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compounds against *Coniophora puteana*.**

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ABSTRACT

The aim of the research was to increase durability of Scots pine wood treated with 3-Iodo-2-propynyl butylcarbamate (IPBC) through organosilicon compounds. The IPBC was combined with organosilicon compounds (alkylsilane and alkylsilane with amino groups) for supporting its antifungal properties. The mycological examination against *Coniophora puteana* was performed to determine the resistance of wood treated with the tested preservatives. The ergosterol identification in treated wood allowed to confirm the fungal activity of *C. puteana*.

Key words: IPBC, organosilicon compounds, decay, ergosterol

INTRODUCTION

Since many popular active ingredients have been withdrawn from DIY wood preservatives` market, IPBC is currently the main alternative for PT7 (wood coatings), PT8 (wood preservatives) and even PT6 (in cane preservatives) according to BPD. IPBC is one of the most commonly used fungicides in wood protection (Reinprecht 2007). Toxicity of IPBC and its allergic properties has been extensively studied and described in the literature (Hernando 2003, Edlund 1999, Bryld 2001). IPBC is an inhibitor of acetylcholinesterase in animals and it is a highly effective fungicide and bactericide. Its main characteristic is extreme activity towards mold, bluestain and alternaria fungi. Preservatives containing IPBC exhibit potential fungistatic properties against wood destroying fungi (Hansen 1984). Despite many advantages also this fungicide shows some detrimental effects. One of the main defect of IPBC is its low stability in solvent-based preservatives. There are the following frequent destabilizing factors: i.a. water action, UV radiation, high temperature, bacteria or iron pigments.

The benefits of IPBC-based formulations containing organosilicone compounds have been already shown in numerous papers (Donath 2006, De Vetter 2009, De Vetter 2010, Mazela 2012).

METHODS

Sapwood of Scots pine (*Pinus sylvestris* L.) in the form of samples with dimensions 0,75 x 25 x 50 mm (the last dimension along the fibers) were used in the study. Cross-sectional areas of species were blocked to prevent penetration of impregnation of wood along the grain. The examined preservatives were solvent-based formulations consisting of biocide IPBC [Lanxess Preventol MP 100] and organosilicon compounds: aminethyl-aminpropyl trimethoxysilane (AEAPTMO) [Biesterfeld BSC Silane A020], methyltrimethoxysilane (MTMO) [Biesterfeld OSF Silane 6070] and aminpropyl trimethoxysilane (APTMO) [Biesterfeld BSC Silane A010]. There were used two concentrations of IPBC: 0.05 or 0.5% (v/v), and a

mixture of organosilicon compounds at 5% of the total concentration. Samples were treated with the soaking method for a period of 60 min. The control samples were made of pine wood. The isolated compounds were used as reference material. The uptake standard preservative range was between 150 and 230g/m². The chemical composition of the prepared systems is presented in Table 1.

Table 1. Chemical composition of the examined preservatives.

Code symbol	IPBC concentration (% v/v)	Silane	Silane concentration (% v/v)
A	0,05	-	-
B	0,05	MTMOS	5
C	0,05	AEAPT MOS	5
D	0,05	APT MOS	5
E	0,05	MTMOS:AEAPT MOS	2,5:2,5
F	0,5	-	-
G	0,5	MTMOS	5
H	0,5	AEAPT MOS	5
I	0,5	APT MOS	5
K	-	MTMOS	5
L	-	AEAPT MOS	5
M	-	APT MOS	5
N	-	MTMOS:AEAPT MOS	2,5:2,5

The mycological examination.

The samples were exposed to the action of the wood decaying fungus *Coniophora puteana* in accordance with the modified test-method based on the EN 113 standard. Mass loss following the exposure were measured after eight weeks. It was assumed that $\leq 3\%$ of wood mass loss results from the effective dose of tested formulation. The activity of *C. puteana* was estimated on very high level, e.i. over 25%. All the examined systems were subjected to aging tests in accordance with the modified EN 84 standard and then mycological tests were performed according to the above-mentioned procedure.

Chemical analysis of ergosterol (ERG)

The samples containing 100 mg of ground wood were suspended in 2 ml methanol. They were treated with 0.5 ml of 2 M aqueous sodium hydroxide and sealed tightly. Samples were irradiated twice in a microwave oven and then were neutralized with 1 M aqueous hydrochloric acid. After irradiation in the microwave systems the cooled samples were subjected to pentane extraction. The ergosterol separation on a 3.9 x 150 mm Nova Pak C-18, 4 mm column with methanol:acetonitrile (90:10, v/v) as a mobile phase at a flow rate of 1.0 ml min⁻¹ was performed. ERG was detected with a Waters 2996 Photodiode Array Detector (Waters Division of Millipore, Milford, MA, USA) set at 282 nm. The presence of ergosterol was confirmed by a comparison of time of retention with the external standard and by co-injection of every tenth sample with an ERG standard.

RESULTS AND DISCUSSION

There were 13 solvent-base preservatives tested against *C. puteana*. Two of them: A and F contained IPBC of the concentration 0,05 and 0,5% respectively. Four formulation (K, L, M, N) contained silanes at the total concentration 5%. Seven formulations (B, C, D, E, G, H, I) contained both components e.i. IPBC and organosilicones. According to the assumption it was found that 0,05% of IPBC is an effective concentration against *C. puteana* only when is combined with AEAPTMOS or APTMOS. However these two formulations were inefficient after leaching. IPBC at the concentration of 0,05% appeared to be ineffective regardless to the aging effect.

IPBC at the concentration 0,5% turned out to be very effective (F, G, H, I). The mass loss of wood samples after *C. puteana* exposure were in the range 0,3-2,0%. The most promising results were found in case of the formulation containing MTMOS (G). The wood mass loss of leached samples after exposure to *C. puteana* were relatively low (0,8%).

Table 2. Moisture content and mass loss of wood treated with IPBC and organosilicon compounds and exposed to the action of *C. puteana*.

Code symbol	Wood Moisture Content after test [%]				Mass Loss after test [%]			
	Treated Samples	RSD	Control Samples	RSD	Treated Samples	RSD	Control Samples	RSD
A	60,5	5,4	86,8	17,7	21,2	4,5	31,9	6,0
AL	56,1	9,9	91,8	8,2	23,4	2,3	34,2	4,7
B	48,1	4,0	95,9	5,3	12,8	2,9	36,9	5,5
BL	48,8	9,2	69,1	17,7	12,7	6,9	32,2	7,8
C	34,9	5,8	82,8	5,3	0,9	0,3	37,1	3,6
CL	62,3	5,4	66,4	12,0	27,0	6,9	32,5	6,0
D	50,2	14,2	81,3	3,6	0,3	0,3	37,8	2,0
DL	65,6	2,8	69,8	13,4	30,6	5,4	32,6	5,4
E	60,4	5,0	81,8	6,0	25,8	3,2	30,1	2,3
EL	54,8	10,6	73,5	7,3	25,5	4,0	34,1	2,2
F	38,8	7,4	62,6	9,8	1,0	1,6	31,0	2,9
FL	46,2	7,1	74,0	5,8	20,8	7,0	32,6	2,9
G	37,5	4,4	69,3	1,9	0,3	0,0	32,0	2,1
GL	39,0	13,3	82,9	10,0	0,8	0,3	32,0	2,4
H	50,4	15,3	72,0	11,5	0,9	0,3	37,1	7,9
HL	58,7	6,1	85,7	6,5	27,0	2,6	34,0	2,9
I	47,6	8,2	59,3	25,7	0,6	0,2	29,3	7,1
IL	40,7	2,9	28,4	0,6	8,8	2,4	32,7	2,0
K	79,0	7,7	81,3	4,8	36,0	3,4	35,5	2,1
KL	83,1	7,6	81,7	4,3	31,9	2,3	31,7	3,4
L	47,2	9,1	96,0	3,6	2,0	0,4	39,5	6,9
LL	72,9	4,7	93,6	6,1	35,1	3,5	36,4	5,6
M	64,9	4,6	83,6	9,9	4,7	4,4	42,1	4,8
ML	48,5	14,5	64,0	19,0	15,7	6,4	34,7	3,2
N	49,5	11,9	61,5	19,7	22,3	2,1	37,7	3,7
NL	61,6	9,8	51,7	17,0	26,1	4,3	25,4	6,7

Except the standard method of preservative effectiveness evaluation (e.i. ML) the ergosterol identification of fungal mycelium in wood samples (e.i. ERG) was performed. The results of ML were confirmed by the ERG analysis. In most cases ERG concentration increases along with the ML increase. The observed correlation is not evident in case of other preservatives, e.g. ERG concentration in wood treated with IPBC after leaching was 187 $\mu\text{g}/\text{ml}$ and for the control wood was 130 $\mu\text{g}/\text{ml}$. The value of wood mass loss was 20,8% and 34,0% respectively.

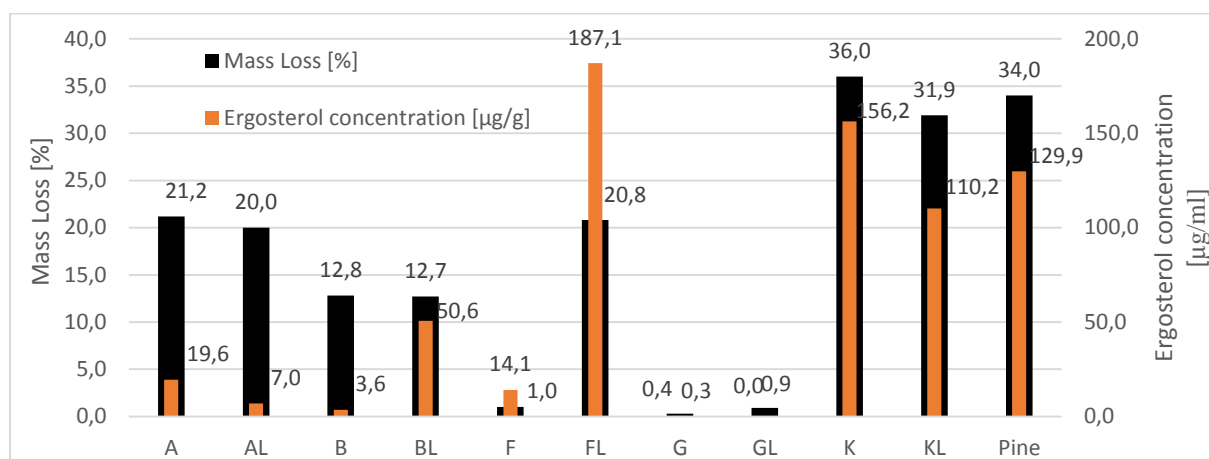


Figure 1. Relation of wood mass loss and ergosterol concentration as a result of *C. puteana* activity to wood treated with IPBC and MTMOS.

CONCLUSIONS

Wood preservatives consisting of IPBC (0,05% or 0,5%) and AEAPT MOS or APTEOS exhibit a protective activity against *C. puteana*. However, this type of formulation did not show any effectiveness when wood was subjected to leaching. Wood preservative consisting of 0,5% IPBC and 5% MTMOS showed very promising results. Low mass loss values of leached samples after *C. puteana* exposure, where proved by the ergosterol identification in wood samples.

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