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Treatment of wood with silica sols against attack by wood-decaying fungi and blue stain

Abstract: Pine sapwood was treated with various types of silica sols. Whereas alkaline sols were not able to penetrate deeper into wood, neutral and acidic sols showed good penetration. The weight percent gain of treated specimens amounted to 20–25%; bulking was negligible or even slightly negative. All silica sols in the treated specimens were stable against water leaching. A water submersion test revealed hydrophobation of the wood only after treatment with a cationic silica sol; all other silica sols increased the rate of water uptake. The addition of 2% cationic sol to a malt-agar growth medium caused growth inhibition of 40–50% of the wood decay fungi Coniophora puteana and Trametes versicolor, whereas the other silica sols did not inhibit growth. Pine sapwood and beech wood blocks treated with the cationic sol showed a strong reduction in mass loss compared to the control samples after incubation with C. puteana (pine) and T. versicolor (beech) according to EN 113 and CEN/TS 15083-1; all other silica sols did not inhibit fungal decay. The cationic silica sol reduced blue staining by Aureobasidium pullulans compared to the untreated control but did not fully prevent it; all other silica sols did not inhibit blue staining.

Keywords: blue stain, brown rot, cationic surface, fungal decay, silica, silica sol, water glass, white rot, wood modification

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Introduction

Among the substances used for wood modification are anhydrides, isocyanates, aldehydes, epoxides, formaldehyde-based resins and others (Rowell 1983; Militz et al. 1997), which can improve dimensional stability, resistance to wood-destroying fungi and insects, flammability, strength and hardness. Silicon is an abundant non-toxic element that can form a wide variety of inorganic and organic compounds (Römpp 2001). For treatment of wood, water glass (sodium silicate, Na$_2$SiO$_3$) has a long history. It was first described as a fire protection agent by Fuchs (1825) and intensely studied as a fire retardant coating (Metz 1939). Recently, wood was impregnated with water glass to increase dimensional stability, decay resistance and water uptake. The alkaline water glass precipitates partly on contact with the slightly acidic wood. After impregnation, it can leach out upon contact with water and has to be fixed in some way (Furuno et al. 1991, 1992, 1993; Matthes et al. 2002).

Silicas are the silicon analogs (Si$_{n}$H$_{2n}$) of alkane hydrocarbons. Alkoxysilanes introduced into wood can undergo condensation reactions and form a silicon network inside wood via a sol-gel reaction. Saka et al. (1992) observed cell wall bulking (chemical swelling) of alkoxysilane-impregnated wood, which indicates that silicon compounds can be incorporated into the cell wall. Silanes enhance decay resistance to fungi and reduce water uptake (Donath et al. 2004, 2006a,b). However, alkoxy groups in silanes are easily hydrolyzable. Application of most organosilanes requires solving in organic solvents or their aqueous mixtures. Therefore, a practical application of silanes for treatment of solid wood is not viable.

Silica sols are produced by controlled removal of alkali from water glass (e.g., aqueous sodium silicate solution) through ion exchange techniques. This causes polycondensation of the silicic acid, which forms growing colloid particles of amorphous silicon dioxide. When the condensation process is stopped at a certain stage by the addition of some alkali, a sol of polysilicic acid molecules is obtained (Römpp 2001). Silica sols are therefore alkaline, and the colloid particles precipitate at acidification because they are stabilized by negative charge. Sols can also be stabilized either sterically (modifications with silanes) or by positive charge (cationic surface of the particles). These modified colloids can be stable under neutral or acidic conditions (Greenwood 2010).
Götte et al. (2008) treated wood with commercially available alkaline silica sol and found some reduction in water uptake. Treatment of wood with neutral and acidified silica sols imparted increased dimensional stability and reduced moisture uptake (Yamaguchi 1994b). Vacuum pressure treatment with nonfunctional alkaline silica sol afforded good protection against the brown rot fungus Coniophora puteana (according to EN 113 1996) when the samples had not been leached before incubation (Temiz et al. 2006). Leaching reduced fungal resistance.

Complete protection against *C. puteana* was achieved by dip treatment of wood specimens in 5% solutions of sols formed from tetraethoxysilane (sol-gel process) in ethanol (Böttcher et al. 1999). Silica sols are not classified as biocidal products (Römpf 2001) but can support biocidal properties by physically embedding soluble biocides in the silica matrix (Böttcher et al. 1999; Böttcher 2000; Haufe et al. 2005) or by covalently binding biocides to the sol surface. Covalently bonded biocides are often poly-cationic and are assumed to interact with the negatively charged cell membranes of microorganisms. This can lead to removal of anionic phospholipids from the cell membrane and leakage of proteins and other constituents (Mahltig et al. 2008; Tiller 2011).

In this study, various modified silica sols are investigated for their capability to improve wood properties through impregnation.

## Materials and methods

### Chemicals

Various commercially available silica sols (Akzo Nobel Chemicals GmbH, Düren, Germany) were tested (Table 1). Bindzil CAT 650 is a product with acidic pH, very high surface area and low particle diameter, which served only for testing cell wall penetration as expressed by weight percent gain (WPG) and cell wall bulking. The number in the brand names of Levasil and Bindzil indicates the surface area of the particles in square meters per gram. This number is directly related to the diameter of the particles.

### Wood specimens and their dimensions

In the following, "pine" and "beech" indicate *Pinus sylvestris* L. and *Fagus sylvatica* L., respectively. Penetrability: pine sapwood 40 mm×40 mm×40 mm [Radial (r)×Tangential (T)×Longitudinal (L)]; water uptake, bulking: pine sapwood, 25 mm×25 mm×10 mm (R×T×L); resistance to basidiomycetes: pine sapwood and beech (15 mm×mm 25×50 mm (R×T×L); blue stain: pine sapwood, 40 mm×5 mm×40 mm (R×T×L), growth rings oriented 45° with the tangential face.

### Uptake of silica sols

Wood blocks were dried at 103°C and the dry mass was determined. Then the specimens were conditioned at 20°C and 65% relative humidity (RH) and five faces were sealed with a coating (Pyrotect – Holz Color finish, Rüters Organics, Mannheim, Germany) to leave only the tangential side open. The specimens were impregnated with a 15% (w/w) aqueous solution of the silica sols (except Bindzil CAT 650) by vacuum pressure impregnation (100 hPa, 1 h; 1.3 MPa, 2 h). The samples were weighed.

### WPG and cell wall bulking

These values were calculated according to the following formulas with all measurements taken at oven-dry state:

\[
\text{Bulking (\%)} = \frac{\text{(rad after tr} \times \text{tan after tr})}{\text{(rad before tr} \times \text{tan before tr})} - 1
\]

\[
\text{WPG (\%)} = \frac{\text{weight after tr}}{\text{weight before tr}} - 1
\]

where rad is the radial length, tan is the tangential length and tr is treatment.

### Water uptake

Eight specimens of each treatment (treated together with the penetrability samples) were submerged in approximately 300 ml of water and weighed after 2, 4, 6 and 24 h of submersion time. After the last measurement, a vacuum of approximately 40 hPa was applied for 1 h. The specimens were left in the water for another day to ensure maximum water uptake. Water uptake was calculated based on the dry weight of the specimens before treatment. Water repellent effectiveness (WRE) was calculated based on the mean values of water uptake (WU) of the untreated and treated specimens after 24 h according to:

\[
\text{WRE (\%)} = 100 \left( \frac{\text{WU}_{\text{unt}} - \text{WU}_{\text{tr}}}{\text{WU}_{\text{unt}}} \right)
\]

<table>
<thead>
<tr>
<th>No.</th>
<th>Name</th>
<th>Concentration of stock solution (%)</th>
<th>Surface modification</th>
<th>pH of stock solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Levasil 200E</td>
<td>20</td>
<td>Unmodified</td>
<td>2.9</td>
</tr>
<tr>
<td>2</td>
<td>Levasil 2005</td>
<td>30</td>
<td>Aluminum oxychloride</td>
<td>3.7</td>
</tr>
<tr>
<td>3</td>
<td>Bindzil CC151</td>
<td>17.5</td>
<td>Epoxypropylsilane</td>
<td>7.6</td>
</tr>
<tr>
<td>4</td>
<td>Modified Bindzil CC151</td>
<td>17.5</td>
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<td>6</td>
</tr>
<tr>
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<td>30</td>
<td>Aluminate</td>
<td>10</td>
</tr>
<tr>
<td>6</td>
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</tr>
<tr>
<td>7</td>
<td>Bindzil CAT 650</td>
<td>15</td>
<td>Aluminum oxychloride</td>
<td>3.6</td>
</tr>
</tbody>
</table>

Table 1 Commercially available silica sols used for impregnation of wood.
Growth test on agar

Silica sols were mixed with agar growth medium prior to fungal inoculation to test their effect on the growth of wood-decaying fungi. Malt-agar solution (4% malt, 2.5% agar) was autoclaved at 121°C for 20 min. Silica sols no. 1–4 were separately heated in a beaker on a hot plate to 60–70°C and then added to the malt-agar solution, which had been left to cool down to approximately the same temperature. The tested silica sol concentrations were 0.10%, 0.25%, 0.50% and 2.00%.

The solutions (25 ml) were poured into Petri dishes of 9-cm diameter. After cooling and hardening of the medium, the plates were inoculated in the center with a 9-mm-diameter inoculum, which was cut out from approximately 2-week-old cultures of either the brown rot fungus *C. puteana* (CTB 863A) or the white rot fungus *Trametes versicolor* (BAM Ebw. 15). The Petri dishes were incubated at 22°C and 70% RH for 8 days. The growth of the fungi was evaluated daily by measuring the diameter of the overgrown zone and subtracting the diameter of the inoculum. Five replicates were made for each fungus and for each concentration of a specific chemical.

Fungal decay

Wood specimens were dried at 103°C for 48 h and the dry weight was determined. The specimens were then treated with 15% silica sol solutions no. 1–4 according to EN 113 (1996). The specimens were placed in a desiccator, and a vacuum (7 hPa) was applied for 15 min. Subsequently, the solution was injected and the vacuum was released. The specimens were left in the solution for 2 h and the weight was determined. They were subsequently dried slowly to prevent cracking and finally at 103°C. The dry weight was measured; a leaching according to EN 84 (1997) was done, and the specimens were dried again. The specimens were conditioned at 20°C and 65% RH and inoculated in Kolle flasks on 4% malt and 1.5% agar according to EN 113 (1996). Ten replicates were used per treatment. Treated and untreated specimens of pine were tested with standard CEN/TS 15083-1 (2005). The tested samples were conditioned at 20°C and 70% RH for 8 days. The blue-stained area was analyzed with an Epson Expression 10000XL scanner (Seiko Epson Corp., Nagano-Ken, Japan) and GIMP image processing software (GNU General Public Licences). To determine the percentage of staining, the front and reverse side were scanned and converted to black (stained) and white (unstained) images by adjusting a threshold value in a way that the stained areas were converted to black and the unstained areas to white.

Statistics

Each treatment was compared with the control by a Student’s t-test because the data did not fulfill the requirements for analysis of variance. Significance was assumed if the resulting P-value was smaller than or equal to 0.05.

Results and discussion

Uptake of silica sols

The uptake of the various silica sols was pH dependent: higher in the case of four acidic and neutral silica sols and approximately 50% lower of the two alkaline sols than that of the control (Figure 1). The results with acidic sols

![Figure 1](https://example.com/image.png)

Figure 1 Solution uptake of wood blocks treated with 15% solutions of various silica sols through the tangential side in a vacuum pressure impregnation procedure [mean values±standard deviation (SD)]. All values except for Levasil 200 E are significantly different from the control (P ≤ 0.05).
can be explained with their higher density compared to water. Another reason for the low uptake of alkaline sols could be related to minor water solubility under more acidic conditions. Both alkaline sols are generally stable toward precipitation from the solution, Levasil 200A due to its alumininate-modified surface and Levasil 50 due to its large sol particle size. Still, it is assumed that the acidic pH of wood caused precipitation of the alkaline sols at the wood surface by neutralization, resulting in blocking of the main penetration pathways for capillary solution uptake. Acidic sols nearly do not change their pH in contact with wood and are able to penetrate into the fine pore system without precipitation.

**WPG and cell wall bulking**

All treated anti-shrink efficiency (ASE) specimens showed a high uptake of the silica sols due to their small size and their big cross-sectional area relative to the total volume. The WPGs were therefore similar (Figure 2) irrespective of the sols’ pH. As a result of water leaching, all specimens lost only 1–2% of their weight, whereas the control specimens lost 1% of their weight as water-soluble extractives. Accordingly, the condensed silica in wood is stable toward leaching (Figure 2).

None of the sols, however, induced cell wall bulking; for most specimens, bulking was even negative after the treatment. Probably, the particle diameter of the sols is greater than the diameter of the cell wall pores. Assuming a density of the silica of 2 g cm⁻³ and a surface area of 200 m² g⁻¹, a mean particle diameter of 15 nm is obtained. Several authors determined the diameter of the cell wall pores of wood in the water-swollen state by the solute exclusion technique. Tarkow et al. (1966) determined the penetration of polyethylene glycol into the cell wall of never-dried sitka spruce and found that penetration is possible up to a molecular mass of approximately 3000 g mol⁻¹, corresponding to average pore diameters of 1.8–2 nm. Kerr and Goring (1975) measured the maximum pore size of birch wood to be approximately 10 nm. This value is similar to that of pine wood measured by Grethlein (1985) by observation of dextran penetration up to a molecular diameter of approximately 11 nm. The quoted author, however, pointed out the uncertainty of absolute values for pore diameters, which could be approximately 50% smaller than reported. Flournoy et al. (1991) reported pore sizes of kiln-dried sweet gum wood to be smaller than 1.5 nm. The maximum pore size of never-dried black spruce was found to be 3.6 nm (Stone and Scallan 1968).

In summary, the maximum pore diameter in the cell wall is likely to be smaller than 10 nm.

Nevertheless, Bindzil CAT 650 with a specifically large surface area of 650 m² g⁻¹ and a mean particle diameter of approximately 5 nm was also not able to penetrate the cell wall. Generally, sol particles are polydisperse with a Gaussian size distribution. Therefore, a part of the particles of Bindzil CAT 650 might have diameters below 5 nm. Hence, even silica sols with the smallest particle diameters could not enter the cell wall. This might be due to agglomeration of the particles when entering the pore system of wood. This result corresponds to findings of Matsunaga et al. (2009), who treated wood with nanoparticles of copper carbonate with minimum diameters of 1 nm and did not find cell wall penetration of these particles either.

Negative bulking values are normally attributed to deterioration of hemicelluloses. It was found that negative bulking increased with increasing drying time and temperature (results not shown). This may be due to hydrolysis of cell wall polysaccharides. These results on the lack of dimensional stabilization correspond to findings of Temiz et al. (2006), who found only minor penetration of the silica sols into the cell wall and very low ASE. A silica sol prepared from sodium silicate by cation exchange technique exhibited a pH of approximately 3 and imparted dimensional stability of about 30% ASE to treated wood (Yamaguchi 1994b). Obviously, the sol particles had very low diameters, enabling cell wall penetration. The sol, however, was not stabilized and gelled after a few hours (Yamaguchi 1994a).
Water uptake

Silica is hydrophilic and not expected to impart water-repellent properties to wood. Reduction in capillary water uptake might be brought about by blocking main penetration paths such as ray cells or tracheids. This was, however, only achieved with the cationic sol Levasil 200S, whereas all other silica sols increased the velocity of water uptake in a submersion test compared to the control (Figure 3). Even the alkaline silica sols Levasil 200A and Levasil 50, which immediately precipitated in the upper layers of the wood specimens during impregnation, did not reduce capillary water uptake. The effect of Levasil 200S might be explained with ionic interactions between the cationic surface of the sol and anionic groups of cell wall polymers such as carboxyl groups of hemicelluloses or phenolate groups of lignin. These interactions might allow closer attachment between wood and silica.

Temiz et al. (2006) reported that wood treated with alkaline silica sol bearing a particle size of 30 nm exhibited lower water uptake than wood treated with silica sol of 15-nm particle size. Treatment of spruce wood specimens with silica sols reportedly reduced water uptake by approximately 25% during 6 days of water immersion (Götze et al. 2008). In contrast, water glass increased the water uptake of wood and was highly susceptible to leaching (Furuno et al. 1992; Matthes et al. 2002). Increased water uptake was explained with the hygroscopic counter ions of silicate in the wood and with the hygroscopicity of the precipitated water glass itself.

Growth test on agar

The growth of the brown rot fungus *C. puteana* and the white rot fungus *T. versicolor* was only inhibited by Levasil 200S added in 2% concentration. All other sols did not show significant effects compared to the control (Figure 4a and b). Thus, neither the silica sol itself in the malt-agar growth medium nor its pH (Table 1) has an inhibiting effect on fungal growth.
Levasil 200S can be considered as a modified product of the alkali-stabilized Levasil 200/30; its silanol groups (Si-OH) form bonds with aluminum oxychloride (Figure 5), and therefore it has a cationic surface, whereas that of Levasil 200E is predominantly uncharged. Growth inhibition can be attributed to aluminum chloride (AlCl₃) or to the cationic surface of the sol. AlCl₃ in a state of a soluble salt is known to have fungicidal properties (Kollmann 1951), but this was explained only by its acidic reaction (Metz 1939). This is an unlikely explanation, because Levasil 200S is only slightly acidic. AlCl₃ was shown to inhibit xylanase of the brown rot Gloeophyllum trabeum (Ritschkoff 1996). In Levasil 200S, however, AlCl₃ is covalently bound to the surface of the sol particles and the ions are immobilized. Most likely, the cationic surface of the sol particles causes inhibition of fungal growth.

Tiller (2011) divided bioactive surfaces into biocide-releasing and non-biocide-releasing surfaces. The latter were further classified into (a) those that inhibit the microorganism by contact with a biocide attached to the surface via a spacer and (b) cationic surfaces, which kill the microorganism by removal of vital hydrophobic anions (e.g., phospholipids) from the cell membrane.

The concept (b) may be the reason for the effectiveness of Levasil 200S against fungal growth. The cationic groups are attached directly to the sol surface and are therefore not able to penetrate the fungal cell membrane (as it would be possible if attached via a spacer or if free to move). The cationic sol surface might interact with the negatively charged phospholipids of fungal cell membranes, leading to removal of these anions and, as a consequence, to leakage of constituents of cytoplasm (Mahltig et al. 2008; Tiller 2011).

Addition of emulsions of amino-functional polysiloxanes to agar growth medium was previously shown to inhibit the growth of white and brown rot fungi (Ghosh et al. 2008). Cationic primary ammonium groups were considered to be the main reason for this effectiveness, but an effect of the emulsifiers seems to be likely. In the case of Levasil 200S, the effect is directly attributable to cationic groups on the sol surface; a higher concentration of the sol was required to inhibit fungal growth compared to that of the amino-functional siloxanes.

**Fungal decay**

Resistance of sol-treated wood specimens to basidiomycete decay was tested with *C. puteana* and *T. versicolor*. Mass loss (ML) of the respective untreated pine or beech specimens exceeded 20%, so that the test was valid according to the European standard EN 113 (1996). WPG of the treated specimens after leaching was very similar between the treatments; only Levasil 200S was tested with two WPGs. The cationic sol Levasil 200S (at 15% concentration) reduced ML caused by *C. puteana* to 1.7% and by *T. versicolor* to 5.3%. The lower concentration did not show high effectiveness in ML reduction (Figure 6a and b). The moisture content (MC) of these specimens after fungal incubation was below 80%; prevention of decay due to water soaking and the resulting lack of oxygen could thus be ruled out. Treatment of pine wood with Bindzil CC151, modified Bindzil CC151 and Levasil 200E resulted in higher ML compared to the control specimens, which was not attributable to leaching of silica during fungal incubation (Figure 6a). It is assumed that these sols enhanced the capillary water uptake of the test specimens (see above) and, thus, promoted fungal colonization in the initial part of incubation. In the case of *T. versicolor*, specimens treated with Bindzil CC151, modified Bindzil CC151 and Levasil 200E displayed MLs similar to that of the control (Figure 6b).

Hydrophobation as the main reason for high fungal resistance of Levasil 200S-treated wood can be ruled out because the MC of the specimens after incubation (57%) was sufficient to allow colonization. As described above, cationic groups on the surface of Levasil 200S may interact with anionic groups of cell wall polymers, which can replace chloride as a counter ion of the aluminumoxygroups. Cationic compounds are known for their biocidal effect against fungi and other microorganisms (Kenawy et al. 2007). Especially, quaternary ammonium compounds are used as cationic wood preservatives (Worley and Sun 1996; Pernak et al. 1998; Zabielska-Matejuk et al. 2004). Amino-functional silanes and siloxanes, which are expected to be cationic at the pH of wood, can also inhibit fungal decay (Donath et al. 2006a; Weigenand et al. 2008). In contrast to the cationic siloxanes, which are usually mobile, the sol particles in this case are completely immobile, cannot enter the fungal cell wall and

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**Figure 5** Idealized structure of a cationic Levasil 200S sol particle.
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therefore have a different mode of action (Tiller 2011). As described above, the cationic silica surface itself might be responsible for the effectiveness (Mahltig et al. 2008; Tiller 2011). Anionic or uncharged surfaces are not able to interact with phospholipids in fungal cell membranes; this explains the lack of effectiveness of all other sols tested.

Several authors found reduction of fungal decay by basidiomycetes due to treatment with silica sols (Yamaguchi 2002; Temiz et al. 2006) or water glass (Furuno et al. 1991, 1992; Matthes et al. 2002), but the mode of action of these treatments is attributed either to the high pH of the treatment solution or to the addition of biocidal compounds such as borax and boric acid. The present study shows that silica sols themselves do not have inhibitory effects on wood-decaying fungi, but that cationic surface modifications can improve decay resistance.

Blue stain

None of the silica sols tested inhibited the colonization by *A. pullulans* on the side facing the vermiculite compared to the control specimens. Colonization of the upper surface was only strongly reduced by the cationic silica sol Levasil 200S (Figure 7). The staining fungi apparently started to overgrow the sample from the side facing the vermiculite, most probably due to higher moisture and high concentrations of spores in the vermiculite. It is likely that the reduced fungal colonization on the upper side caused by Levasil 200S is due to lower MC. As shown above, Levasil 200S clearly reduces the capillary water uptake, which results in a dryer upper surface and a lower rate of spore germination. It is also possible that Levasil 200S inhibits the penetration of fungal hyphae through the wood specimens.

Similar tests of water glass-treated specimens revealed high resistance to staining, which was attributed to the high alkalinity (pH 10–12) of the specimens (Pfeffer et al. 2011). Strong reduction of blue stain colonization was also observed with wood treated with amino siloxane (Ghosh et al. 2009). This was attributed to the presence of amino groups and its effect on fungal physiology.

In the present study, treatment with silica sols did not inhibit surface colonization of wood by blue stain fungi directly, irrespective of the surface modification of the sols. Cationic silica sols, however, may exert an indirect
effect on surface staining because they reduce capillary water uptake and, thus, lower surface moisture. The cationic properties of the sol seem to have only little effect on A. pullulans.

Conclusion

Bulk penetration of solid wood can only be reached with neutral or acidic silica sols; alkaline sols are not stable enough to enter the acidic wood structure. After treatment, silica sols showed complete stability toward leaching once dried in the wood. Cell wall penetration cannot be achieved even with the smallest particle sizes available. The treatment therefore cannot be classified as real modification or as impregnation modification (according to Norimoto and Gril 1993).

The velocity of water uptake was only reduced by the cationic silica sol. The same sol type was also the only one that enhanced the resistance against brown and white rot fungi, whereas staining by A. pullulans was only partly inhibited. In contrast to water glass, which most likely enhances fungal resistance due to its high alkalinity, cationic silica sols possibly inhibit microorganisms by affecting the integrity of the cell membrane. Compared to “classical” chemical modification, high fungal resistance is not mainly attributed to changes in the material properties of wood but to a direct effect of the cationic sol surface on fungal physiology. This antifungal effect is expected to be stable over time, whereas water glass is neutralized by ambient carbon dioxide and thus loses its effectiveness. In addition, silica sols are stable toward leaching, whereas water glass is easily leachable as long as its pH is below 7. Cationic silica sols thus appear to be well suited to protect wood exposed outdoors in hazard class 3 according to EN 335 (2006).

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