

Performance of Copper-Ethanolamine-impregnated Scots Pine Wood during Exposure to Terrestrial Microorganisms

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Copper-ethanolamine-based wood preservatives are still the most important solutions for protecting wood in ground applications in Europe. Wood in the ground is exposed to a variety of organisms that can act synergistically. In order to simulate these conditions in the laboratory, Scots pine (*Pinus sylvestris*) specimens impregnated with copper-ethanolamine preservative of three different concentrations ($C_{Cu} = 0.125\%$, 0.25% , and 0.5%) were exposed to three different soils according to procedure ENV 807, for periods between 12 and 32 weeks. After the relevant period of exposure, samples were isolated, and their mass loss, bending strength, and modulus of elasticity were determined. In the final step, the remaining copper in the samples was determined. The results showed that, in spite of significant copper leaching, the tested copper-ethanolamine-treated wood exhibited good performance in ground applications. Furthermore, a good correlation was found between the mechanical properties and mass loss, regardless of the chemical treatment applied.

Keywords: Copper-ethanolamine; *Pinus sylvestris*; Terrestrial microorganisms; Cu leaching; Bending strength; Soft rot

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INTRODUCTION

Copper-based wood preservatives are the most important group of biocidal products that are allowed for the protection of wood in ground contact after implementation of the Biocidal Products Directive (98/8/EC 1998) in 2006 in the European Union. The majority of competitive biocidal products were banned and thus removed from the market. One of the key reasons for their broad use is the good relationship between efficacy and toxicity (Richardson 1997). However, copper-based wood preservatives also have limitations. The most important of these is insufficient fixation. Copper therefore has to be combined with additives. In the past, it was mainly combined with chromium. However, during implementation of the Biocidal Products Directive (98/8/EC 1998), the use of wood preservatives containing chromium was considerably limited and even banned in some EU countries. In order to meet legislative requirements, chromium compounds in wood preservatives were replaced with amines, predominately ethanolamine (Pankras *et al.* 2012; Tascioglu *et al.* 2013) or they are prepared in the form of so-called micronized copper (Matsunaga *et al.* 2009; Wu *et al.* 2012), which limits copper leaching from wood. Unfortunately, fixation of copper-ethanolamine-based wood preservatives is not as effective as the fixation of copper-

chromium ones (Humar *et al.* 2001; Cooper and Ung 2009). There are several reasons for inadequate copper fixation. Most are linked to the depolymerisation of lignin due to the presence of ethanolamine. Another key reason for copper leaching from wood during use is related to microbial action. Fungi and bacteria that are naturally present in soil excrete copious amounts of organic acids: oxalic, acetic, formic acid, *etc.* (Steenkjær-Hastrup *et al.* 2012). These organic acids react with copper-ethanolamine complexes to form new complexes that can easily be leached from wood (Cooper *et al.* 2000; Humar *et al.* 2004a; Terzi *et al.* 2012). Additionally, wood decay fungi can actively translocate copper from the wood to the surroundings (Schilling and Inda 2011). The aim of this study was to elucidate the importance of these processes and to determine how much copper remained in wood after wood degradation by soil microorganisms.

EXPERIMENTAL

The experiment was performed on Scots pine sapwood specimens (*Pinus sylvestris*) of the following dimensions: 10 mm × 5 mm × 100 mm. Samples were free of any physical defects and biological damage. They were impregnated with Silvanolin commercial copper-ethanolamine solution (Silvaproduct, Slovenia) of different strengths, as shown in Table 1, in order to achieve different retentions. Silvanolin consists of copper hydroxide (Cu), alkyldimethylbenzylammonium chloride (ADBAC), boric acid (B), octanoic acid, and ethanolamine. The ratio between active ingredients was the same in all solutions used. Before and after impregnation, the copper concentration in the preservative solution was determined with a Twin X XRF machine (Oxford Instruments, UK) in order to ascertain the possible influence of selective absorption.

Table 1. Composition of the Preservative Solutions Used for Impregnation

c_{Cu} (g/L)	$c_{\text{ethanolamine}}$ (g/L)	c_{ADBAC} (g/L)	c_{B} (g/L)
1.25	7.2	1.25	0.625
2.5	14.4	2.5	1.25
5	28.8	5	2.5

Cu - copper hydroxide, ADBAC - alkyldimethylbenzylammonium chloride, B - boric acid

A standard full cell process was used for impregnation (vacuum 0.1 mbar; 30 min; pressure 9 bar; 4 h; vacuum 0.1 mbar; 15 min) (Unger *et al.* 2001). The uptake of preservative solutions was determined gravimetrically. Copper retention was calculated from the solution uptake and the copper concentration in the preservative solution. After impregnation, samples were conditioned for four weeks, as prescribed by standard EN 113 (2004). In the first week the samples were kept in closed chambers, the second and third weeks in half-open chambers, and the fourth week in open chambers. The samples were then artificially aged according to procedure EN 84 (2002). The EN 84 standard requires vacuum pre-treatment of the specimens with water as the first step of leaching; specimens thus take up as much water as possible. Samples impregnated by the same treatment were positioned in the vessel and immersed in distilled water. The volume of the water was five times greater than the volume of the samples. The water was not stirred during leaching. The water was changed ten times, every 14 days.

Wet specimens were exposed to various soil organisms, as prescribed by ENV 807 (2004). There were three types of soils used: compost being the most active, forest

soil to represent intermediate activity, and field soil as an example of inactive soil. These three soil types were chosen to observe the effects of long-term leaching (soil from field), on the one hand, and to elucidate the influence of various spectra of microorganisms (compost), on the other. Non-impregnated and copper-ethanolamine-treated specimens were inserted in the soil, with 9 cm of the specimen in the soil and the remaining 1 cm above soil level. The moisture content of the soil was regularly monitored and kept at the level prescribed by the standard. After the periods shown in Table 3, six parallel specimens per concentration/soil were isolated and used for further analysis. The samples were first oven dried (103 ± 2 °C; 24 h) and their moisture content and mass loss were determined gravimetrically. In the next step, the bending strength was determined according to EN 310 (1996) with a static three-point bending test on a Zwick Z005 universal testing machine. Specimens were incubated in a standard climate ($65 \pm 5\%$ relative humidity (RH); 20 ± 2 °C) until a constant mass was achieved. The specimens were tested for bending strength immediately after incubation in the standard climate. After the test, the modulus of elasticity (MOE) and bending strength were calculated.

Broken specimens were then milled in a Retch mill SM 2000 and pressed with a Chemplex Spretro pellet press (Chemplex Industries Inc., USA) into tablets ($r = 16$ mm; $d = 5$ mm), as required for XRF measurements. The copper in the wood was determined with a TwinX XRF spectrometer (Oxford Instruments, UK). Measurements were performed with a PIN detector ($U = 26$ kV, $I = 112$ μ A, $t = 360$ s). Since the volume of the specimens was fairly small, all specimens were milled together, homogenized, and analyzed at the same time. Samples that were impregnated with the same solution strength and exposed to the soil organisms for the same time were grouped together. XRF measurement was performed on three parallel tablets.

RESULTS AND DISCUSSION

Scots pine sapwood is very easy to impregnate (EN 350-1 1994). Furthermore, the cross-section of the wood used was quite small, which was reflected in the fairly high uptake of the preservative solution – 794 kg/m^3 on average. The concentration of active ingredients did not influence the uptake of preservative solutions (Table 2), but it did considerably influence both copper retention and total retention. The retention of copper varied between 0.99 kg/m^3 and 3.98 kg/m^3 , depending on the concentration applied. Wood impregnated with similar copper retention is usually used for ground contact applications.

Table 2. Retention of Copper and Copper-Ethanolamine Preservative in Scots Pine Sapwood Specimens

c_{Cu} (%)	Uptake of preservative solution (kg/m^3)	Cu retention (kg/m^3)	Silvanolin retention (kg/m^3)
0.125	791 (23)	0.99 (0.03)	8.2 (0.25)
0.25	792 (26)	1.98 (0.07)	17.3 (0.51)
0.5	796 (31)	3.98 (0.15)	35.1 (1.1)

As already mentioned in the methods section, three types of soil were used. From the mass loss of the control specimens shown in Table 3, it is evident that the microorganisms in the soils did not show the same aggressiveness. Compost was found to be

the most aggressive, while field soil was almost inactive. Control specimens that were exposed to compost for 32 weeks lost approximately 31% of their mass, samples exposed to forest soil lost 11% of their initial mass, while specimens exposed to field soil remained almost non-degraded (2.2%) (Table 3). The first evidence of degradation of non-impregnated pine wood was visible after 12 weeks. The mass loss of specimens exposed to forest soil and compost increased with time, while the mass loss of specimens exposed to field soil remained almost constant. Comparing these results to data in the literature, it is evident that mass losses of the control specimens exposed to compost are comparable to the mass losses of pine wood after 40 weeks of exposure to compost soil (Westin and Alfredsen 2011).

Table 3. Mass Loss of Scots Pine Sapwood Specimens Treated with Copper-Ethanolamine-based Solutions and Exposed to Different Soils for Periods Between 12 and 32 Weeks

c_{Cu} (%)	Soil	Weeks of exposure			
		12	18	24	32
		Mass loss (%) (n = 6)			
control	forest	1.2 (0.5)* cd**	3.7 (1.4) d	10.9 (2.7) d	10.7 (3.3) e
	compost	15.4 (1.1) h	22.6 (1.9) g	29.2 (2.1) f	30.8 (4.5) h
	field	2.8 (0.6) e	1.9 (0.5) bc	3.0 (2.2) bc	2.2 (2.6) c
0.125	forest	0.9 (0.1) cd	1.5 (0.3) b	1.4 (0.5) ab	1.3 (0.4) bc
	compost	6.8 (0.7) g	11.9 (0.9) f	20.2 (1.6) e	21.4 (1.6) g
	field	-0.5 (0.4) b	-0.9 (0.4) a	0.1 (0.9) a	-1.3 (1.6) ab
0.25	forest	0.9 (0.3) c	1.3 (0.4) b	1.0 (0.6) ab	1.2 (0.3) bc
	compost	3.9 (0.5) f	6.7 (1.0) e	10.3 (2.4) d	14.2 (1.3) f
	field	-1.4 (0.2) a	-1.6 (0.5) a	-0.1 (0.6) a	-1.7 (1.4) a
0.5	forest	0.7 (0.2) c	1.2 (0.3) b	0.7 (0.2) ab	1.0 (0.3) abc
	compost	1.4 (0.4) d	3.3 (0.6) cd	4.5 (0.9) d	6.8 (1.2) d
	field	-1.5 (0.4) a	-1.6 (0.8) a	-0.1 (0.4) a	-1.0 (0.7) ab

*Values in brackets stand for standard deviations

** Different letters represents different homogeneous groups within the same exposure time

Impregnation with copper-ethanolamine solution considerably slowed down the degradation processes. Even the lowest concentration of copper ($c_{Cu} = 0.125\%$) was enough to prevent degradation of impregnated specimens in contact with forest and field soil (Table 3). However, microorganisms in compost were considerably more aggressive. Namely, a mass loss of 21.4% was measured from the samples impregnated with preservative solution of the lowest Cu concentration after 32 weeks of exposure. Impregnation of samples with twice the concentration was only slightly more effective (mass loss = 14.2%). Even impregnation of samples with the highest concentration ($c_{Cu} = 0.5\%$) was not enough to prevent fungal degradation in compost soil completely (mass loss = 6.8%). Special attention has to be given when comparing data between wood species, as the species of wood will influence mass loss and the retention. Reference literature data (Westin and Alfredsen 2011) showed that even impregnation of specimens with a preservative solution based on chromium, copper, and arsenic compounds (CCA) with a retention of 9 kg/m^3 , which is generally recommended for ground applications,

does not ensure complete protection against terrestrial microorganisms in all soil conditions. Westin and Afredsen (2011) reported that mass losses between 1% and 12% were determined with CCA-treated pine specimens (9 kg/m^3) after 40 weeks of exposure in different soils. This comparison clearly indicates that the conditions of ENV 807 testing are very severe, and that low mass losses of impregnated specimens are expected/tolerated even for wood impregnated with high performance wood preservatives.

Since mass loss is only one indicator of the chemical changes in wood due to degradation, the bending strength and modulus of elasticity of the exposed specimens were also determined. Loss of mechanical properties is usually the first sign of degradation (Curling *et al.* 2002) and is evident before changes in mass occur. As can be seen from Figs. 1 and 2, there was a correlation between the mass loss of the specimens and the MOE ($r^2 = 0.51$) and MOR ($r^2 = 0.54$). The different activities of the soils used in this experiment are also evident from changes in MOR and MOE. Compost was the most active and field soil the least active (Table 4), as indicated from mass loss data.

Table 4. Bending Strength of Copper-Ethanolamine-treated Scots Pine Wood after Different Periods of Exposure to Different Soils

c_{Cu} (%)	Soil	Weeks of exposure				
		0	12	18	24	32
		MOR (N/mm^2) (n = 6)				
control	forest		110 (7)*bcde**	89 (9) a	66 (10) a	73 (8) a
	compost	120 (7) a	92 (9) a	84 (0) ab	73 (7) ab	69 (13) a
	field		102 (10) ab	98 (2) abcd	99 (5) c	87 (11) a
0.125	forest		120 (7) ef	104 (3) bcde	99 (7) c	107 (6) cdf
	compost	121 (6) a	101 (10) abc	88 (10) a	76 (3) ab	74 (10) ab
	field		127 (8) f	108 (8) cdef	113 (5) cdf	115 (9) f
0.25	forest		103 (9) bcd	105 (5) cde	105 (6) cd	105 (8) cdf
	compost	116 (6) a	98 (7) ab	98 (9) abc	94 (3) bc	92 (7) bc
	field		114 (14) de	114 (4) ef	120 (6) df	116 (8) f
0.5	forest		111 (11) cde	109 (12) def	101 (9) cd	107 (5) cdf
	compost	114 (7) a	112 (7) cde	108 (12) cdef	99 (11) c	96 (4) cd
	field		107 (7) bcd	117 (8) f	125 (8) f	114 (11) df

*Values in brackets stand for standard deviations

** Different letters represents different homogeneous groups within the same exposure time

The control specimens and specimens impregnated with a low Cu concentration in compost showed the first signs of changes in bending strength after 12 weeks of exposure. The bending strength of the control specimens exposed to compost soil decreased from an initial 120 N/mm^2 to 92 N/mm^2 after 12 weeks of exposure and down to 69 N/mm^2 after 32 weeks of degradation. The lowest MOR value (66 N/mm^2) was determined for control specimens that were in contact with forest soil for 24 weeks, in spite of the fact that the lowest mass loss was not determined from these specimens. However, specimens that were treated with the highest concentration of copper-ethanolamine solution ($c_{\text{Cu}} = 0.5\%$) showed no MOR changes after 12 and 18 weeks of exposure in compost. As reported for mass loss, the first signs of MOR losses became

evident after 24 and 32 weeks of exposure (Tables 3 and 4). Comparing these results to those from previous studies performed on specimens exposed to brown rot fungi, it is evident that soil microorganisms do not decrease the mechanical properties of wood as prominently as brown rot fungi. For example, after 8 weeks of exposure of Norway spruce (*Picea abies*) specimens to brown rot fungus *Gloeophyllum trabeum*, the specimens lost, on average, more than 75% of MOE (Humar *et al.* 2006), while 32 weeks of exposure of pine wood specimens to the most aggressive compost caused a MOE loss of only 45% (Fig. 2).

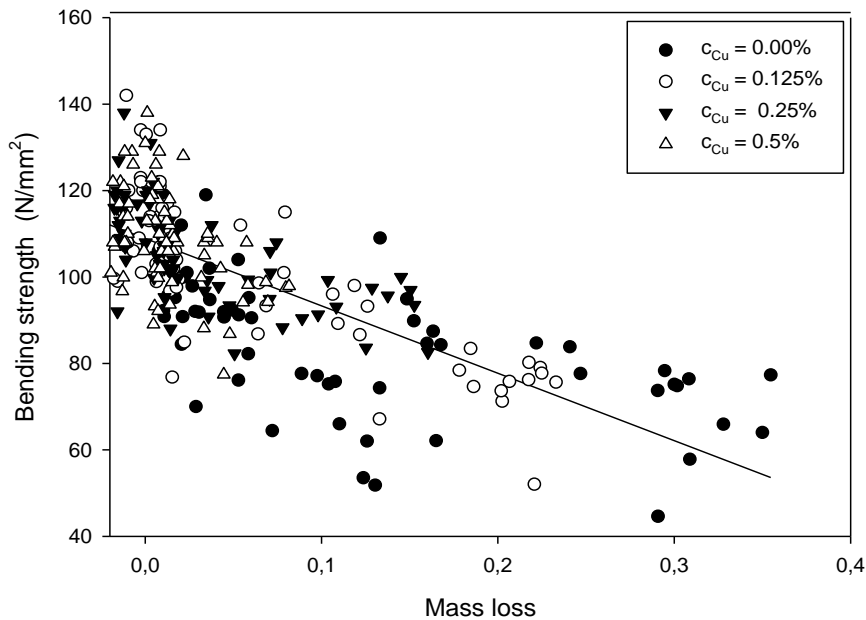


Fig. 1. Correlation between mass loss and bending strength of copper-ethanolamine-treated pine specimens after exposure to soil microorganisms

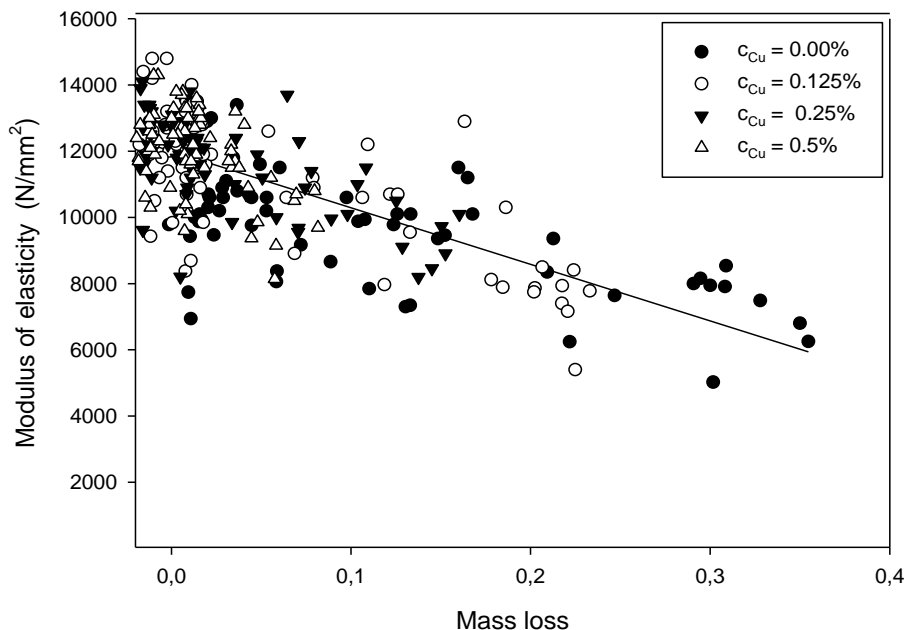


Fig. 2. Correlation between mass loss and modulus of elasticity (bending) of copper-ethanolamine-treated specimens after exposure to soil microorganisms

Degradation by microorganisms is not the only reason for mass loss changes of copper-ethanolamine-treated specimens. As already elucidated earlier (Humar *et al.* 2008), non-evaporated ethanolamine causes depolymerisation of lignin, which results in the deterioration of mechanical properties as well as copper leaching (Humar *et al.* 2008). However, since there was no noticeable loss of MOR and MOE from specimens exposed to almost non-active field soil, this mechanism is presumably not the key one for loss of MOE and MOR. It can therefore be presumed that soil-inhabiting microorganisms are the most important cause of the deterioration of mechanical properties.

Finally, copper leaching from the specimens was examined. Initial EN 84 leaching resulted in some loss of the active ingredients from wood. Namely, the most intense leaching was observed from specimens impregnated with the solution of the highest copper concentration ($c_{Cu} = 0.5\%$) (21%) and the lowest from specimens impregnated with an aqueous solution of the lowest copper concentration (9%). This copper leaching predominately originates from the leaching of copper deposits on the surface of the specimens and from copper crystals in the cell lumina. When interpreting leaching, it must be kept in mind that the specific surface (surface-to-volume ratio) of the specimens was fairly high, so leaching was considerably more prominent than has been reported for specimens of larger dimensions. Exposure of the impregnated specimens to the soil also resulted in fairly significant leaching. However, in contrast to mechanical properties, leaching data and performance data were not clearly correlated. The highest leaching rates were determined from specimens exposed to forest soil, in which medium decay rates were determined. Surprisingly, considerable leaching was already determined after 12 weeks of exposure to forest soil. Twelve weeks of exposure to forest soil resulted in 75% loss of copper from the specimens impregnated with the preservative solution of the highest concentration. Leaching continued throughout the exposure and, after 32 weeks, only 15% of copper remained in the impregnated wood (Table 5). Nevertheless, the remaining copper was sufficient to protect the wood against decay organisms in the forest soil (Table 3). Similar, but slightly lower leaching rates were also determined from specimens treated with lower concentrations of copper (Table 5). Unfortunately, with specimens impregnated with solutions of the lowest concentration, the remaining copper after 32 weeks of exposure was insufficient to protect the wood against forest soil organisms.

There are several reasons for such extensive leaching from wood in contact with forest soil, as well as with other types of soil. The most important one is related to pH. Forest soil is fairly acidic, with a pH value of 3.5, and copper leaching from wood impregnated with copper-ethanolamine solutions in acidic conditions is much more severe. Humic acids in particular are one of the forest soil components with the highest influence on copper leaching (Kartal *et al.* 2007). Humic acids contain carboxyl and phenolic OH groups, which play an important role in the solubility of heavy metals. A study by Cooper *et al.* (2001) showed that the effect on CCA leaching increased with the amount of humic acid in the leaching solution and that copper was most affected. In the presence of organic acids, new water-soluble complexes are formed that are easily leached from wood (Humar *et al.* 2004b). Some fungi also have the ability to actively translocate copper (Pohleven *et al.* 1999). The importance of the presence of organic acids and fungal translocation is further supported by the less extensive leaching of copper from wood in contact with field soil. This soil was less acidic and contained fewer organic acids. Since even the control specimens were not degraded, it can be presumed that no microorganism-mediated translocation occurred. It can therefore be concluded

that leaching of copper from impregnated wood in contact with field soil was caused by only pure, diffusion-driven leaching.

Table 5. Remaining Percentage of Copper in Copper-Ethanolamine-treated Specimens after Different Periods of Exposure to Different Soils

C _{Cu} (%)	Soil	Weeks of exposure				
		0 (after EN 84)	12	18	24	32
		Remaining copper (%)				
0.125	forest		45	26	22	24
	compost	91	59	51	40	25
	field		67	64	63	62
0.25	forest		29	24	19	17
	compost	88	62	53	51	42
	field		66	61	53	54
0.5	forest		26	22	16	15
	compost	79	47	44	41	36
	field		53	50	44	42

Comparing this data to data in the literature on leaching in ground applications after five years of exposure (Evans 2002), it is evident that fairly extensive leaching from wood impregnated with copper-ethanolamine has also been reported. After five years of exposure, between 20% and 40% retained copper was leached. However, the conditions for copper leaching in field test sites are not as consistent as in the laboratory, due to the dry season in summer and freezing temperatures in winter.

CONCLUSIONS

1. The highest activity of soil microorganisms was evident with compost soil and the lowest with field soil.
2. The copper-ethanolamine preservative exhibited good performance in soil.
3. During exposure of copper-ethanolamine-treated wood, there was fairly significant copper leaching. However, the remaining copper was sufficient to protect the wood in most conditions.
4. Comparison of the mass loss data and the mechanical properties revealed a fairly good correlation between these two parameters.

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