DECAY RESISTANCE OF TREATED WOOD WITH FUNCTIONALISED COMMERCIAL SILICONES

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Three commercial silicone emulsions with different functional groups i.e., quat-silicone micro-emulsion (<40 nm particle size), amino-silicone macro-emulsion (110 nm), and silicone macro-emulsion with alkyl-modified side groups (740 nm) were used to protect wood samples against fungal decay. The addition of the emulsions to an agar growth medium revealed that all three silicone formulations inhibited the growth of *Coniophora puteana* and *Trametes versicolor* compared to the controls without silicone. Wood mini-blocks of pine sapwood and beech wood were treated with 2%, 5%, 10%, and 15% concentration of silicone emulsions and tested for their resistance against basidiomycete decay. Quat-silicone and amino-silicone emulsions at higher concentrations imparted resistance of wood to both types of basidiomycetes, while the alkyl-modified silicone formulation did not enhance the resistance. In a soft rot test according to ENV 807, wood treated with an amino-sililcone emulsion showed the lowest weight loss and loss of dynamic MOE. Quat-silicone micro-emulsion had a lower effect, while the alkyl-modified silicone emulsion did not cause reduction in weight and strength loss compared to the untreated controls. The increased resistance against soft rot might be attributed to a lag in fungal colonization rather than to a sustained effect of protection.

Keywords: Amino-silicone; Brown rot; Macro-emulsion; Micro-emulsion; Quat-silicone; Soft rot; White rot

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INTRODUCTION

Wood is preferred as a building material because it is renewable, environmentally friendly, and has a high strength-to-density ratio compared to other materials. Untreated wood, however, can be degraded through the action of micro-organisms at high moisture content and availability of oxygen and nutrients. Three main types of wood decay are reported in the literature according to the appearance of wood following degradation (Eaton and Hale 1993; Hill 2006; Goodell et al. 2008). The most common types of wood decay are brown rot and white rot caused by basidiomycete fungi. Wood decay is also caused by members of the Ascomycotina and Deuteromycotina phyla, commonly termed as soft rot (Eaton and Hale 1993).

Conventional chemical wood preservation is based on a broad spectrum of biocide formulations to combat wood decay fungi (Mai and Militz 2007). However,

consumer awareness and stringent regulations on the use and disposal of wood preservatives has led to a search for novel approaches for wood protection.

Different strategies of wood protection encompass (a) killing the fungi by biocidal agents, (b) using biochemical methods to interfere with fungal decay agents (enzymes, oxidants, redox mediators) to prevent breakdown of wood (e.g. Green and Schultz 2003), or (c) thermal or chemical modification of wood (Hill 2006). It is assumed that chemical modification hinders penetration of low molecular weight diffusible agents required for fungal degradation into the cell wall, because of cell wall bulking and micro-pore blocking (Papadopoulos and Hill 2002; Hill et al. 2005).

Each component of the wood cell wall absorbs moisture to a different extent; hemicelluloses absorb the most, followed by cellulose and lignin (Rowell 1982). This renders wood hygroscopic and enables fungal colonisation and degradation (Schmidt 2006). It is generally believed that fungal colonisation of wood becomes possible when the moisture exceeds the fibre saturation point. Water uptake of wood can be reduced in different ways: either by grafting chemicals onto the OH-groups of cell wall polymers responsible for adsorption of water or by physically blocking the flow paths of water in wood through impregnation of hydrophobic agents (Hill 2006). The first method mainly reduces the absorption of moisture (gaseous water), while the second reduces the uptake of water in the liquid phase.

Natural and mineral oils, waxes, silicones, and silanes are different hydrophobic agents that are used to impregnate wood in order to impart water repellency (Donath et al. 2006). Silicones applied on wood are mainly polydimethylsiloxanes (Mai and Militz 2004), which do not irritate human and animal skin and show minor acute toxicity at oral, dermal, and inhalation exposure. No special target organs displaying toxic effects were identified from long term exposure in animal tests. Marquardt and Schäfer (1994) mentioned that silicones do not cause teratogenic, mutagenic, or carcinogenic effects on animals or aquatic life and thus have no adverse effect on ecosystems.

Several alkyl ammonium compounds such as tertiary amine salts and quaternary ammonium compounds are conventionally utilized as wood preservatives (Preston et al. 1987; Matejuk et al. 2004; Pernak et al. 1998, 2004; Urbanik et al. 1997; Thang and Ruddick 2000). Silicon compounds have been studied for their efficacy against decay fungi in a number of research works (Tanno et al. 1998; Hill et al. 2004; Donath et al. 2004; De Vetter and Van Acker 2005; Donath et al. 2006; Weigenand et al. 2008). In some of these studies it has been argued that amino- and quaternary ammonium groups have strong influence on the decay resistance of wood. Donath et al. (2006) showed that amino silanes under acidic conditions can affect fungal growth. Weigenand et al. (2008) also reported that wood treated with an amino-silicone micro emulsion can resist decay by basidiomycetes. In a previous study, Ghosh et al. (2008) reported on the efficacy of silicones with quaternary ammonium, primary amino and alkyl groups against blue stain and mould fungi in wood along with the material properties in terms of dimensional stability and capillary water uptake. The same emulsions have been studied here to determine the effect on decay resistance of treated wood.

EXPERIMENTAL

Chemicals

Quat-silicone micro-emulsion (QSMiE), amino-silicone macro-emulsion (ASMaE), and alkyl-modified silicone macro-emulsion (AmSMaE) with particle size <40 nm, 110 nm, and 740 nm respectively were supplied by Momentive GmbH (Leverkusen, Germany). All emulsions contained 35% (wt/wt) silicone (Table 1). In order to adjust the treatment concentration, the stock solutions were diluted with demineralised water, taking into account the silicone content (Ghosh et al. 2008).

Table 1. Name and Chemical Properties of Silicone Emulsions Tested

Formulation	Emulsion particle (nm)	Silicone content (%)	Solid content (%)	N-content (mmol g^{-1})	Structure of functional group
Quat silicone micro-emulsion (QSMiE)	<40	35	47	0.25	quaternary ammonium, R^1-$N^+(CH_3)_2$-R^{2*}
Amino silicone macro-emulsion (ASMaE)	110	35	38	0.25	Amino, $(CH_2)_3$-NH_2
Alkyl modified silicone macro-emulsion (AmSMaE)	740	35	38.5	0	Alkyl modified, $(-CH_{12}$-$C_{14})$

* The quat-groups are incorporated into the dimethylsiloxane chain (Polymer-CH_2-$N^+(CH_3)_2$-CH_2-Polymer)

Methods

Growth reduction

The silicone formulations were mixed with agar growth medium prior to fungal inoculation, in order to test their effect on the growth of wood decay fungi. Malt agar solution (4%) was autoclaved at 120°C for 20 min. After a short cooling period, 0.11, 0.21, 0.43, 0.86, and 2.15 g of the silicone stock formulations were added to 75 ml of the malt agar solution to achieve silicone concentrations of 0.05, 0.1, 0.2, 0.4, and 1%. The mixture (25ml) was poured in Petri dishes (90 mm diameter), while malt agar solutions without silicone served as controls. After hardening of the medium, the plates were inoculated at the centre with a mycelium plug (9 mm diameter), which was cut from two weeks old fungal cultures of the brown rot fungus *Coniophora puteana* (CTB 863 A) or the white rot fungus *Trametes versicolor* (BAM Ebw. 15). The Petri dishes were incubated at 22°C and 70% RH for 7 days, and the diameter of mycelium growth was subsequently recorded. The area of mycelium was calculated by subtracting the area of the inoculum and fungal growth was expressed as percentage of the total area of the Petri dish. Three replicate Petri dishes were used for each silicone formulation and concentration.

Treatment of wood

The wood specimens were vacuum-pressure impregnated at 100 mbar (1h) and 12 bar (1h) with the aqueous silicone emulsions. Subsequently, the specimens were dried gradually from 30°C to 103°C and weighed. All treated specimens were subjected to an accelerated leaching procedure according to the European standard EN 84 and again dried gradually from 30°C to 103°C in order to determine the dry mass. The weight percent gain (WPG) was calculated from the dry masses before and after treatment as previously described (Donath et al. 2004). The specimens were then stored in a climate chamber at 20°C and 65 % RH for about 2 weeks in order to reach moisture equilibration.

White and brown rot decay

Resistance of wood treated with silicone emulsions against basidiomycete decay was evaluated using mini-blocks (5 x 10 x 30 mm^3, R x T x L) according to Bravery (1978). The wood blocks were treated with 2, 5, 10, and 15% silicone content, as described above. Treated and untreated beech wood (*Fagus sylvatica* L.) was tested with *T. versicolor* (BAM Ebw. 15) and Scots pine sapwood (*Pinus sylvestris* L.) with *C. puteana* (CTB 863 A). After conditioning (20°C, 65±5% RH) and sterilisation through γ-irradiation (25 kGy), the specimens were aseptically inoculated on 4% malt agar (Scharlau, Spain) in Kolle flasks. In each Kolle flask, three treated samples were exposed together with three control specimens. Nine treated and untreated replicates were tested per treatment over an exposure time of 12 weeks. After incubation, the specimens were oven dried to determine the mass loss.

Soft rot decay

Resistance of wood treated with silicone emulsions against soft rot fungi was assessed at 24°C and 65±5% RH according to ENV 807 (2001). Wood stakes (10 x 5 x 100 mm^3, RxTxL) were treated with 4% and 10% silicone emulsions as described above. Eight replicates were used per treatment. Untreated wood stakes served as controls.

The dynamic modulus of elasticity (MOE_{dyn}) of the specimens was measured in water-saturated condition before exposure to the soil bed, using a Grindosonic (J. W. Lemmers N.V., Leuven, Belgium). During soil exposure MOE_{dyn} was measured periodically after 8, 16, 24, and 32 weeks of incubation, and weight loss was measured after 32 weeks exposure. MOE_{dyn} was calculated using the following equation,

$$MOE_{dyn} = \frac{4 \times \pi^2 \times l^4 \times f^2 \times \varphi \times A}{m_l^{\,4} \times I} \times (1 + \frac{I}{l^2 \times A} \times K_l); \qquad I = \frac{b \times h^3}{12}$$

where, l = length (mm), f = frequency (KHz), φ = density (g mm^{-3}), A = area of cross section (mm^2), K_1 = 49.48, m_1 = 4.72, I = moment of inertia (mm^4), b = width (mm), and h = height (mm). Loss of MOE_{dyn} after a certain period of exposure was calculated as the percentage of strength loss from the initial strength of the respective sample.

RESULTS AND DISCUSSION

Growth Reduction

All silicone emulsions reduced the growth of *C. puteana* and *T. versicolor*, when added to the agar medium (Fig. 1). With the brown rot fungus *C. puteana*, there was no clear difference in the degree of growth reduction between the three silicone emulsions. The alkyl-functionalised AmSMaE tended to inhibit the growth of *C. puteana* to a somewhat greater extent than the other silicone formulations. Even the lowest silicone concentrations of 0.05% inhibited the growth of the brown rot fungus by approx. 50% compared to the control, irrespective of the silicone type (Fig. 1A). In case of the white rot fungus *T. versicolor*, however, the amino-silicone macro-emulsion (ASMaE) allowed only a growth area by 8.3% at the lowest silicone concentration tested (0.05%), while the mycelium grew by 25.1% in presence of the quat-silicone micro-emulsion (QsMiE) and by 53.8% in presence of the alkyl-silicone macro-emulsion (AmSMaE, Fig. 1B).

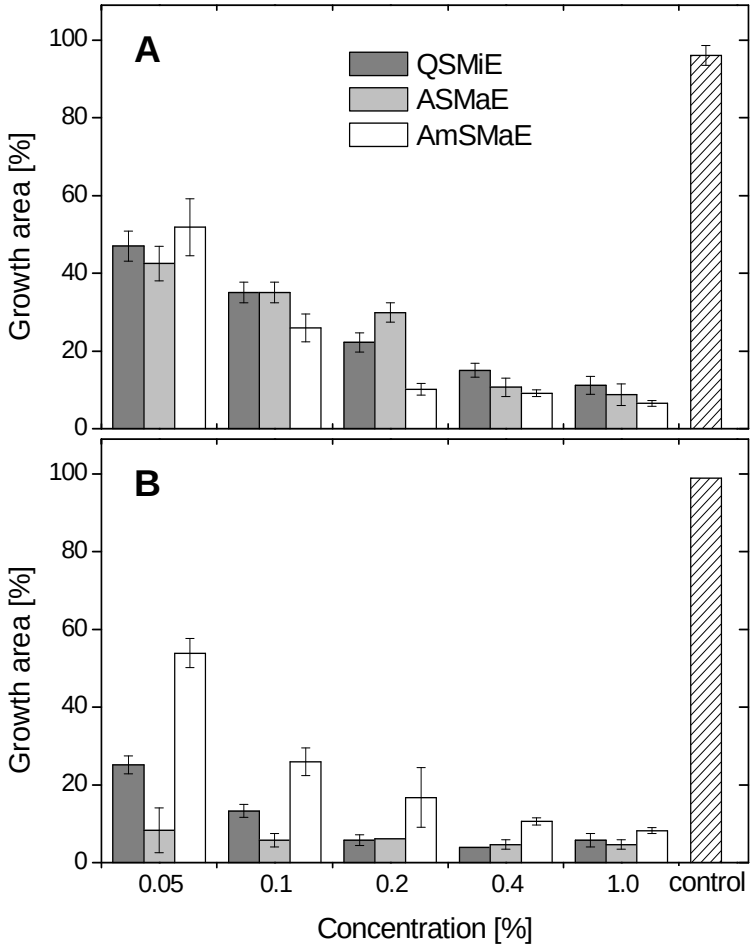

Fig. 1. Growth area (%) of *Coniophora puteana* (A) and *Trametes versicolor* (B) on agar media supplemented with QSMiE, ASMaE and AmSMaE. The area was related to the total area of the Petri dish.

Basidiomycete Decay

A mini-block test was adopted to evaluate the resistance of silicone-treated wood against white and brown rot decay (Bravery 1978). Mini-blocks display a greater surface-volume ratio than the test specimens used in the European standard EN 113 (1996) and allow for faster fungal colonization. Silicones were previously shown to impart water-repellent properties to wood (Ghosh et al. 2008; Weigenand et al. 2007). A relatively long incubation time (12 weeks) was therefore used in this study in order to rule out the possibility that the reduction in mass loss was only due to hydrophobation of the wood sample and, thus, a prolonged time is needed for fungal colonisation (Weigenand et al. 2008).

Treatment of pine and beech mini-blocks with 2, 5, 10, and 15% silicone emulsions resulted in linearly increasing weight percent gains (WPG) in the wood. The WPG in pine was generally somewhat higher than that of beech due to the lower density of pine (Table 2).

Table 2. Weight Percent Gain of Pine Sapwood and Beech Wood Treated with Different Concentrations of Silicone Emulsions.

Formulation	Wood species	2%	5%	10%	15%
QSMiE	Pine	2.1±0.3	5.7±0.8	15.6±1.3	20.2±1.3
	Beech	1.7±0.6	4.4±1.3	10.6±1.5	12.4±2.7
ASMaE	Pine	1.2±0.3	4.7±0.5	10.4±3.1	16.0±1.2
	Beech	1.0±0.5	3.0±1.0	8.0±1.9	9.5±2.7
AmSMaE	Pine	1.5±0.4	7.0±0.5	14.0±1.2	16.9±2.5
	Beech	1.3±0.8	5.2±0.8	8.8±2.1	12.4±2.4

At low treatment concentration (2%) the mass loss of pine caused by *C. puteana* (Fig. 2A) or of beech caused by *T. versicolor* (Fig. 2A) did not show any significant difference from that of untreated samples. At higher silicone concentrations (5 - 15%), quat-silicone micro-emulsions (QSMiE) and amino-silicone macro-emulsion (ASMaE) caused increased resistance to brown and white rot decay. The macro-emulsion (AmSMaE) did not increase the resistance of wood, irrespective of the concentration used. Pine wood treated with 5% amino-silicone (ASMaE) showed lower mass loss (19.3%) than pine treated with QSMiE (47.2%) and AmSMaE (41.6%) after incubation with *C. puteana*. The same trend was found with 10% treatment concentration. Only 2% mass loss through *C. puteana* was found in pine treated with the amino-silicone ASMaE (15% concentration and 16% WPG).

The decay resistance of beech against *T. versicolor* increased with increasing concentration of amino- (ASMaE) and quat-silicone (QSMiE), while the alkyl modified silicone (AmSMaE) did not show any effect. The minimum mass loss was 17.7% (at 12.4% WPG) and 10.2% (at 9.5% WPG), respectively, with QSMiE and ASMaE (Fig. 2B).

Weigenand et al. (2008) recently reported on relatively high efficacy of amino-silicone micro-emulsion against wood decay by white and brown rot fungi; however, they

did not present a comparison with other types of functionalised silicones. Amino-functional silanes showed efficacy against wood decay fungi (Donath et al. 2006). This was attributed to the effect of cationic amino groups on fungal physiology, whereas the alkyl groups in the silane system had a minor effect on decay resistance.

The results of the decay test are partly not in accordance with the test on growth reduction reported above. In the latter, all silicone emulsions exerted strong reduction of fungal growth – even at very low concentrations, irrespective of their functional side-groups. Only the amino- and the quat-silicone, however, clearly reduced fungal decay, when the silicones were incorporated into the wood and after a leaching test; the alkyl-functionalised silicone did not exhibit any efficacy. In addition, the amount of amino- or quat-silicone that was necessary to clearly reduce the weight loss of wood was higher than it could be deduced from the growth reduction test.

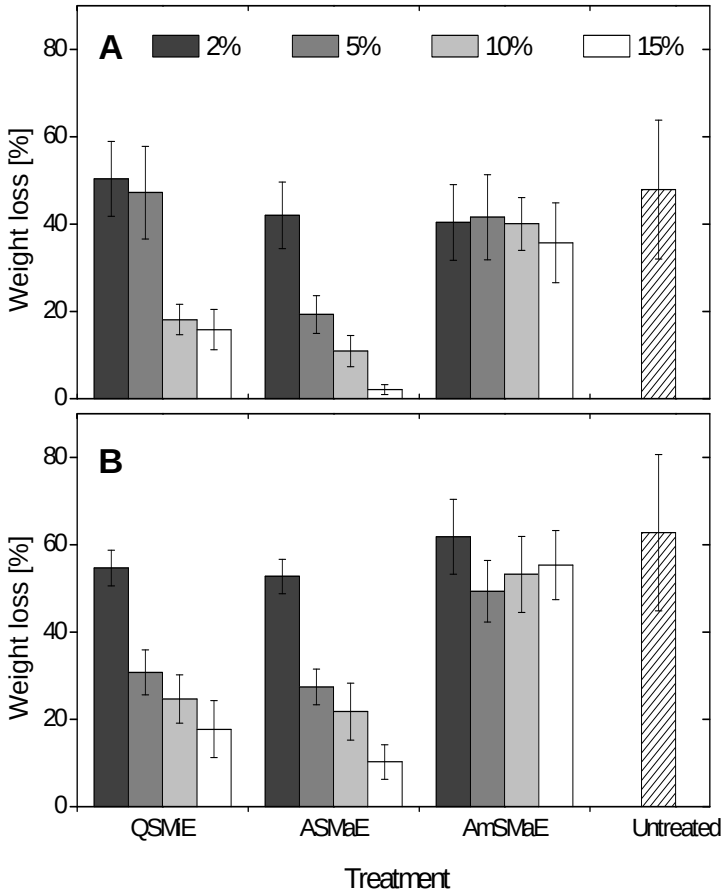

Figure 2: Mass loss of pine mini-bocks caused by *Coniophora puteana* (A) and of beech mini-bocks caused by *Trametes versicolor* (B) after 12 weeks incubation according to Bravery (1978)

Three main reasons could be mentioned for the discrepancy between the wood decay and the growth reduction test: (1) the mode of action of silicone in agar medium could be different from wood. It is known that silicone shows a tendency of spreading on various surfaces and of forming a thin layer (Noll 1968). This could lead to different effects on fungal growth in agar and in the wood matrix due to changes of the substrate

surfaces. (2) Silicones could undergo stronger adhesion to the wood matrix than to the agar polysaccharide. This could cause an immobilisation of the silicone in wood and, in case of an effect on fungal physiology, reduce the availability of silicone to prevent fungal colonisation. (3) The emulsifiers that are used to produce the silicone emulsions could have a detrimental effect on the fungal growth. These emulsifiers are available in the agar test, but should be washed out in the leaching procedure according to EN 84 (1996) prior to the decay test. (4) The alkyl-modified silicone macro-emulsion (AmSMaE) with a particle size of 740 nm does not penetrate as deep into the wood as ASMaE or QSMiE (not shown). Thus, the inner parts of wood treated with AmSMaE could remain unprotected and be decayed at the same velocity as untreated wood.

Soft Rot Decay

Untreated pine sapwood showed higher resistance to soft rot decay than untreated beech wood in a soil bed test according to the European pre-standard ENV 807 (Fig. 3). Rapid losses of cellulose followed by hemicelluloses and lignin is observed in beech during soft rot decay (Levi and Preston 1965), whereas in Scots pine the change is chemical composition occurs at a slower rate (Mohebby and Militz 2002). The higher mass and strength loss in beech wood compared to that of Scots pine might be ascribed to higher lignin content in pine.

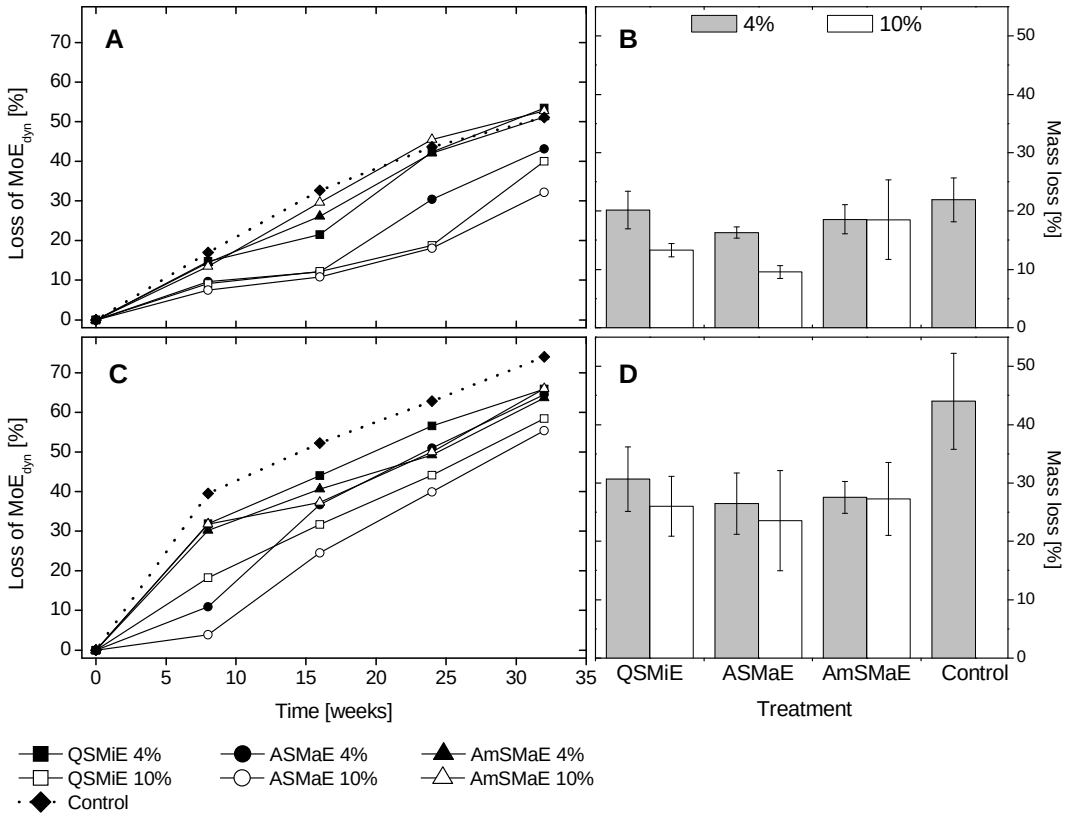

Figure 3: Loss of dynamic MOE of pine sapwood (A) and beech wood (C) during 32 weeks exposure in a soil bed according to ENV 807 and final weight at the end of the exposure (B: pine sapwood; D: beech)

Amino-silicone (ASMaE) and quat-silicone (QSMiE) reduced the strength loss in both pine and beech wood, while ASMaE displayed the highest efficacy. Higher silicone concentration caused greater reduction in strength loss (Fig. 3A, C). In contrast, alkyl-modified silicone (AmSMaE) clearly showed no resistance to decay at both treatment concentrations (4% and 10%) (Fig. 3A, B, C, D).

Strength loss reduction caused by ASMaE and QSMiE, however, occurred only at the beginning of the exposure (first 8 weeks). After this period, the pace of the strength loss was not different from that of the untreated specimens. The differences in mass loss after 32 weeks (Fig. 3B, D) are basically attributable to this initial phase, which might be due to a lag in colonisation of the treated samples. The reduction of colonisation and initial decay could be due to hydrophobation and an effect of silicone on the wood surface properties. Growth of fungal hyphae in the treated wood might be impeded due to changes in the surface energy in wood. It was recently shown that the amino-silicone emulsion ASMaE imparted a comparable degree of hydrophobation to wood as the alkyl-modified silicone emulsion AmSMaE, while the water-repellent effect of the quat-silicone emulsion QSMiE was lower (Ghosh et al. 2008). The reduction of fungal colonisation through ASMaE can therefore not mainly be explained by the hydrophobation of wood, but by the effect of amino-groups attached to the siloxane backbone on the mode of fungal colonisation. The latter was previously shown for basidiomycete fungi (Weigenand et al. 2008) and in this study as well as for staining fungi such as blue stain and moulds (Ghosh et al. 2008). Nevertheless the silicone formulations used in this study are commercial ready-to-use formulations and might contain emulsifiers having unknown effects on decay fungi. Further research is therefore necessary to study the effect of the interaction of silicone and wood and of emulsion particles on the decay resistance caused by silicone emulsions. A study on solutions of silicones with different molecular weights and functional groups, which are free of emulsifiers, is in progress

CONCLUSIONS

1. Commercial emulsions of silicones bearing different functional groups, i.e., quaternary ammonium, primary amino, and alkyl groups were recently studied against blue stain and mould fungi (Ghosh et al. 2008). In this study, decay resistance of silicone-treated wood was tested against wood decay fungi. Even though all the silicone emulsions could inhibit fungal growth on agar medium at very low concentrations, only the amino- and quat-silicones showed effectiveness when incorporated into the wood matrix, while the alkyl-silicone displayed no effect of preservation.
2. Quaternary ammonium compounds are widely used as wood preservative agents (Preston et al. 1987; Matejuk et al. 2004; Pernak et al. 1998, 2004; Urbanik et al. 1997). In this study, however, silicones bearing primary amino-groups were more effective in preventing wood decay than those bearing quat-groups. It is unlikely that the tested silicones are taken up and metabolised by the fungi, because they have a relatively high molecular weight and are very inert towards enzymatic transformation (Noll 1968). Therefore, the effect of silicones on fungi appears to

be based on a change in wood's surface properties rather than on a direct effect on fungal physiology.

3. Amino- and quat-silicones bear a higher charge than alkyl-silicones. The positively charged amino- and quat-groups might form ionic bonds with carboxylic groups, e.g. in hemicelluloses. In addition, amino-groups might bind covalently with hemiacetal groups, the reducing end-groups of cell wall polysaccharides. This should result in a stronger interaction of amino-silicone (and partly quat-silicone) with the wood cell wall as compared to uncharged alkyl-silicones.

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