

Weathering, Water Absorption, and Durability of Silicon, Acetylated, and Heat-Treated Wood

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ABSTRACT: In this study, two colloidal dispersions of pure amorphous silicon dioxide particles, acetylated, and heat-treated samples were tested for a possible application as a wood protection agents. The silicon, acetylated, and heat-treated samples were exposed to an accelerated weathering experiment, and their dimensional stability was assessed. The weathering experiment comprised cycles of 2 h UV-light irradiation followed by water spray for 18 min. The surface changes of the weathered samples were characterized by FT-IR spectroscopy and color measurements. According to results, the silicon treatments showed lower color changes than untreated ones. However, acetylated and heat-treated samples provided the lowest color changes. The resistance of the silicon, acetylated, and heat-treated wood to decay was studied by means of brown and white rot fungi in laboratory decay tests. Decay test results revealed that acetylated and

heat-treated wood samples showed better decay resistance against *P. placenta* and *C. versicolor* than silicon treatments. Samples modified with silicon were exposed in above ground standard lap-joint test in Ultuna, Sweden. The dispersions of pure amorphous silicon dioxide impregnated in wood did not significantly influence its hygroscopic and dimensional behavior. However, the silicon treatment reduced the color changes caused by weathering. The silicon impregnated samples showed a weak fungal discoloration similar to that of chromated copper arsenate impregnated controls in above ground standard lap-joint test. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 102: 4506–4513, 2006

Key words: decay test; dimensional stability; durability; field test; modified wood; silicon; degradation; photochemistry; FT-IR

INTRODUCTION

Wood is a renewable biological material that has made it one of the most commonly used construction materials. However, depending on the application, there are certain properties that restrict its use. In particular, these properties are related to its dimensional instability, a function of moisture content, biodegradability, and flammability.^{1–4} These undesirable properties can be improved by chemical modification of the main wood constituents. Various types of chemicals such as anhydrides, isocyanates, silicon, aldehydes, epoxides, alkyl chlorides, etc. have been used for wood modification.^{5,6} The most studied modification methods for wood are acetylation and heat treatment.^{2,7} Acetylation can be defined as a reaction of hydroxyl groups in wood with acetic anhydride as modifying agent.^{2,8} Heat treatment of

wood at temperatures above 160°C improves its dimensional stability and decay resistance in a way that no additional treatment with preservatives is needed for some applications but the decrease of strength is a significant drawback.^{7,9}

Silicon is naturally found in the form of mineral silicates (salt of silicic acid), which mainly consist of polymeric SiO₂ [*m*SiO₂·*n*H₂O or [Si(OH)₄]_{*n*}].⁶ Silicates and most other silicon compounds are classified as nontoxic. A wide variety of organic and inorganic silicon compounds can be applied in the modification process of wood. Inorganic silicon compounds are mainly based on condensation products of silicic acid (colloidal silicic acids, silicates, “water glass”) or tetraalkoxysilanes, which undergo hydrolysis and condensation steps to form sols and finally gels (sol-gel technology). When the sol-gel process is completed, an inorganic silicate, free of organic groups, is formed.^{6,10–15}

The public concern about the health risks of using chromium and arsenic-treated wood has resulted in ceasing of arsenic as an active ingredient in wood preservatives. The research and industry try to establish new trends in wood protection focused on

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environmentally friendly modification methods. The objective of the present study was to reveal the weathering features of three types of modified wood. The silicon, acetylated, and heat-treated samples were exposed to accelerated weathering cycles, their dimensional stability and surface chemistry changes were studied by FT-IR spectroscopy. Data about durability of modified wood were obtained by laboratory and field decay tests.

MATERIAL AND METHODS

Treatments of wood specimens

Scots pine (*Pinus sylvestris* L.) sapwood specimens were impregnated with two colloidal dispersions of silica having a particle size of 15 and 30 nm. A full-cell impregnation procedure consisting of 30 min vacuum (90%) and 90 min pressure (11 bars) was applied. The specimens were then removed from the autoclave and weighed to determine the retentions of each solution. Commercially available acetylated and heat-treated wood was tested in the present study as well. The acetylated samples had 26% weight gain caused by the retention of acetic anhydride. The heat-treated wood was processed according to the Finnish technology (so-called Thermo Wood). The maximum temperature achieved under the treatment was 240°C.

Accelerated weathering test

The weathering experiment was performed by cycles of UV-light irradiation for 2 h followed by a water spray for 18 min in an accelerated weathering test cycle chamber.¹⁶ The average irradiance was carried out at 340 nm wave length and the temperature in the chamber was 45°C. Four replicates of each treatment were exposed for 0, 400, and 800 h. Each sample, 5 × 76 × 152 mm³ was analyzed for color changes according to ISO 7724, and for chemical degradation of the surface by FT-IR spectroscopy.

Color measurements

Color evaluation was carried out according to ISO 7724 standard. The L^*a^*b color space (according to

the Commission International de l'Éclairage-CIE) was used for color evaluation, in which L^* represents the lightness in the range from black (0) to white (100), and a^* and b^* are the chromaticity coordinates; $+a^*$ for red (+60), $-a^*$ for green (-60), $+b^*$ for yellow (+60), and $-b^*$ (-60) for blue. L^* , a^* , and b^* color coordinates for each sample were determined before and after exposure to accelerated weathering. These values were used to calculate the color change ΔE^* as a function of the UV irradiation period according to the standard.¹⁷

Fourier transform infrared spectroscopy

The FT-IR spectra were obtained by a PerkinElmer Spectrum One FT-IR instrument with a Universal ATR Diamond/ZnSe crystal with one reflection. Four accumulated spectra for each sample with a resolution of 16 cm⁻¹ were obtained. The four spectra for each treatment and weathering time were transformed into absorbance spectra averaged before baseline correction (1800–1850 cm⁻¹) and normalization to 1.5 absorbance units for the highest peak at 1030 cm⁻¹.

Water absorption and antiswell efficacy test

Sets of 10 treated wood specimens (15 × 25 × 50 mm³) and 20 untreated control samples were placed into a beaker, filled with deionized water. Water was replaced with fresh one daily during 14 days. The samples were weighed and their dimensions, water absorption values (WA), and antiswell efficacy (ASE) were calculated according to the eqs. (1) and (2) after each water replacement:

$$WA = [(W_2 - W_1)/W_1] \times 100 \quad (1)$$

$$ASE = [(S_u - S)/S_u] \times 100 \quad (2)$$

Where W_2 = wet weight of the wood specimen after wetting with water, W_1 = initial dry weight, S_u = volumetric swelling of untreated wood, S = volumetric swelling of treated wood.

TABLE I
Color Changes of Weathered Samples

Samples	400 h				800 h			
	ΔL	Δa	Δb	ΔE	ΔL	Δa	Δb	ΔE
Control	-18.29 (1.88)	3.59 (0.28)	-6.03 (1.07)	19.62 (1.44)	-14.33 (5.14)	0.55 (1.65)	-12.80 (2.04)	19.64 (2.69)
Silicon 15	-15.02 (5.19)	2.61 (1.25)	-5.65 (2.98)	16.83 (3.51)	-8.19 (3.82)	-0.50 (1.13)	-12.61 (1.65)	15.48 (0.99)
Silicon 30	-14.64 (4.37)	3.22 (1.47)	-3.30 (2.31)	15.66 (3.74)	-11.55 (4.43)	0.25 (1.14)	-9.46 (1.95)	15.33 (2.93)
Acetylated	1.45 (2.72)	0.51 (0.71)	-3.63 (3.48)	4.82 (3.12)	-2.31 (7.12)	0.29 (0.81)	-7.30 (0.81)	9.53 (1.99)
Heat treated	0.73 (9.93)	-2.11 (1.17)	-6.14 (0.65)	9.63 (0.59)	5.51 (9.73)	-2.93 (2.28)	-9.24 (2.05)	12.50 (6.34)

Values in parentheses indicate standard deviations.

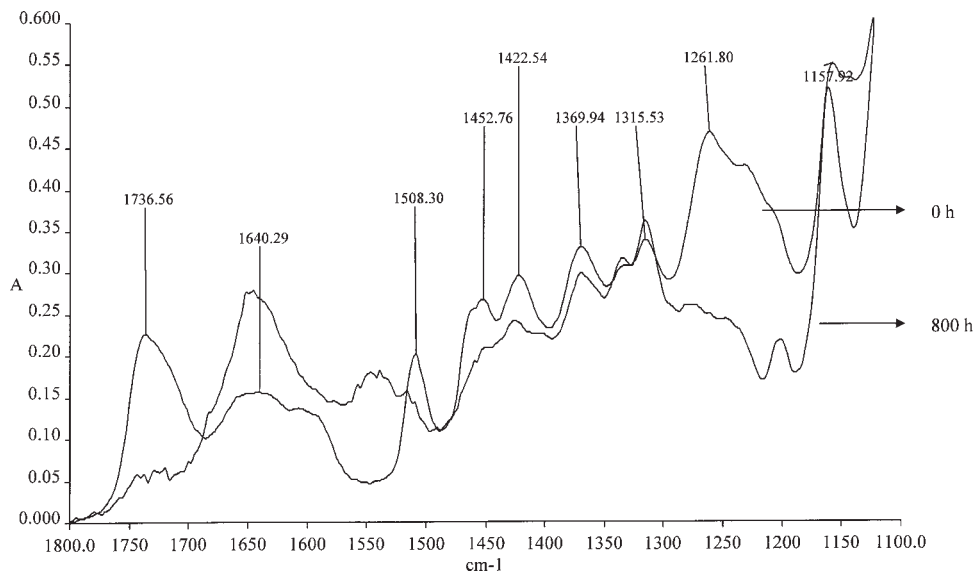


Figure 1 FT-IR spectra of control (untreated) sample.

Fungal resistance tests

Standard EN 113 test

Decay resistance of the silicon-treated samples ($15 \times 25 \times 50 \text{ mm}^3$) was tested according to the European standard EN 113.¹⁸ The brown rot fungus, *Coniophora puteana* was used in the test. Water absorption and ASE samples were used for decay so as to determine the leaching effects that would occur in service. All wood samples were packed in air-tight plastic bags and sterilized by γ -radiation using a ^{60}Co source. Each kofle flask contained one treated and one untreated control sample. After 16 weeks of incubation (25°C and 65% relative humidity), samples were removed and dried at temperature of $(103 \pm 2)^\circ\text{C}$, weighed and the mass loss was calculated.

Modified soil block test

Modified soil block test was applied to the silicon, acetylated, and heat-treated samples. Glass jars (500 mL) were filled to three-quarters of their volume with commercial planting soil and sterilized in an autoclave for 60 min at temperature of 120°C and pressure of about 0.1 MPa. Two subsamples and two samples (one untreated and one treated) ($15 \times 25 \times 50 \text{ mm}^3$) were placed in each jar. The jars with samples were desterilized under same conditions. After cooling, the samples and soil were inoculated with a 12 mL mycelia suspension of the brown rot fungus *Poria placenta* (FPRL 280). The jars were stored in a climate room at temperature of 25°C and 65% relative humidity. The samples were cleaned, dried at

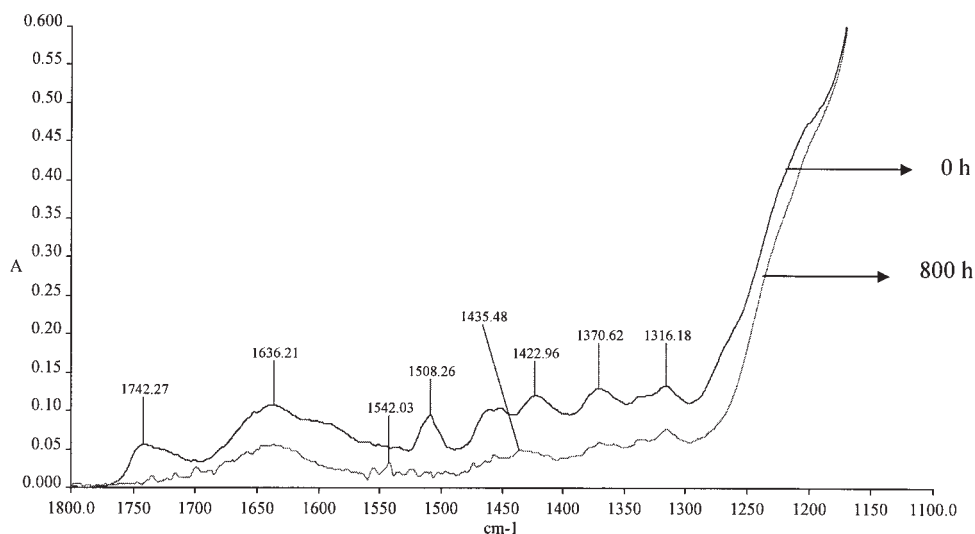


Figure 2 FT-IR spectra of silicon 15 treated sample.

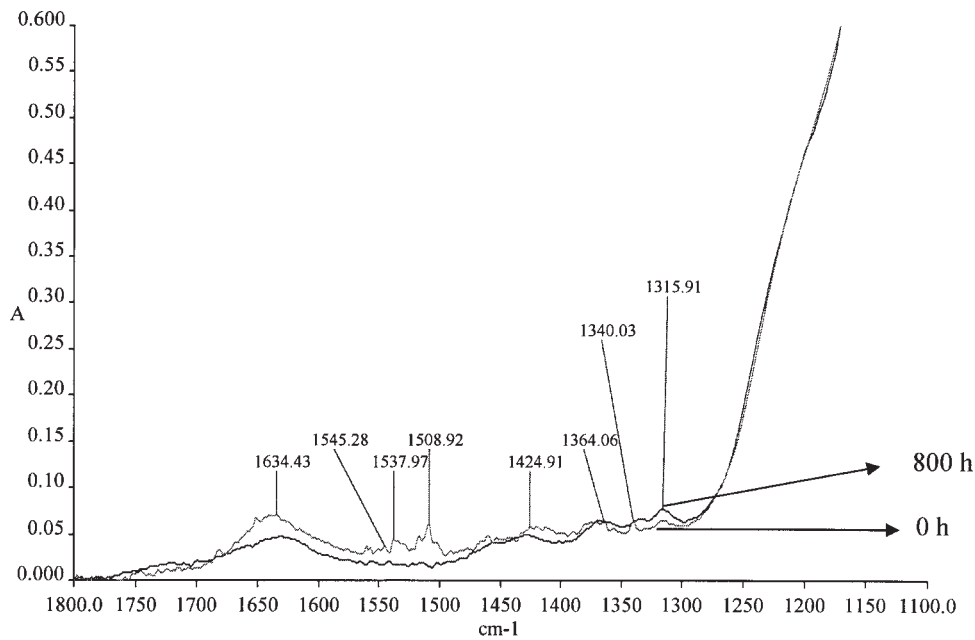


Figure 3 FT-IR spectra of silicon 30 treated sample.

temperature of $(103 \pm 2)^\circ\text{C}$, weighed, and the mass loss was calculated after 84 days of exposure.

($5 \times 15 \times 30 \text{ mm}^3$), then specimens were dried at $(103 \pm 2)^\circ\text{C}$, and the mass loss caused by the fungus was calculated.

Decay test in petri dishes

Only acetylated and heat-treated samples were tested in petri dishes. Agar plates, containing 25 mL 4% (w/v) malt agar were inoculated with the white rot fungus *Coriolus versicolor* (CTB 863A). A plastic mesh was used to avoid the direct contact between the sample and medium. The dishes were incubated at 25°C and 65% RH for 7 weeks. After incubation, fungal mycelium was removed from the samples

Field test

Field test was carried out only with silicon-treated samples. The Scots pine samples treated with silicon were exposed in above ground field testing according to standard ENV 12,037 (lap-joints) in Ultuna, Sweden.¹⁹ The lap-joints had dimensions of $38 \times 85 \times 300 \text{ mm}^3$. Additional reference samples were impregnated with chromated copper arsenate (CCA)

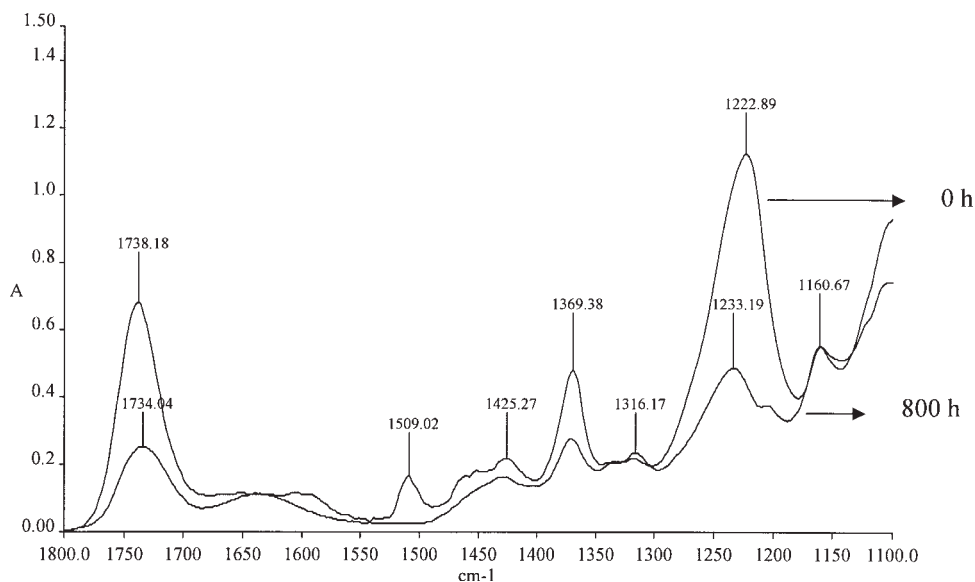


Figure 4 FT-IR spectra of acetylated sample.

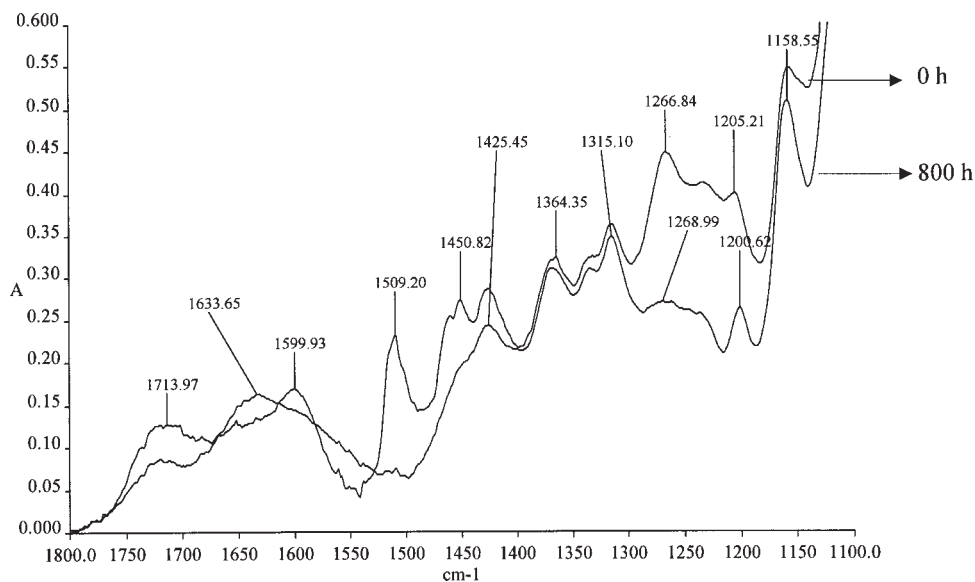


Figure 5 FT-IR spectra of heat-treated sample.

and tributyltin oxide (TBTO) for the lap-joints. The inspection of samples was done annually according to a rating scale from 0 to 4, and median values for stain and decay were estimated.

RESULTS AND DISCUSSION

Accelerated weathering test

Color changes of pine wood samples treated with silicon, heat-treated, and acetylated are shown in Table I.

The positive values of Δb^* show an increase of yellow color, while the negative values indicate an increase of blue color. The positive values of Δa^* indicate a tendency of wood surface to reddish, while the negative values indicate a tendency to greenish. During the weathering, the wood surfaces become darker, thus negative lightness stability (ΔL^*) values occur. The surface of heat-treated wood, being brownish in the beginning, becomes lighter. The lowest values of ΔL^* were obtained on untreated wood samples. ΔL^* was the most sensitive parameter for the wood surface quality on the UV irradiation and water spray.

The lowest color changes (ΔE^*) were determined on the acetylated and heat-treated samples, respectively. However, colloidal dispersions of silica particles treatments provided a better protection against color changes compared with the untreated samples. Color changes of wood surface reflect the chemical changes in wood components during photo degradation. The color change is attributed to the carbonyl group of conjugated ketones, aldehydes, and quinines resulting from the modification of lignin and other related compounds.^{20,21} Lignin is good absorber of light. The phenolic hydroxyl groups in lignin react with light rapidly to generate phenolic radical. These radicals transform into *o*- and *p*-quinonid structures by demethylation or cleavage of the side chain.^{3,20} Acetylation and heat treatment possibly provide better color resistance to wood surface by blocking the reactive sites of photo-induced degradation. The results indicate that color changes (ΔE^*) of silicon and control samples are approximately equal after 400 and 800 h. It can be concluded that all changes of these groups take place during the first 400 h. The color differences between untreated and treated samples are high and visible to the naked eye having an

TABLE II
Assignments of Absorption IR Spectra Bands in Wood

Fr (cm ⁻¹)	Group and class	Assignments and remarks
1720–1740	C=O in unconjugated ketones aldehydes and carboxyl	C=O stretching
1645–1660	C=O in para-OH substituted aryl ketones, quinines	Same
1600	C=C in aromatic ring in lignin	Aromatic skeletal vibrations
1510	Same	Same
1261	CO in lignin and hemicellulose	C–O stretching vibration
1162	C–O–C in cellulose	Antisym. Bridge oxygen stretching

TABLE III
Water Absorption of Treated and Untreated Wood Expressed as a Percentage of Absolute Dry Weight of Sample

Samples	Immersion time (h)								
	24	48	72	96	168	192	240	264	336
Control	62.20 (5.96)	63.84 (5.76)	64.54 (5.91)	67.99 (6.25)	80.01 (7.66)	83.02 (7.55)	85.84 (7.07)	87 (7.11)	90.55 (7.16)
Silicon 15	61.37 (4.34)	64.21 (4.19)	67 (4.57)	72.55 (4.90)	75.61 (4.77)	76.16 (4.91)	77.64 (4.99)	78.32 (5.05)	80.17 (5.17)
Silicon 30	46.08 (2.58)	48.06 (1.78)	49.36 (1.92)	53.89 (2.37)	57.52 (1.94)	58 (2.04)	58.87 (2)	59.36 (2.09)	60.43 (2.12)
Acetylated	47.56 (3.26)	51.27 (3.49)	55.81 (4.04)	66.61 (5.51)	74.57 (4.61)	75.88 (4.83)	78.84 (5)	79.77 (5.02)	83.74 (5.12)
Heat treated	24.70 (1.78)	32.66 (2.05)	38.67 (1.95)	40.10 (1.94)	56.0 (1.77)	60.38 (1.78)	67.56 (1.78)	68.19 (1.83)	79.39 (1.87)

Values in parentheses indicate standard deviations.

ability to perceive differences of at least 2–3 units of ΔE^* .³

The FTIR spectra of treated and untreated samples before and after weathering are shown in Figures 1–5.

When investigating the effect of photo-induced degradation on treated and untreated wood, the attention was focused on the changes of the intensity of the absorptions at 1720–1740, 1592, 1508, 1261 cm^{-1} .^{20,22–25}

The intensity and changes of these bands are related to changes in chemical composition of the functional groups and chemical structure of wood components. The assignments of these characteristic IR absorption peaks in wood are listed in Table II.

The spectral changes of exposed and unexposed control group showed significant changes in the IR spectra of wood surface. The major differences of absorption band for control group occurred in band at 1736, 1640, 1508, 1452, 1422, and 1261 cm^{-1} during to weathering. The absorption at 1510 cm^{-1} is a characteristic peak for lignin because of the C=C stretching vibrations of the aromatic ring present in lignin. This peak usually appears at 1515–1500 cm^{-1} depending on the ring constituents. According to results, significant changes at 1508 cm^{-1} can be seen on the control and heat-treated groups, which indicates the degradation of lignin. However, intensities of absorption band at 1508 cm^{-1} were decreased by silicon and acetylation treatments.

The 1734 cm^{-1} band is characteristic for nonconjugated carbonyl group, which arises due to C=O stretch (in xylan in hemicelluloses). No changes in the absorption band of carbonyl groups (1710–1745 cm^{-1}) were found on the silicon 30 treated samples after weathering, suggesting no photo-oxidation of

wood surface (Fig. 3). The intensities of cellulose band at 1157 cm^{-1} were not significantly affected by weathering for all treatments (Figs. 1–5).

The absorption at 1630–1660 cm^{-1} is related with C=O (conjugated aryl ketones), unconjugated C=C and water. This band changed significantly for the control samples. All treatments reduced the changes in the region of 1630–1660 cm^{-1} .

The absorption at 1261 cm^{-1} is characteristic absorption of C–O stretching vibration in lignin and hemicellulose. During the weathering test, this band for control group, acetylation, and heat treatments decreased. However, no changes at that band were determined on silicon treatment samples.

Some studies showed that an increase of carbonyl absorption band at 1720–1745 cm^{-1} caused by UV light degradation. However, a decrease of the carbonyl absorption band at 1710–1745 cm^{-1} was found in the present study. This can be attributed to the water sprayed over the samples and contributing to leaching of carbonyl components formed during the UV degradation. These findings have been proven by studies, using both UV and water spray system.^{26–33}

Water absorption values and ASE results are shown in Tables III and IV, respectively. Water absorption of the control samples increased from 62% (after the first day) to 91% after 14 days of exposure. Silicon-treated samples had lower water absorption, respectively, 46 and 80% lower than that of the control. The results showed that all silicon modified wood samples displayed high water absorption values. However, the water absorption of silicon with 30 nm particles was less than that of silicon with 15 nm granules. Water absorption of silicon 15 treated wood was, in most

TABLE IV
Antiswell Efficacy (ASE) of Treated Wood

Samples	Immersion time (h)								
	24	48	72	96	168	192	240	264	336
Silicon 15	7.52 (4.98)	3.32 (2.23)	4.51 (3.38)	5 (2.56)	4.08 (2.72)	4.52 (3.25)	5.49 (3.69)	4.27 (2.92)	4.42 (2.60)
Silicon 30	5.31 (3.46)	5.01 (2.58)	4.05 (2.91)	3.17 (3.54)	3.06 (2.77)	3.72 (3.71)	3.41 (3.27)	3.65 (3.40)	3.19 (3.39)
Acetylated	84.48 (3.75)	85.63 (5.46)	86.18 (5.78)	86.06 (6.04)	85.75 (5.20)	86.80 (5.32)	86.50 (5.56)	87.14 (5.38)	86.95 (5.63)
Heat treated	76.63 (1.87)	71.70 (2.54)	70.62 (1.68)	70.30 (1.51)	67.13 (1.64)	67.15 (2.05)	67.06 (1.69)	66.79 (1.63)	66.50 (1.88)

Values in parentheses indicate standard deviations.

TABLE V
Mass Loss of Treated and Untreated Specimens Caused by *Poria placenta* in Modified Soil Block Test

	Treated		Untreated controls	
	Average (%)	S.D.	Average (%)	S.D.
Silicon 15	6.27	4.54	47.55	3.61
Silicon 30	20.73	2.12	42.06	8.17
Acetylated	0.65	0.39	35.76	4.82
Heat treated	2.07	1.51	34.32	6.56

cases, similar to untreated control. High water absorption can be attributed to hygroscopicity of silicon compounds.⁶ Heat-treated samples showed lower water uptake than the other treatments. Low values of water uptake for the heat-treated wood are attributed to the thermal degradation of hemicellulose, which is the most hygroscopic polymer in the wood cell wall.^{7,9}

Both silicon treatments had very low ASE values after 168 h of immersion in water. The highest ASE values were calculated for the acetylated and heat-treated samples, respectively, a fact explained by the blocking or eliminating of hydroxyl groups in wood by the treatments. According to the results obtained, silicon 30 did not cause better ASE than silicon 15. Differences in silicon treatment did not influence the ASE results noticeably (Table IV). Large changes of ASE (3–69%) of silicon-treated wood have been reported in the literature.⁶

Decay resistance against *P. placenta*, *C. versicolor*, and *C. puteana* fungi is shown in Tables V, VI, and VII. According to the results, untreated wood specimens were decayed significantly by the fungi. Acetylation and heat treatments provided better decay resistance against *P. placenta* and *C. versicolor* than silicon treatments. The lowest weight loss was determined for the acetylated samples (Table V). The effectiveness of acetylation process is attributed to blocking of the hydroxyl groups of the main structural compounds, thus making wood unrecognizable for decay fungi.⁸

High mass losses of silicon-treated samples were generally found for leached ones. Decay results showed that considerable amount of silicon compounds were washed out from wood by leaching. These findings are also reported by other studies.⁶ Unleached wood specimens treated with silicon 15

TABLE VI
Mass Loss of Treated and Untreated Specimens Caused by *Coriolus versicolor* (Decay Test in Petri Dishes)

	Treated		Untreated controls	
	Average (%)	S.D.	Average (%)	S.D.
Acetylated	0.11	0.04	20.33	3.29
Heat treated	0.38	0.25	18.17	4.24

TABLE VII
Mass Loss of Silicon-Treated and Untreated Specimens Caused by *Coniophora puteana* in Laboratory Test According to EN 113

	Treated	Control
Silicon 15		
Leached	47.66 (2.96)	35.53 (1.34)
Unleached	0.38 (0.41)	46.19 (3.36)
Silicon 30		
Leached	37.24 (2.96)	28.41 (11.05)
Unleached	15.73 (7.91)	26.25 (2.67)

Values in parentheses indicate standard deviations.

showed low mass loss for all fungi tested. However, silicon 30 samples were not well-protected from the attack by *P. placenta* and *C. puteana* (Tables V and VII). Poor defense ability of some silicon compounds against decay fungi has been reported by several authors. To improve the effect of silicon treatments, they often have been combined with fungicides such as boric acid, borax, CCA, quaternary ammonium compounds, etc.^{6,12,34,35}

Silicon treatments were not tested against *C. versicolor*. The silicon impregnated samples were tested in an above ground standard lap-joint test (ENV 12,037) in Ultuna, Sweden. The decay and fungal discoloration rates of the lap-joints is shown in Table VIII. The silicon-treated samples showed a fungal discoloration similar to that of CCA impregnated controls in above ground standard lap-joint test.

CONCLUSIONS

This study evaluated weathering, water absorption, and durability behavior of wood treated with two colloidal dispersions of silica particles (SiO₂), acetylation, and heat treatment. Accelerated weathering test of silicon treatment showed that colloidal dispersions of silica particles provided better protection against color changes than untreated samples. According to FT-IR results, it was found that the silicon 30 treated samples showed no changes in the absorption band of carbonyl groups (1710–1745 cm⁻¹) after weathering.

TABLE VIII
Assessment of Lap-Joints According to ENV 12037 After 4 Years of Exposure

	Upside		Bottom		Joint area	
	Stain	Decay	Stain	Decay	Stain	Decay
Silicon 30	2	0	0	0	0	0
References						
TBTO (1 % concentration)	3	0	3	0	3	0
CCA (0.8 % concentration)	1	0	1	0	0	0
Untreated	3	0	3	0	3	0

Silicon and acetylation treatments decreased the intensities of absorption band at 1508 cm^{-1} , characteristic peak for lignin. Silicon treatment of wood did not significantly improve the water repellence and dimensional stability of wood, because of the hygroscopic characteristic of silicon. Above ground field, test of silicon-treated samples showed significantly better protection against fungal discoloration when comparing with control and TBTO treated reference samples. Both silicon 15 and 30 showed high mass loss in laboratory decay test after leaching. The comparison between the behavior of silicon treated samples in laboratory and above ground field test proves that only laboratory test is not enough for evaluation of modified wood durability.

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