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The performance of supercritical impregnated wood

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1 Background and purpose

The present report outlines the supercritical wood treatment process and the performance of supercritical treated wood. More than 10 years have passed since the supercritical wood impregnation process was commercialized in 2002 in Hampen, Denmark. The purpose of the present report is to review the most important performance tests and documentation involving supercritical impregnated wood conducted since 2002 and to present the results in a single report.

2 Assignor

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3 Supercritical wood impregnation

The supercritical wood impregnation process is different from traditional pressure impregnation processes because it uses supercritical CO₂ as carrier solvent instead of liquid solvents. Supercritical CO₂ is essentially a heavy gas that exists beyond the critical conditions for carbon dioxide (Figure 1).

Supercritical carbon dioxide has a combination of liquid and gas like properties that makes it an attractive solvent for wood impregnation purposes – both from a technical and environmental point of view. In an acknowledgement of the potential environmental benefits, Superwood was awarded the European Awards for the Environment in 2002. Like liquids, supercritical CO₂ has a high density allowing for dissolution of biocides. However, supercritical CO₂ has no surface tension and the viscosity is closer to that of gasses. Consequently, supercritical CO₂ penetrates wood easier than liquid solvents allowing for the impregnation of refractory wood species like spruce, which are difficult to impregnate using liquid solvents.

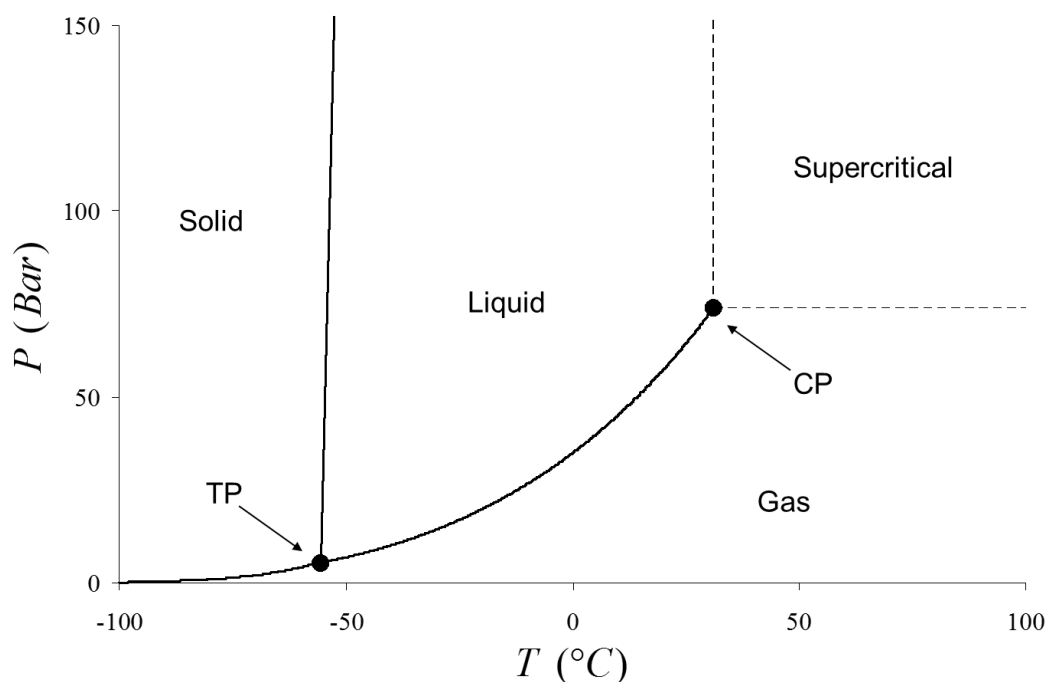


Figure 1. Phase diagram of CO₂. CP: critical point. TP: triple point.

Another feature, which separates the supercritical impregnation process from conventional liquid impregnation techniques, is that the process is a dry impregnation process. Being a gas, supercritical CO₂ does not wet the wood and the wood is dry before, during, and after impregnation. Because the wood is dry after impregnation, quality control of chemical uptake cannot be checked by simply weighing the wood, but has to be carried out by chemical analysis. Supercritical CO₂ acts as a non-polar solvent and does not allow for impregnation of wood with metal ions such as copper or boron. The choice of available actives for supercritical wood impregnation is therefore limited to molecules that can be solubilized in supercritical CO₂, typically organic molecules.

When Superwood (then Supertræ) started impregnating wood with supercritical CO₂ in 2002, the wood preservative used was called SC100, which contained a mixture of the fungicides tebuconazole, propiconazole, and IPBC in ratio 3:1:1. Soon after, the company shifted to the wood preservative SC200 which contains the same fungicides but in a slightly different ratio of 2:2:1. SC200 is used for commercial impregnation at Superwood today. However, some of the tests presented in this report refer to wood impregnated with SC100.

4 Performance testing

The performance of a preservative treated product is the result of its service life, maintenance requirement (wood oil, surface coating etc.), mould growth, resistance to UV-light, impact on the environment etc. Service life prediction is difficult because it depends on a number of different factors such as wood species, quality of the wood, exposure scenario, etc. In the European standardization system a road map is outlined. In order to be able to define appropriate preservative requirements the European Standard EN 335-1 (EN335-1 2006) graduates structural timber into five hazard classes.

The hazard class defines, by its environment and corresponding risk of decay, the minimum laboratory testing of appropriate wood preservatives and optional field test (Table 1).

Table 1. Hazard classes defined by EN335-1, 2006.

Hazard Class	Service	Laboratory test	Field test
1	Above ground Dry	None	None
2	Above ground Risk of wetting, protected	Brown rot	None
3	Above ground Exposed to wetting, non-protected	Brown rot	Stake test – 5 years or rot factor > 3 (optional)
4	Contact with ground or fresh water	Brown, white, and soft rot	Stake test – 5 years (optional)
5	Marine environment	Brown, white, and soft rot. Marine borers	Marine test – 5 years (optional)

The overall requirements for efficacy are described in the European Standard EN 599-1 with references to individual test standards (EN 599-1 2009). The standard is mainly focused on laboratory testing (EN 113), where the principle is to determine the toxic values of the actives against biological attack (brown rot, white rot and soft rot) depending on the hazard class. Furthermore, additional field tests can be carried out e.g. EN 330, L-joint (Table 2).

Table 2. Performance criteria in biological tests for hazard class 3, (penetrating processes).

Requirement	Min. requirement for fungal/field tests			Additional/local tests		
	With or without coatings	Only under coatings				
	Basidiomycetes	Basidiomycetes	Field test	Field test	V	B
Test	EN 113 Without (V)	EN 113 Without (V)	EN 330	EN 330	EN 113	EN 152-2
Ageing	EN 73, EN 84 (Separately)	EN 73	No additional ageing according to laboratory method	No additional ageing according to laboratory method	EN 73, EN 84 (Separately)	EN 152-2
(V) <i>Coriolus versicolor</i>						
(B) Blue stain						

Superwood has been tested according to EN 599-1 for Hazard class 3. This involves laboratory testing to determine the toxic value against brown rot fungi according to EN 113, also after accelerated ageing by evaporation (EN 73) and leaching (EN 84). Additional laboratory tests have been performed documenting the performance against bluestain (EN 152-2). Furthermore, additional field tests (L-joint) have been carried out in order to document its performance in field exposure scenarios. The extent of biocide leaching has been determined through a semi-field test study.

In the field tests, the performance of Superwood is compared with the well-known products GORI press 10 (water-based product for vacuum-pressure treatment) and GORI TH 92 (solvent-based product for double-vacuum treatment) as well as other products. GORI Press 10 and GORI TH 92 contain the same actives as SC200 (the preservative used in impregnation of Superwood) namely propiconazole, tebuconazole and IPBC. However, the ratio between the actives is slightly different for the different products. For GORI Press 10 and GORI TH 92

the ratio between tebuconazole, propiconazole, IPBC is 3:1:1 while the ratio for SC200 is 2:2:1. The present section presents an evaluation of the performance of supercritical treated wood in comparison with the well-known GORI products and other products with similar background of references.

Full test reports, summarized in the references, are on file at Superwood.

4.1 Durability

The durability of Superwood against attack from fungi has been investigated according to laboratory (EN 113 + EN 73 and EN 84) and field tests (EN 330, L-joint). In addition, the performance against blue stain fungi has been tested according to EN152-2.

4.1.1 EN 113 (Laboratory determination of toxic value)

The purpose of the EN 113 (EN 113 1997) is to estimate the toxic value of a given wood preservative against wood destroying fungi without considering the application method. The dimensions of the test sample ensure that in theory no matter the application method applied, a full cell treatment is achieved with evenly distributed actives in the wood structure. In practice, however, this is not always the case. This is why the samples are kept in a wet state for 2-3 weeks and regularly rotated to achieve an even distribution of actives.

The toxic values against wood-destroying fungi were determined for SC200 using the EN 113 as a stand-alone test, and by using the EN 113 after accelerated ageing according to EN 73 (evaporation) and EN 84 (leaching). The test is a requirement in the EN 599 to have a preservative approved according to the Biocidal Product Regulation of the EU. The tests were performed by MPA in Eberswalde (MPA 2002a-c). GORI TH 92 and GORI Press 10 have previously been tested at RUG in Gent.

Principle of the test

EN 113

Test samples of Scots pine sapwood are impregnated with a range of concentrations of the test preservative test. The method aims at determining the toxic values of the test product against wood-destroying fungi cultured on an agar medium. Other parameters affecting the fungicidal effectiveness of the material such as the method of application, the properties of the timber to be treated and the stability of the chemicals are out of the scope of this test. The toxic values are defined as the value where the weight loss is less than 3%, while the untreated specimens are degraded with a mass loss higher than 20%. The mass loss is adjusted by the mass loss of untreated, sterilized specimens following the same ageing and leaching procedures. The test is often carried out on samples which have been subjected to accelerated ageing procedures according to EN 73 (EN 73 1994) and EN 84 (EN 84 1997).

EN 73

The test samples are placed in a wind tunnel at 40°C for 12 weeks prior to the EN 113 test.

EN 84

The test samples are subjected to a leaching procedure consisting of an initial vacuum impregnation with water followed by submersion in water for two weeks during which the water is changed 9 times. The test samples are then subjected to the EN 113 test.

Results

An overview of the results for SC200 compared with GORI TH 92 and GORI press 10 is presented in Table 3. SC200 was tested against three brown rot fungi, *Coniophora puteana*, *Poria placenta*, and *Gloeophyllum trabeum*. Based on the EN 113 test, MPA Eberswalde concluded that the toxic value for SC200 is 60 g/m³ when tested according to EN 113 alone, and 120 g/m³ when tested according to EN 113 + EN 73 and EN 113 + EN 84.

Table 3. Results of the EN113 test for SC200.

	<i>Coniophora puteana</i>			<i>Poria placenta</i>			<i>Gloeophyllum trabeum</i>		
	EN 113	EN 113/73	EN 113/84	EN 113	EN 113/73	EN 113/84	EN 113	EN 113/73	EN 113/84
SC200	60	120	120	60	120	60	60	60	120
Press 10	123	100	120	122	100	124	119	120	151
TH 92	246	155	264	314	153	290	262	156	262

4.1.2 EN 330 (Field test, L-joint)

Field tests of SC200 according to EN 330 (EN 330 1994) "Wood preservative - Field test method for determining the relative protective effectiveness of a wood preservative for use under a coating and exposed out-of-ground contact: L-joint method" were run under tropical conditions in Malaysia from 2004 to 2009 (Figure 2). The product was tested on supercritical impregnated spruce (Superwood) at a concentration of 160 g/m³ and on supercritical impregnated pine sapwood at 120 g/m³, 160 g/m³, and 250 g/m³ and compared with GORI TH92, GORI 356 (internal standards), TBTO (standard reference) and heat treated wood, cf. Table 9. The EN 330 is considered an additional test according to EN 599-1 and is especially suitable for surface coated preservative treated wood. The field tests were set up at UNIMAS (University of Malaysia, Sarawak). The Danish Technological Institute examines the acceleration factor when comparing degradation in tropical Malaysia with degradation in temperate Denmark. From these studies an accelerated degradation by a factor of up to 5 is expected.

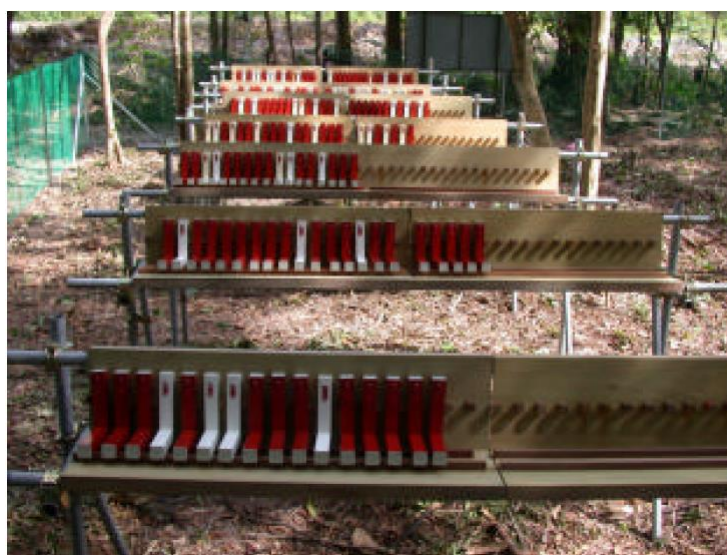


Figure 2. L-joints exposed in Malaysia. Photo: DTI 2005.

Principle of the L-joint test

Jointed samples (L-joints) are treated, assembled, and coated. After the coating is completely dry, the L-joints are taken apart so that the coating is broken across the L-joint itself. The L-joints are then reassembled and placed outdoors, out of ground contact and exposed to normal environmental factors, which affect coated wood exposed in practice. The broken coating at the joint creates a water trap, which accelerates fungal colonization and the natural breakdown of the sample. The fungi that colonize such units invade in their natural sequence of moulds, blue stain fungi, soft rot fungi and *Basidiomycetes*. Colonization by *Basidiomycetes*, as shown by the presence of visible decay is assessed at least annually by visual inspection of the L-joints after being disassembled. In addition, periodically, sets of samples are examined after sawing to reveal their internal condition. These data are compared with those generated using a reference preservative and untreated samples to assess relative performance. The rating system used for evaluation is given in table 4.

Table 4. Rating system used for evaluation of decay in L-joints.

Rating	Description	Definition
0	Sound	No evidence of deterioration
1	Slight attack	Slight discoloration, often dark and in streaks; no significant softening or weakening of the wood
2	Moderate attack	Distinct discoloration, but in discrete patches and streaks, with small areas of decay (softened, weakened wood); typically no more than 25% of the visible area affected
3	Severe attack	Marked softening and weakening of the wood typical of fungal decay and in extensive patches or streaks; distinctly more than 25% of the visible area affected
4	Failure	Very severe and extensive rot; Tenon often capable of being easily broken

Results

The final evaluation of the L-joint test was done in 2009 (DTI 2010). The results are given in table 5. The conclusion of the test was that SC200 impregnated spruce, with a rating of 1.4, performed equally as well as the internal standards GORI TH 92 (1.2) and GORI 356 (1.3). The standard reference, 2% TBTO, was severely attacked (3.6) and the untreated pine sapwood samples had completely failed (4.0). Untreated pine heartwood (1.7) performed quite well and only a slight improvement was seen by impregnation of heartwood with SC200 (1.4). Heat treated pine sapwood performed with varying results approaching severe attack as mean (2.7). SC200 treated pine sapwood also performed with varying results (2.2-3.4) and the treatments were moderately to severely attacked. No clear dose response could be seen from 0.12-0.3 kg/m³.

Table 5. Results from the L-joint test after 5 years exposure in Malaysia.

Treatment	Retention (kg/m ³)		Malaysia 5 years Rating
	Tenons	Mortises	
SC200 spruce	0.16	0.16	1.4
SC200 pine sap	0.12	0.12	2.8
SC200 pine sap	0.16	0.16	2.2
SC200 pine sap	0.3	0.3	3.4
SC200 pine heart	0.16	0.16	1.4
GORI TH92 pine sap	30.7 product	30.3 product	1.2
GORI 356 pine sap	183 g/m ²	183 g/m ²	1.3
Heat treated pine	-	-	2.7
TBTO pine sap	25.7 product	27.0 product	3.6
Untreated pine sap	-	-	4.0
Untreated pine heart	-	-	1.7

4.1.3 Field test with the obsolete SC100 wood preservative

In 2002, a field test involving supercritical impregnated wood (SC100) was set up in Norway at the Norwegian Forest and Landscape Institute. The purpose of the field test was to document the performance of a number of different treated and non-treated woods against wood destroying fungi. The test was set up as ground proximity tests, where the treated wood pieces are placed directly on the ground which increases the rate of fungal attack. The results were presented at an International wood protection conference in 2013 (Schabacker et al. 2013). The below table show the test results from non-surface treated samples. Supercritical impregnated wood performed in the top end of the 17 treated and non-treated types of wood in the study (Table 6).

Table 6. Decay ratings for different types of treated and non-treated wood without coating in the sited field test.

Ranking No.	Top layer: Treatment	Decay rating	Ranking No.	Core layers: Treatment	Decay rating	Ranking No.	Bottom layer: Treatment	Decay rating
1	Gori SC 100	0.5	1	Gori SC 100	0.0	1	Royalimp clear	1.0
2	ACQ 1900	1.0	2	Styren	1.0	1	Royalimp pigm.	1.0
3	Styren	1.0	2	Royalimp pigm.	1.0	1	Gori SC 100	1.0
3	Tanalith E7	1.0	2	Tanalith E7	1.0	4	Wolmanit CX-8	1.5
3	Royalimp pigm.	1.0	2	Wolmanit CX-8	1.0	5	ACQ 1900	2.0
3	Larch heartwood	1.0	2	Royalimp clear	1.0	6	Styren	2.0
3	Thermal mod.	1.0	2	Tanalith M (color)	1.0	6	Tanalith E7	2.0
3	Tanalith M (color)	1.0	2	Gori Pres 10	1.0	6	Furfurylation	2.0
3	Gori Pres 10	1.0	9	Tanalith M	1.2	6	Tanalith M (color)	2.0
3	Scanimp	1.0	10	Scanimp	1.3	6	Scanimp	2.0
11	Wolmanit CX-8	1.5	11	Larch heartwood	1.3	6	Gori Pres 10	2.0
11	Royalimp clear	1.5	12	ACQ 1900	1.5	12	Larch heartwood	2.5
11	Tanalith M	1.5	13	Furfurylation	2.2	13	Tanalith M	3.0
14	Pine heartwood	2.5	14	Thermal mod.	2.8	13	Thermal mod.	3.0
15	Furfurylation	3.0	15	Pine heartwood	3.0	15	Pine heartwood	3.5
16	UltraWood	3.5	16	UltraWood	3.2	16	UltraWood	4.0
16	Pine Sapwood	3.5	17	Pine Sapwood	3.8	16	Pine Sapwood	4.0

4.2 Blue stain

4.2.1 EN 152-2

The resistance of supercritical impregnated wood against blue stain was tested according to the European standard EN 152-2 (EN 152-2 2006) "Laboratory method for determining the preventive effectiveness of a preservative treatment against blue stain in service Part 2: Application by methods other than brushing". EN 152 is considered an additional local test according to EN 599-1. However, in Denmark it is a requirement according to the association of Danish window manufacturers (*VinduesIndustrien*) if the product is used to protect windows produced under the control of DVV (Dansk Vindues Verifikation).

Principle of the Test

The basic principle of the test method is to provide the conditions for infection by blue-stain fungi and observe the development of infection. Treatments according to the specified methods are applied to "treatment sticks", from which the treated test panels are subsequently cut. The sticks are exposed to natural weathering for 6 months in the period between March and October. Afterwards, the specimens are exposed by a mixed culture of blue stain fungi in the laboratory. A comparison of the extent of blue staining of these test assemblies with untreated control shows the effectiveness of the test product. The specimens are examined for blue stain on the surface according to the following rating 0, 1, 2 and 3. The rating 0 is no occurrence of

blue stain, and the mark 3 is severe attack of blue stain. The specimens are cut and the blue stain free zone is determined. The standard contains no approval requirements. EN 599-1 requires a zone free of blue stain of minimum 1.0 mm and in average 1.5 mm and a maximum fouling degree of 1 (= sporadic fouling). *VinduesIndustrien* requires a maximum character of 1 on the surface and a zone free of blue stain of min. 1.5 mm with an average of 2 mm.

Results

The tested systems are presented in table 7 together with the results of the test (DTI 2005). System 1 had no blue stain on the surface. System 2 and 3 had a surface blue stain rating of 2 and a smallest depth of blue stain free zone of 5.0 mm, which was comparable to the reference with fungicide.

Table 7. Test results from the blue stain test (EN 152-2) involving supercritical impregnated wood.

System	Treatment	Top-coat	Blue stain rating on surface (average)	Smallest depth of blue stain-free zone, mm	Mean depth of blue stain-free zone, mm
System 1	0.16 kg/m ³ SC200	GORI 410 – 10210 + Gori 890 - 12210	0	4.5	5.0
System 2	0.16 kg/m ³ SC200	Standard Alkyd (GORI 90, Farblos 9900)	2	4.5	5.0
System 3	0.30 kg/m ³ SC200	Standard Alkyd (GORI 90, Farblos 9900)	2	5.0	5.0
Reference, top with fungicide	-	Standard Alkyd (GORI 90, Farblos 9900)	2	1.0	1.5
Reference with fungicide	Bondex Holzschutzgrund, Farblos	Standard Alkyd (GORI 90, Farblos 9900)	2	4.0	6.5
Reference without fungicide	-	50% linseed oil / 50% white spirit	3	0.0	0.0
Untreated control, with weathering	-	-	3	0.0	0.0
Untreated control, without weathering	-	-	3	0.0	0.0

4.3 Leaching

The Danish Technological Institute has conducted leaching tests on supercritical impregnated wood as part of a larger project together with manufacturers of active ingredients and formulators of wood preservatives aiming at finding realistic leaching rates from preservative-treated wood in hazard class 3 (above ground). The project focused on developing a field trial method to investigate leaching. The results from the project were used to serve as part of the basic documentation according to the BPD (Biocidal Products Directive). The study included commercially available organic and inorganic fungicides using four different application methods: vacuum-pressure-, double-vacuum-, flow coat and supercritical treatment. Different test set-ups examine the influence of a number of different parameters including sawn-cut or planed surfaces, orientation of the exposure etc. The method investigated has proved to be useful in characterising the leaching behaviour from preservative treated wood.

Principle of the Test

Panels were subjected to outdoor exposure under natural weather conditions at a test field at the Danish Technological Institute (Figure 3). The leachate was collected and monitored by chemical analysis for active ingredients content.



Figure 3. Test set-up for the semi-field test at DTI.

Results

Figure 4 and 5 show the results after approximately 12 months' exposure.

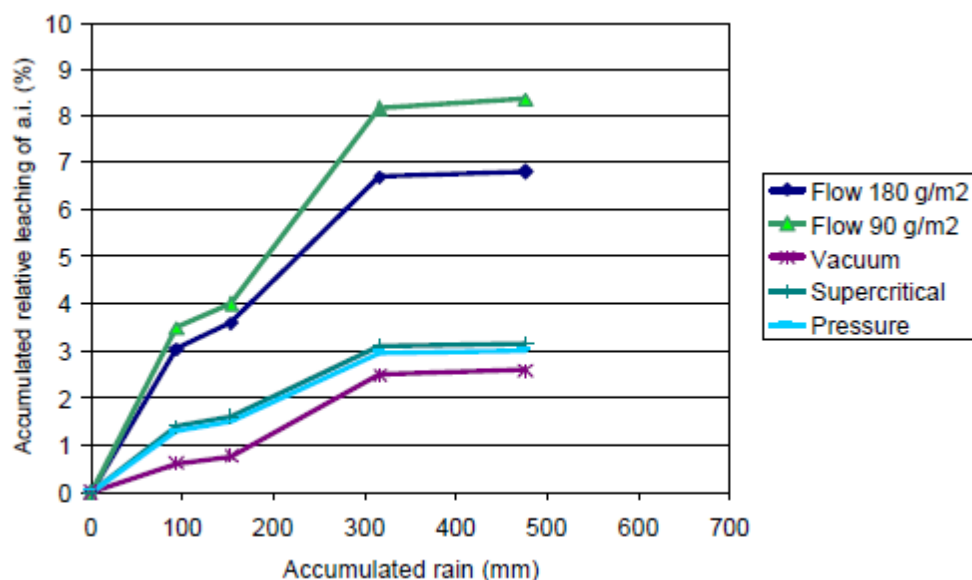


Figure 4. Leaching of active ingredient (propiconazole) in relation to retention of preservative (%). Preservative treatment is compared, i.e. depth and concentration of active ingredient. All panels are exposed vertically, facing South.

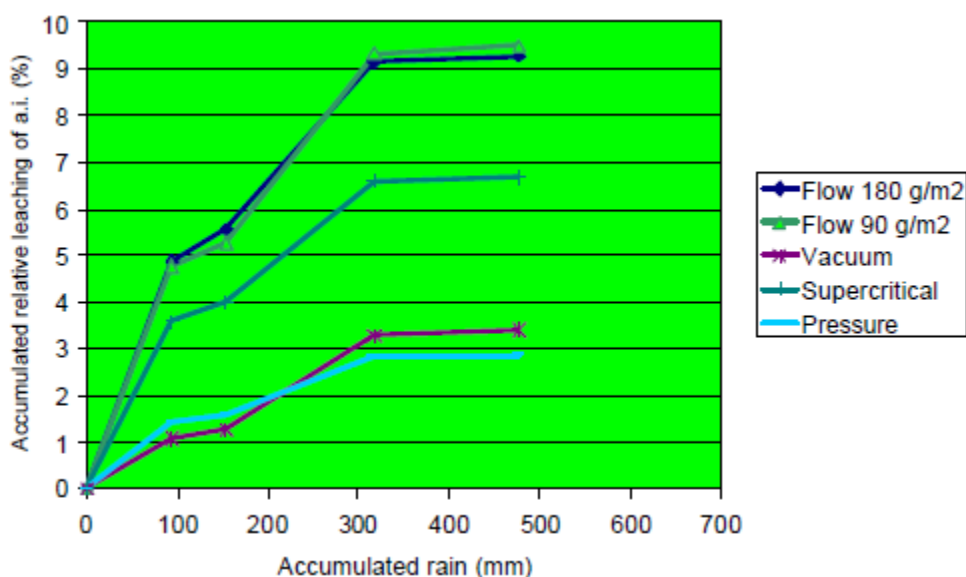


Figure 5. Leaching of active ingredient (Tebuconazole) in relation to retention of preservative (%). Preservative treatment is compared, i.e. depth and concentration of active ingredient. All panels are exposed vertically, facing South.

There is a significant difference between the different treatment methods. The full penetrating methods (supercritical and vacuum-pressure treatment) have, not surprisingly, the lowest relative leaching. The flow-coat treatment on the other hand has the highest relative leaching, which indicates that leaching to a high extent is a surface phenomenon. The first approx. 100 mm of rain represents more than half of the total leaching in the whole observation period. This seems to be the case for all the different methods of application. The last assessment period on the other hand represents less than 5% of the total leaching in the whole observation period. In case of the organic preservative investigated, the relative leaching of copper is at the same level as is the case for the organic based preservatives. The retention of the copper-based product used in this investigation is much higher, and consequently the cumulative leaching is higher.

It must be noted, that the described leaching test was performed with the obsolete product SC100 that contains the same fungicides as the currently used SC200 but in different ratios. In SC100, the ration between tebuconazole, propiconazole, and IPBC is 3:1:1, whereas the ratio between those fungicides in SC200 is 2:2:1. Furthermore, the concentration of fungicides in the tested wood was 341 g/m³ for the three fungicides combined, which is higher than the 120 g/m³ currently authorized for SC200. Because of the lower concentration of fungicides in SC200 impregnated wood, it is likely that the leaching from SC200 impregnated wood would be lower than what was observed for SC100.

4.4 Appearance

Wood exposed to weathering is subject to a gradual colour change towards greyish and darker colours. The greyish colour is the result of the combined effect of discoloration fungi and UV-light. In Denmark the wood is turning grey rather quickly depending on the exposure scenario (covered, not-covered, orientation etc.). As a side effect of the leaching study described above, the colour of SC100-impregnated wood was compared with non-treated wood. In figure 6 and figure 7 the appearance of SC100-treated spruce is compared with untreated pine after 2 years of exposure. The discoloration of SC100-treated wood is limited compared to

untreated wood. Even after 2 years' exposure and with no surface treatment only slight changes in the colour of the wood is noticed. It has not turned grey. The reason for this phenomenon must be that the fungicides restrict the formation of discoloration fungi on the surface. It should be noted that no corresponding test has been performed with SC200.



Figure 6. Supercritical treated spruce (SC100) after 2 years of natural exposure towards south.



Figure 7. Untreated Scotch pine after 2 years of natural exposure towards south.

4.5 Corrosion test of fasteners in Superwood

A corrosion test was performed on fasteners in Superwood (ITW 2008). The test was done by ITW Construction Products ApS as a 2000 hour salt spray test according to EN 9227 (Ref. 15). The fasteners tested were NKT Climate®-X and MULTI+. Climate®-X is a steel fastener with a Climate®-X surface; MULTI+ is a stainless steel fastener. The conclusion of the test was that Superwood was neutral (no influence) on the corrosion of both Climate®-X and MULTI+ fasteners.

5 Authorization, quality control, and working environment

5.1 BPD (BPR) authorization

In order to market a wood preservative in EU, the product must be authorized by the relevant environmental authorities according to the BPR, Biocidal Product Regulation (EU 2012) (formerly BPD, Biocidal Product Directive, EU 1998). To obtain authorization, documentation must be submitted related to the environmental impact of the product and its efficacy against wood destroying fungi. The European standard EN 599-1 lays down the requirements for efficacy.

SC200 has been authorized according to the BPD by the Danish Environmental Protection Agency with BPD registration number 674-1 (EPA 2012). According to the registration, the product is authorized for use as a wood preservative for use class 2 and 3, and can be used for impregnation against wood destroying fungi at a concentration of 120 g/m³, and against blue stain and wood destroying fungi at a concentration of 160 g/m³.

5.2 External quality control

Every 6 months the Danish Technological Institute (DTI) checks the concentration of fungicides in Superwood according to penetration class NP3 (EN 351-1 2007) and use class 3 (EN

335-1). The concentration is checked by chemical analysis of wood samples picked randomly from the Superwood production site by DTI. DTI has performed the quality control since 2006 and the control is still ongoing.

5.3 PEFC

Superwood is PEFC chain-of-custody certified with certificate No. NC-PEFC/COC-000043 (PEFC 2012). This means that all the wood Superwood impregnates originates from certified forests.

5.4 CE Mark

Superwood is CE marked according to EN14915.

5.5 Working environment

According to Danish legislation, the occupational exposure limit value for wood dust in the air is 1 mg/m³ (BEK 2011). Dansk Toksikologi Center (Danish Toxicology Center), examined whether inhalation of dust from SC200 impregnated wood would lead to an increased health risk compared to inhalation of dust from non-treated wood (DTC 2004). The report concluded that dust from SC200 impregnated wood did not present an increased health risk compared to dust from non-treated wood and, consequently, the occupational exposure limit value of 1 mg/m³ is sufficient to protect workers from risk.

5.6 Disposal

As a rule, Danish legislation does not permit incineration of impregnated wood (BEK 2012). Instead, impregnated wood must be permanently deposited at specific sites. The reason for the general rule is that impregnated wood frequently contains metals. However, since Superwood does not contain metals it is exempt from the general deposition rule. Consequently, Superwood can be disposed of by incineration in approved incineration plants.

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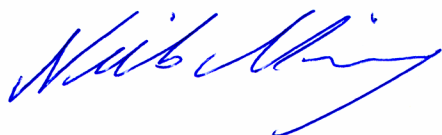
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