV region. At light with a wavelength of 340 nm (maximum of UV-A light applied in this study), cellulose degradation only occurs in the presence of oxygen but at a slow rate. In addition, cellulose as part of the composite material wood is protected against UV-light by lignin which absorbs the main proportion of UV light. Especially phenolic groups in lignin are able to quench radicals formed at the cellulose molecules (Hon 2001).

The degradation processes of lignin are accompanied by various changes in colour, depending on wood species, time of exposure and light spectrum of the irradiation source (Hon and Minemura 2001). FT-IR studies showed a correlation of yellowing, decay of lignin and formation of carbonyl and hydroperoxide groups correlated on UV-irradiated wood surfaces (Hon and Feist 1992).

Leaching of chromophoric compounds (which increases the proportion of white cellulose) lightens the wood surface but has few effects on the chroma (Kamdern and Grelier 2002). Prevention of wood from photo-induced degradation during weathering has been successfully performed by two systems and their combination: UV-absorbers contained in surface topcoats, which hinder UV light from reaching the wood surface and radical scavengers (located at the interface between wood substrate and coating), which trap photo-induced free radicals and thus prevent propagation of radical chain reactions (Hon 2001, Kamdem and Grelier 2002, Müller et al. 2002). According to the law of Lambert and Beer, the transition of lignin-degrading UV irradiation through top coats is inversely proportional to the concentration of the UV absorber and the coating thickness. Therefore, a treatment of the wood substrate beneath the formed siloxane layer with a solution containing radical scavengers is mandatory, since silanes do not form surface layers of sufficient thickness. Still, UV absorbers can cause additional

protection, especially if they have a high concentration at the surface (Müller et al. 2002).

This study shows that the combination of a radical scavenger and a UV-absorber with different types of silane systems was effective in protecting the wood surface, although only a very thin layer of siloxanes was formed. The concentration of UV absorbers on the wood surface was high enough to protect lignin from photo-degradation. The wash-out of lignin breakdown products was additionally reduced due to the strong water repellence of the silane systems; this resulted in a further deceleration of the photo-degradation of lignin.

#### 4 Conclusions

Silane treatment of wood is known to have two basic effects on wood properties: high water repellence but marginal change of sorption behaviour. This profile was reflected in the results from weathering tests.

Reduction in surface checking was not achieved. Obviously, crack development can not be prevented by solely exclusion of liquid water. A change of moisture related wood properties such as moisture flow rate, equilibrium moisture content or dimensional stability is required.

Combinations of UV light stabilizers with silanes led to a higher efficiency of these protectants. The observed ability of silanes to enhance the weathering behaviour of wood is a result of water repellence, which prevents leaching of chemicals and wood constituents.

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