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PERFORMANCE OF IMPRALIT-KDS
NEW PRESERVATIVE, NEW CHEMISTRY

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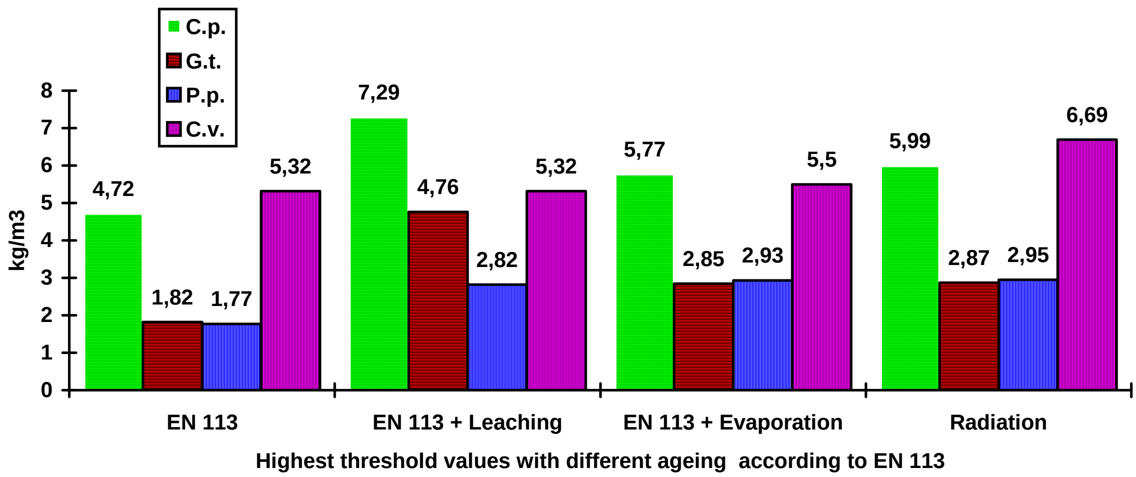
1. Introduction

During the 1980`s a lot of work was done in Europe to introduce new Chromium free water born products for wood preservation to address environmental concerns. Therefore especially in development of wood preservatives the consideration of environmental impact of new products has to be an important part of the strategy, due to lack of control over the environmental fate of preservatives if leached during service or from waste wood. For wood protection in ground contact all these products were based on an organic fungicide and copper. Suitable organic fungicides for wood preservatives are Triazole, HDO, and Quaternary Ammonium compounds etc.

Alkyl ammonium compounds (AAC) have good biological efficacy, acceptable environmental characteristics, and are cost effective. On the other hand, AAC's have some disadvantages regarding their use as wood preservatives due to surface spotting and non-uniform penetration. This was the starting point of our development with the objective to overcome the disadvantages of AAC´s without loss of efficacy and other positive properties.

When the details of the performance of quaternary ammonium compounds were analysed the excellent activity of the Polymer Betaine against copper tolerant fungi has been discovered.

Graph 1: Efficacy of Polymeicr Betaine according to EN-Standards



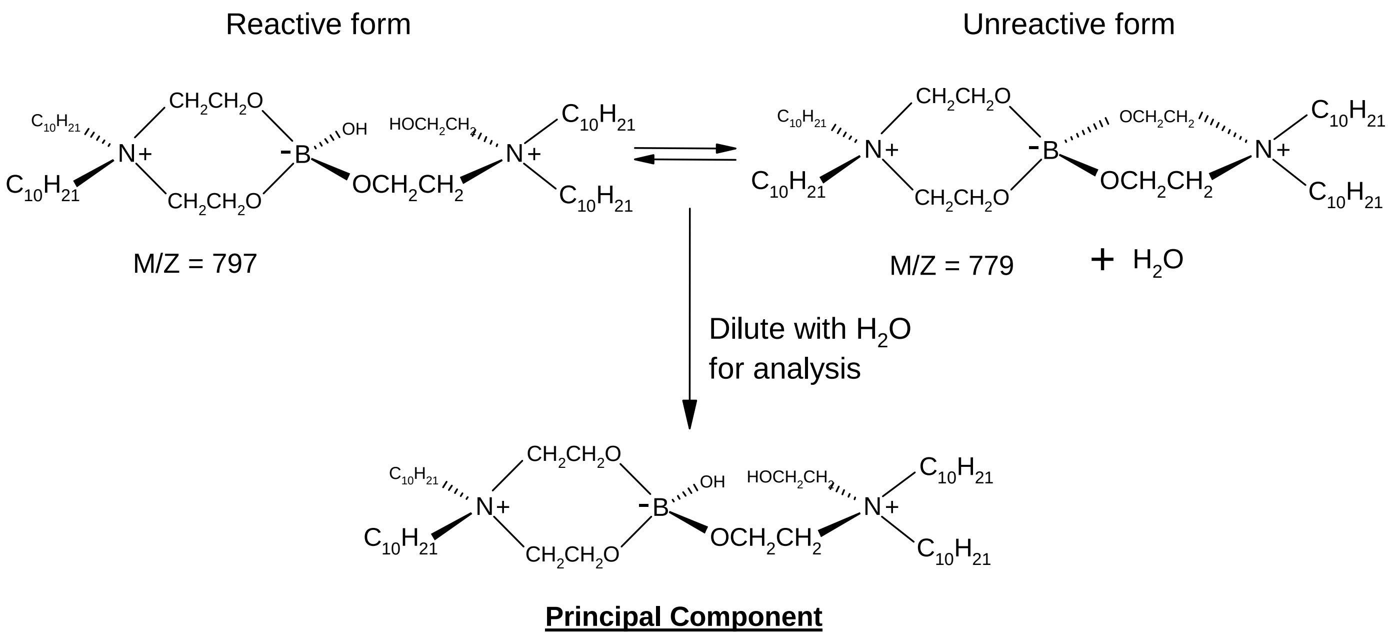
In combination with Copper/Amine a strong synergism between both active ingredients has been found and at the end of the development impralit KDS was born.
Impralit KDS is a water born heavy duty preservative for pressure treatment for in-ground and aboveground applications. The formulation based on Copper, Boric acid and Polymeric Betaine (DPAB).

Impralit KDS was commercialized in Europe in 1992, US EPA approval was obtained in 2006, the Canadian PMRA approval and ICC approval is in progress.

2. Polymeric Betaine - The New Chemistry

We found a new group of compounds, so-called Polymeric Betains, which contain cationic alkyl ammonium groups that can form borate esters. The amphoteric nature of Polymeric Betaine offers unique properties that are advantageous to regular alkyl ammonium compounds. These compounds are synthesized by reaction of Dialkylamines with ethylene oxide and boric acid. They are very stable under alkaline and strong acidic conditions. The active ingredient exists only in solution, as an equilibrium mixture of products.

Graph 2: Structure of Polymeric Betaine:



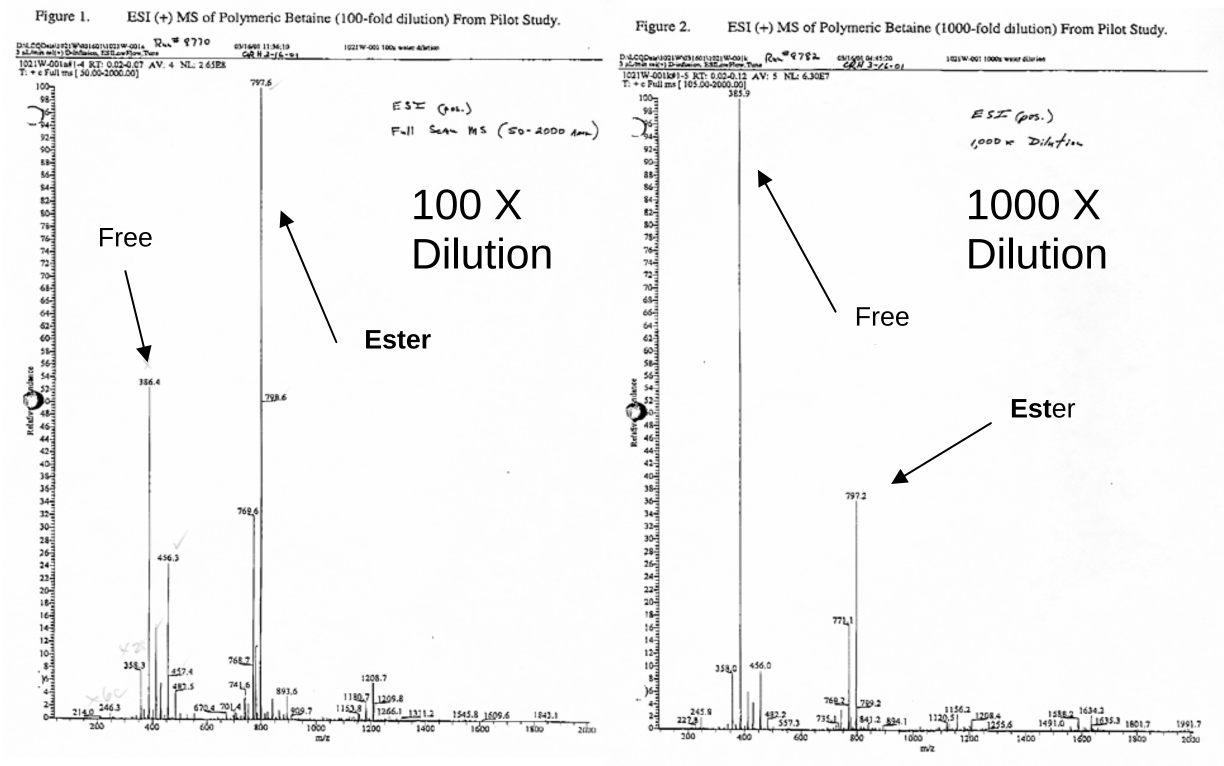
The Betaine structure acts as a carrier for quaternary ammonium salt resulting in excellent penetration properties and uniform dispersion. The cationic nature of the corresponding Dialkyl-dialkoxyammonium compound gives a good Fixation. The Didecyl-substituted compound shows excellent suitability as biocide for wood preservation use.

- Characterization of Polymeric Betaine

In order to analyze the Polymeric Betaine it is very important to take into consideration the fact that the molecules exist as equilibrium (see graphic above). The components cannot be separated from each other and must be analyzed by non-destructive methods like NMR or mass-spectrometry (MS) in electro spray mode (ESI). For quantitative analysis and environment studies isotope labeled molecules have been synthesized.

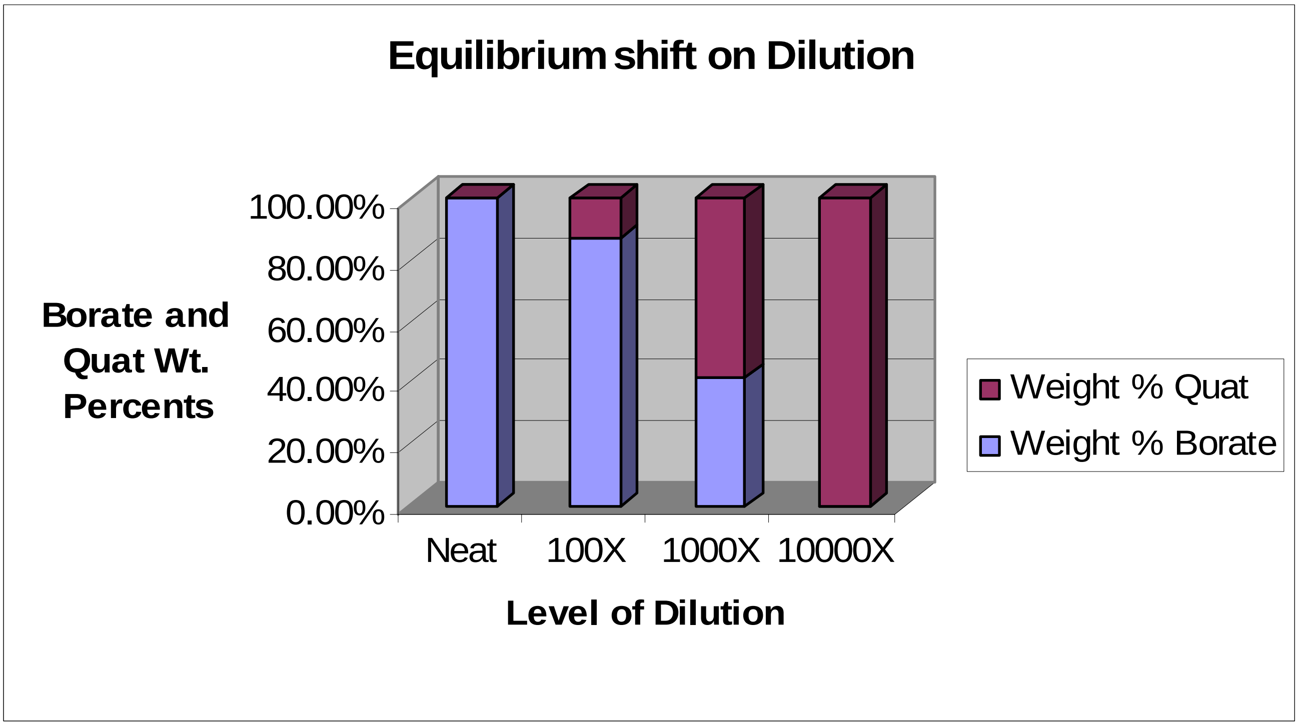
The mass spectrum shows clearly both the mass of the free quaternary ammonium compound and the mass of the borate ester with all variances caused by the different atomic masses of Boron.
By the mass spectrum it is also possible to assess the equilibrium shift on dilution with water.

Graph 3: Mass spectrum of Polymeric Betaine



Pure Polymeric Betaine consists nearly up to 100% of borate ester. Dilution by water shifts the equilibrium to free quaternary ammonium. In a 50% solution of Polymeric Betaine in water the amount of ester is about 99%. If the dilution is higher than 95% a greater content of free quaternary ammonium is detectable (about 6%). In a 0,1% solution the amount of borate ester and free Quats is nearly equal.

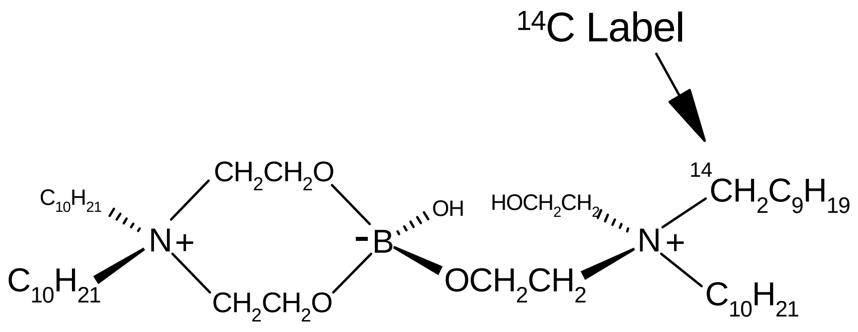
Graph 4: Equilibrium of Polymeric Betaine in solution



Binding of Polymeric Betain to Wood

The mechanism of binding of Polymeric Betaine was examined by using radio labeled material.

Graph 5:

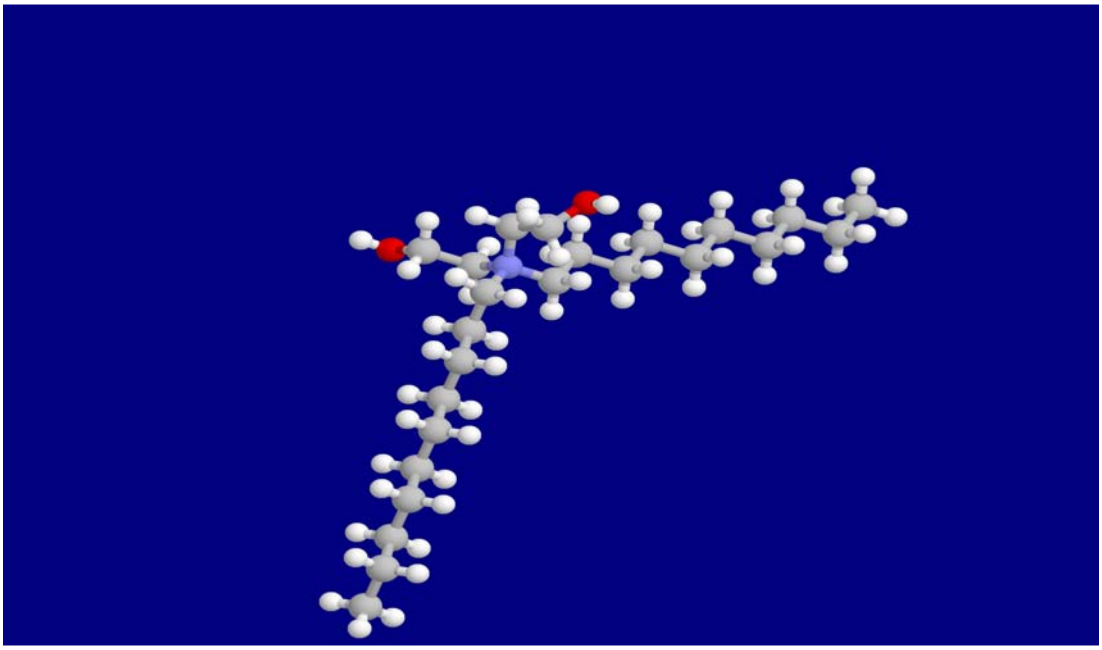


The borate ester can react with all reactive groups in wood like reactive groups in wood, for instance with:

-Pectin
-Hemicelluloses
-Lignin

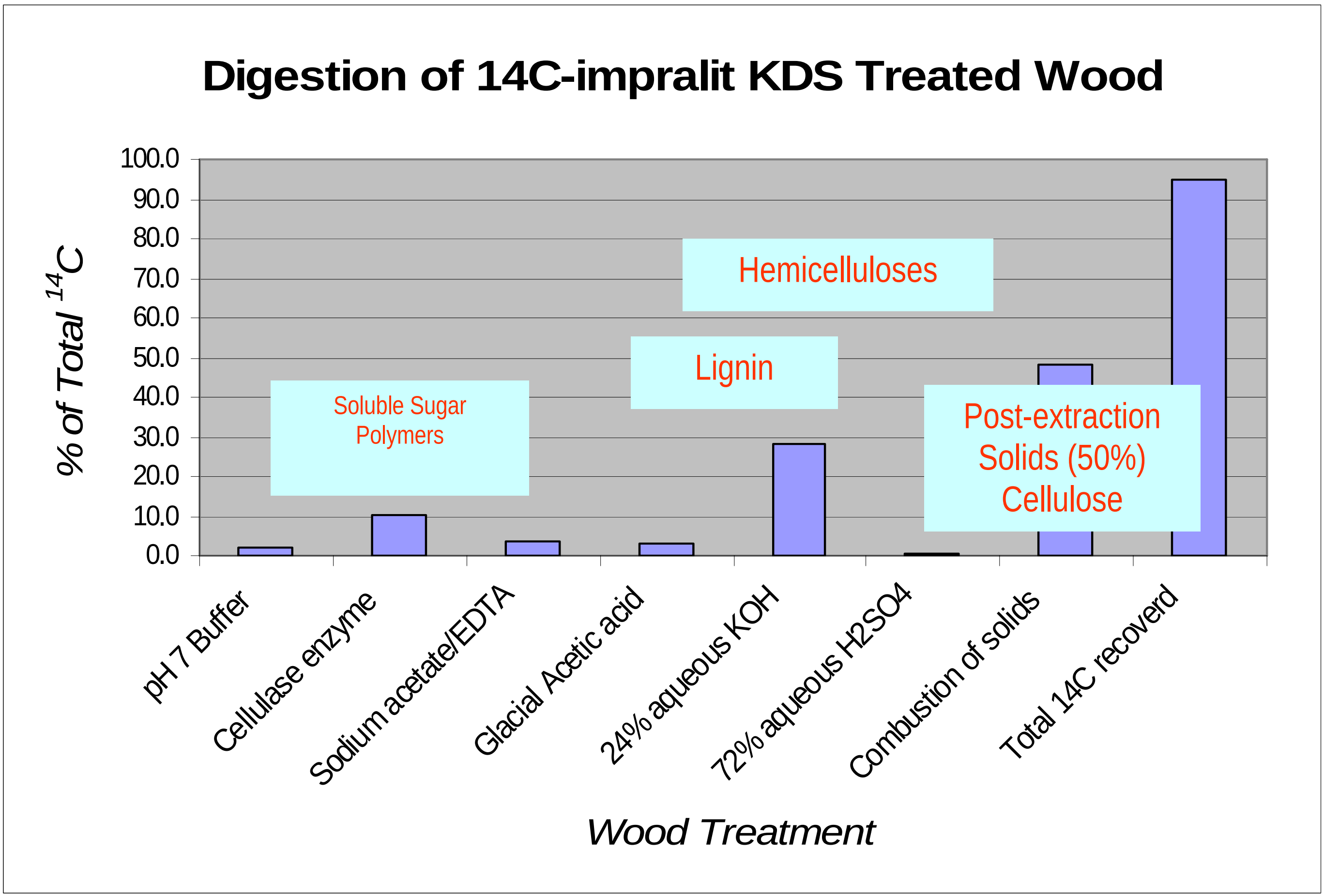
Especially it will react with its active groups containing Oxygen, so after the reaction the active cannot be easily removed from wood. Graph 6 shows clearly the possible binding positions at the end of the polar groups of Polymeric Betaine. The long Hydrocarbon-chains dosnt`t affect the polar groups.strongly.

Graph 6: Structure of Polymeric Betaine (open form):



This strong bounding to wood could be proved by digestions test with various leaching solutions. Only 1.8% of the radiolabled Betain could be removed by using a pH 7 buffer. Even after treatment with different strong complexing agents and strong acids and bases 48% of the Polymeric Betain stayed in the wood.

Graph 7:



Environmental Fate

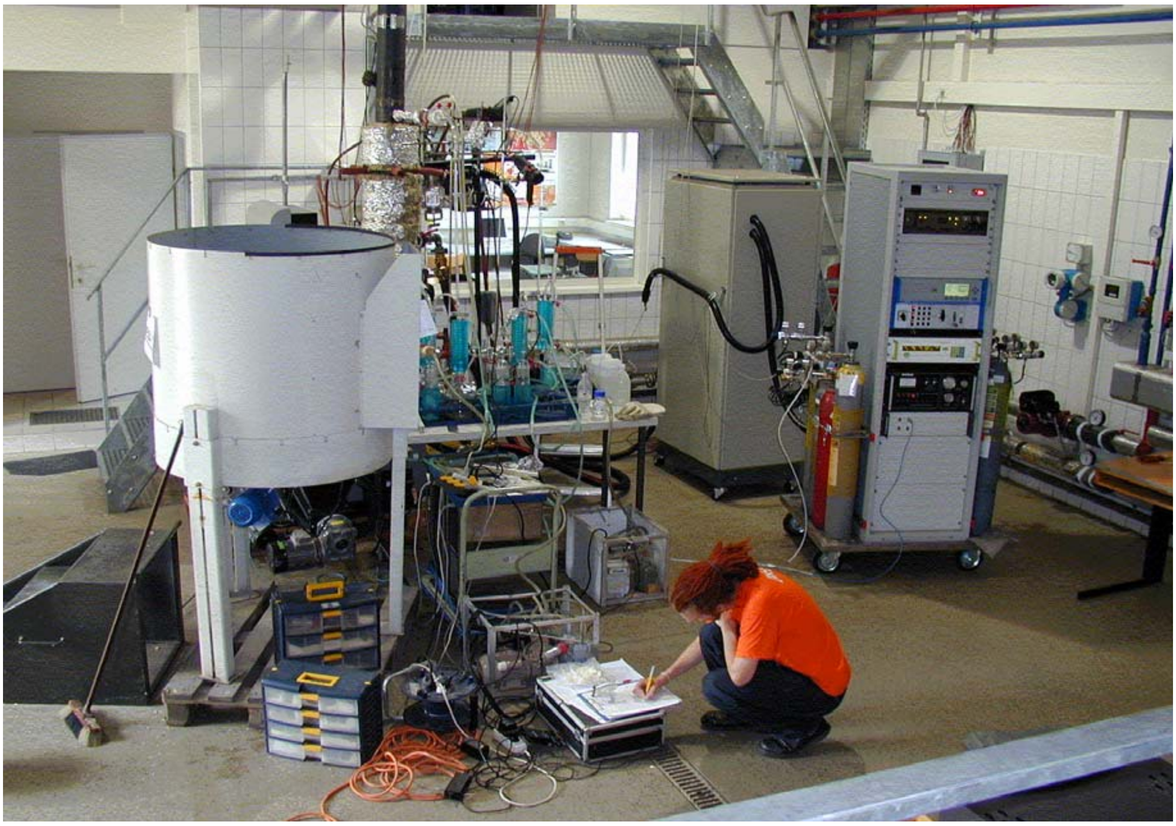
The environmental fate of wood preservatives is mostly determined by leaching behavior and the possibility of reuse and the waste management of the treated timber. In case of water born preservatives an environmental effect by evaporation can be neglected.

The behavior of Polymeric Betaine in the environment on the one hand is affected by the strong fixation properties to all organic and inorganic materials. The transfer of active material in water is therefore minimized. On the other hand due to the organic nature of Polymeric Betaine, it can degrade in soil. An accumulation in the environment is not possible.
Less than 0.1% of VOC was emitted from the biodegradation of Polymeric Betaine in an aerobic soil metabolism study. In loam soil, 34% of Polymeric Betaine was mineralized to CO_2 after 64 days. 20% remained as extractable residue and 39% was non-extractable residue. Similar results were obtained with other soil types.

Graph 8:

Soil type	Loam	Sandy Loam	Silty Clav
CO2	345	475	42%
Extractable Residue	20%	23.7%	13.7%
Non-Extractable Residue	39%	30%	46.3%
VOC	< 0.1%	< 0.1%	< 0.1%

Graph 9: Incineration test:



In incineration tests in a pilot plant untreated timber and timber treated with Polymeric Betain and Polymeric Betaine/Copper/Boron was burned and the flue gases were analyzed.

No significant influence by the impregnation of timber on the incineration behaviour and the flue-gas in the studies were found. Especially, no evidence was found that wood impregnated with Polymeric Betaine leads to generation of Dioxins and Furans.

Impralit KDS:

The new preservative system

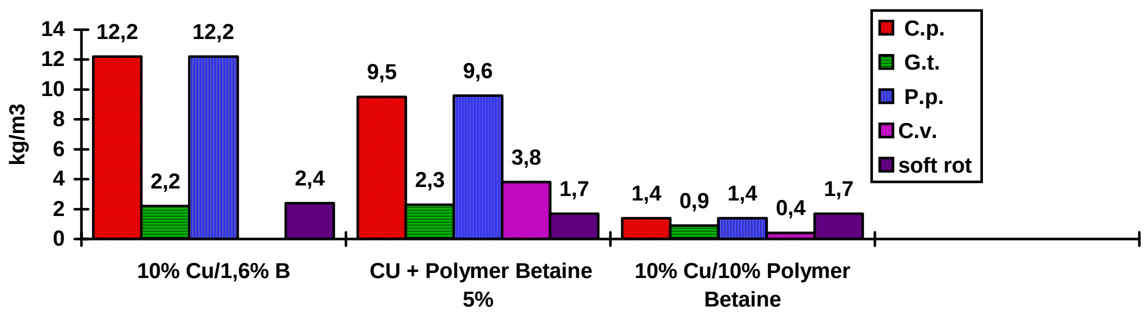
The wide range of biological activity makes the Polymeric-Betain suitable for use in different fields of wood preservation (see graph 1). For ground contact, copper is used in combination with Polymeric Betaine to prevent soft rot attack.

Efficacy against decay:

Lab-Tests:

Copper- or copper/boron-based chromium free formulations show lack of efficacy after leaching against fungi producing oxalic acid. Due to the fact that the Polymeric-Betain has its best efficacy against this kind of fungi (e.g. against poria placenta), we had expected good effectiveness against decay in combination with Copper or Copper/Boron. The threshold values after leaching are much better than calculated from the efficacy test of the single components. In Graph 9 it is quite obvious that the efficacy of Copper/Amine-formulation in the European standard test EN 113 with 10%Copper and 10% Polymeric Betaine has the best effectiveness against basidiomycetes as well as against soft rot.

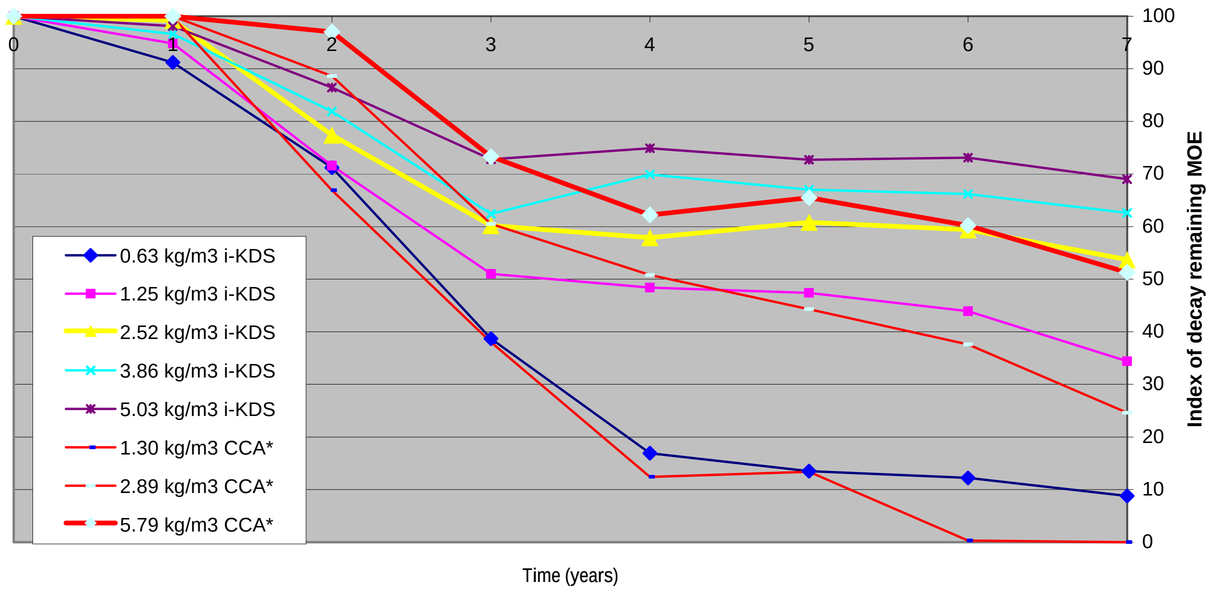
Graph 10: Comparision: Efficacy of Copper/Amine-formulations



Threshold values of Basidiomycetes according to EN 113 with and without leaching and threshold values of soft rot after leaching

In a fungus cellar test, the performance of the KDS preservative was monitored by measuring the modulus of elasticity (MOE) in comparison with CCA-treated stakes (stake 400 mm x 20 mm x 20 mm; CCA-salt according to EN 252). The MOE was chosen because one can see early and measure objectively the changes of the stakes under the test conditions. The stakes were checked annually. The results after seven years show that performance of impralit KDS impregnated stakes (retention rate 2,52 kg/m^3) is similar to the CCA-salt impregnated stakes (loading 5,79 kg/m^3).

Graph 11: impralit KDS: Fungus-Cellar-Test 7 years (BAM): MOE-rating



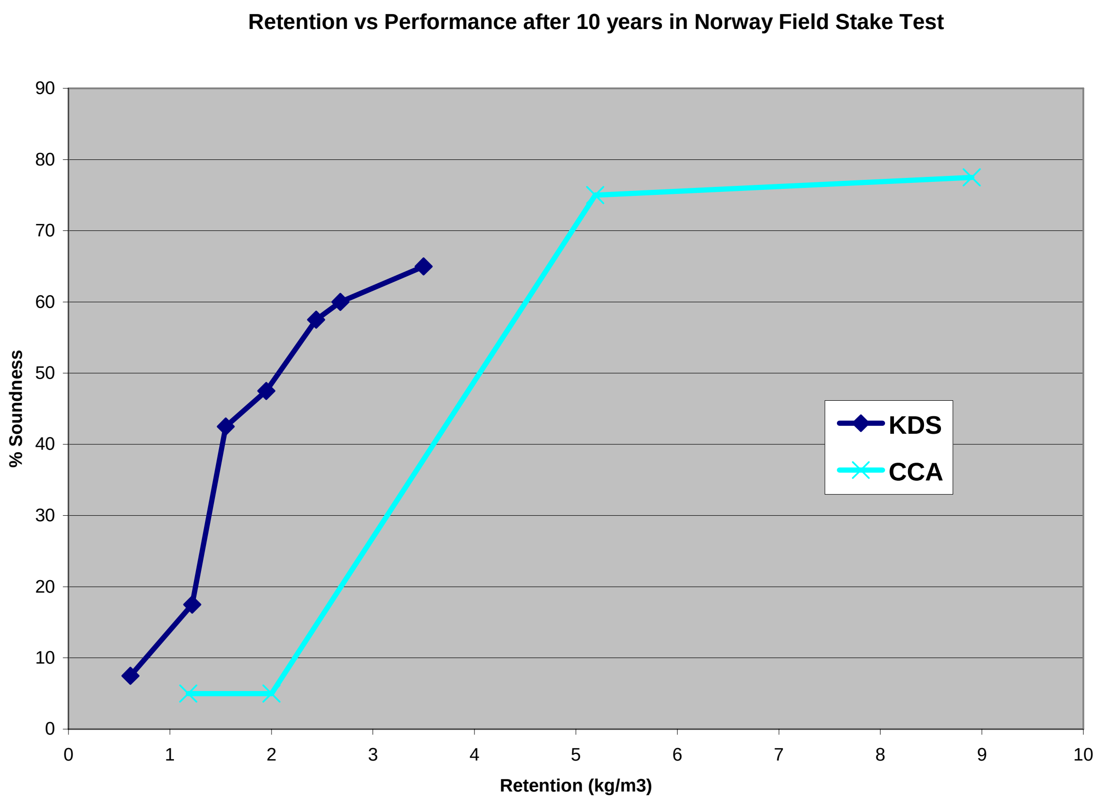
The figure shows the effectiveness of the fungus-cellar. Loadings of 1.3 kg/m^3 CCA-salt didn't prevent fungus attack after two year exposure. Higher loadings on the other hand demonstrate the excellent performance of the Copper/Amine/Polymer Betaine formulations in comparison with CCA-salts

Field Tests:

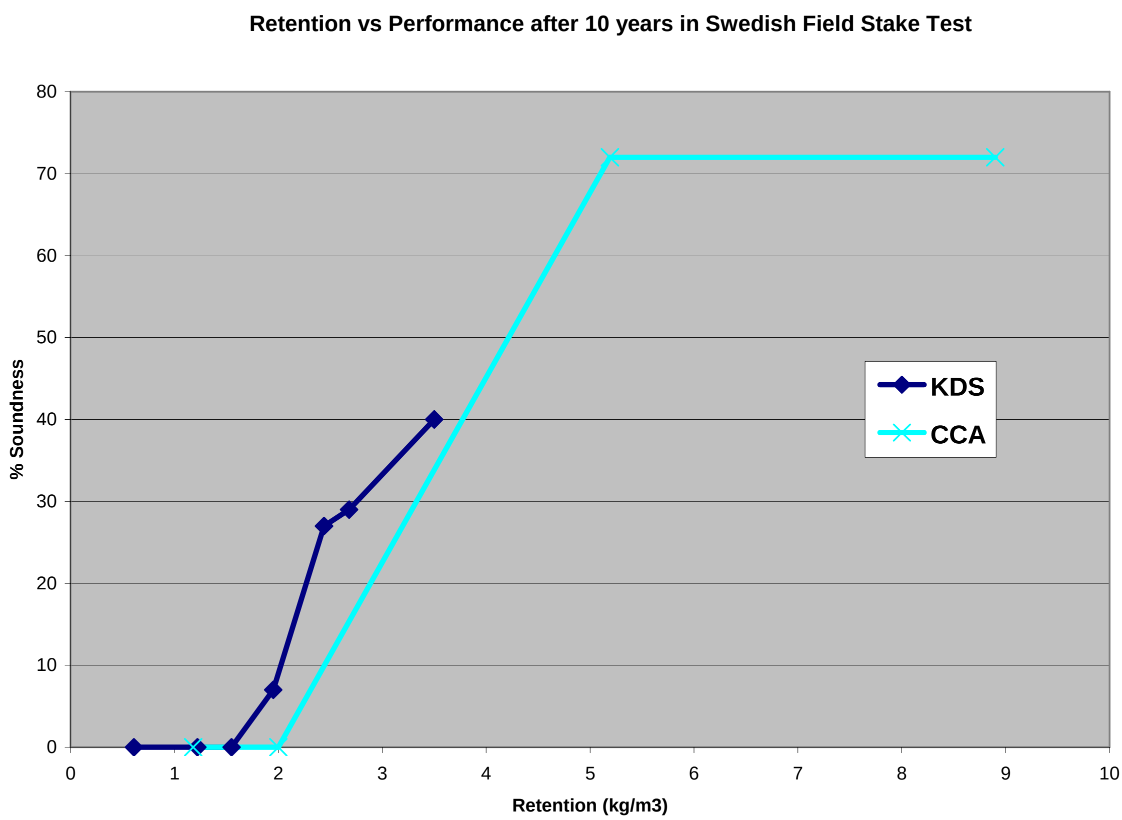
European test side

In order to test the performance of wood preservatives for impregnation of wood for ground contact usually a field test is conducted. Stakes tests according to EN 252 have been performed at two different places in Scandinavia. The tests have been started at the Swedish University of Agriculture Sciences with 30 stakes at each level of retention, 20 in Simlangsdalen (Sweden) and 10 in Sörkedalen (Norway).
Graph 11 shows the results of the Norwegian test side in comparison to CCA according to the European standard.

Graph 12

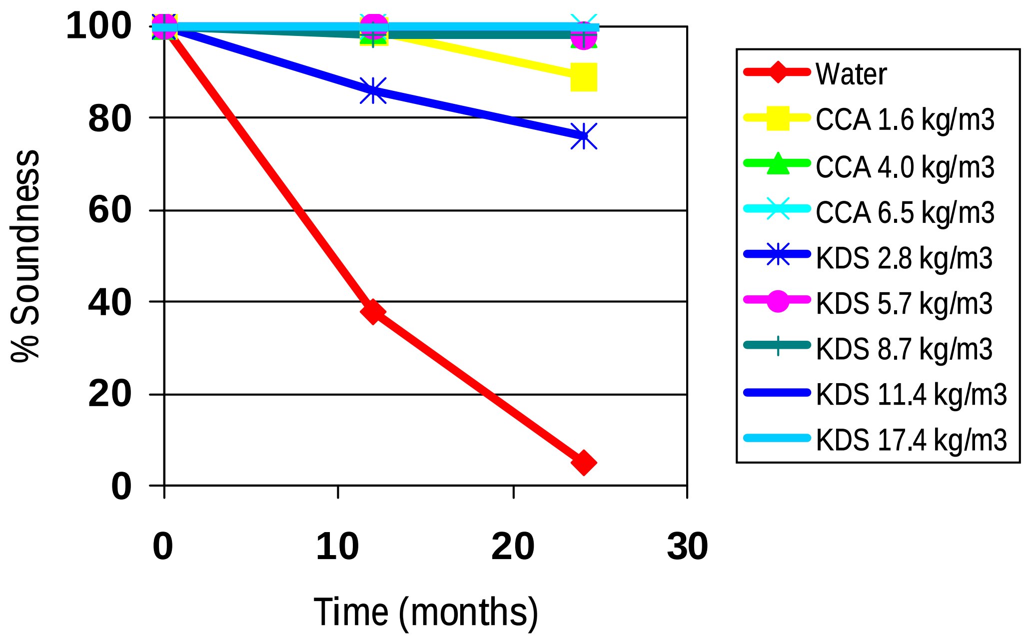


Graph 13



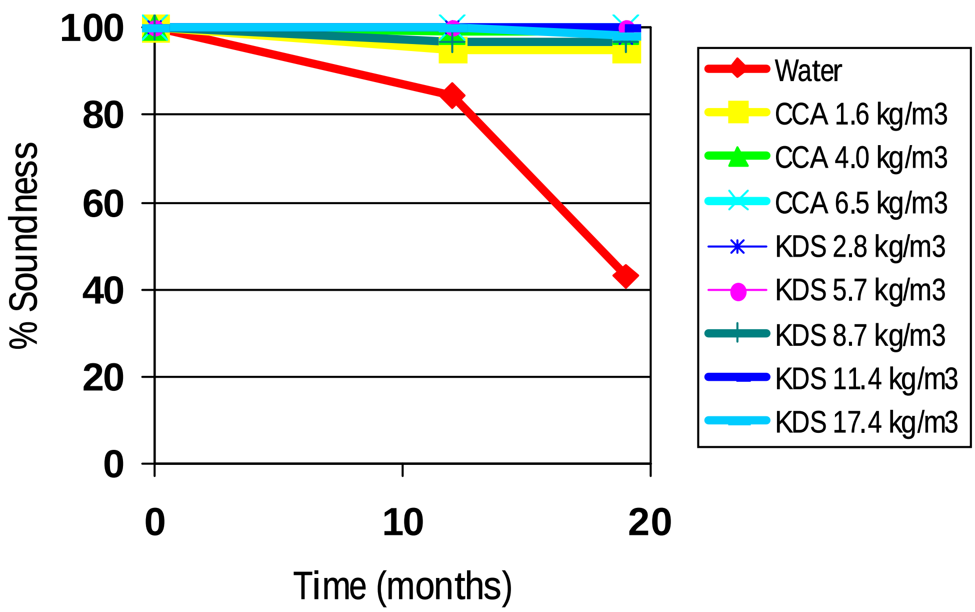
US Field Tests:

Hawaii and Mississippi are the two most aggressive test sites in the US. At both test sites tests according to AWPA-Standard E7-01 have been established.
The evaluation of ongoing field stakes test at Lake Dorman and Saucier test sides show the



good performance of impralit KDS after two years.

Graph 14: Field Stake Test - Dorman, MS



Graph 15: Field Stake Test - Saucier, MS

The great activity of the test fields is demonstrated by the severe attack of the untreated stakes after 1 year of exposure and the heavy decay of the treated stakes with low retentions. At higher retentions both the CCA stakes and stakes treated with impralit KDS performed well.

Lap-Joint Test –MS

Lap-joint test at Mississippi state university according to AWPA standard E16-98 showed no attack at all retention rates after 36 month of exposure while untreated lab-joints were heavily decayed.

Graph 16: Lap-Joint Test -MS

Treatment	Retention (kg/m3)	Decay Ratings		
		12 Month	24 Month	36 Month
Water	-	1.7	2.7	3.3
CCA	1.7	0	0	0
	4.3	0	0	0
	6.9	0	0	0
Impralit KDS	2.1	0	0	0
	4.1	0	0	0
	6.2	0	0	0
	8.4	0	0	0
	12.9	0	0	0

Ground Proximity Test:

Ground proximity tests (AWPA E18-06) were installed in Hilo and in Mississippi. At both fields fungi attacked the untreated controls whereas the treated lab-joints show only in the case of the Hawaii test side very little attack after 36 month at the lowest retention rates.

Graph 17: Ground Proximity Test Hilo

Treatment	Retention (kg/m^3)	Ratings		
		12 Month	24 Month	36 Month
Water	-	9.7	8.6	6.2
Impralit KDS	2.2	10	10	9.6
	4.4	10	10	9.95
	6.6	10	10	10
	8.9	10	10	10
	13.2	10	10	10
CCA	1.5	10	10	10
	1.9	10	10	10
	2.4	10	10	10
	2.9	10	10	10

The same test installed at the Mississippi test side give no evidence for fungal attack in all concentrations, while the untreated controls were attacked.

Graph 18: Ground Proximity Test Mississippi

Treatment	Retention (kg/m^3)	Ratings		
		12 Month	24 Month	36 Month
Water	-	9.3	8	7
Impralit KDS	2.2	10	10	10
	4.4	10	10	10
	6.6	10	10	10
	8.9	10	10	10
	13.2	10	10	10
CCA	1.5	10	10	10
	1.9	10	10	10
	2.4	10	10	10
	2.9	10	10	10

Efficacy against Insects

Lab-Tests:

Threshold values against different termites and house longhorn beetle in standard tests have been collected.

Toxic threshold:

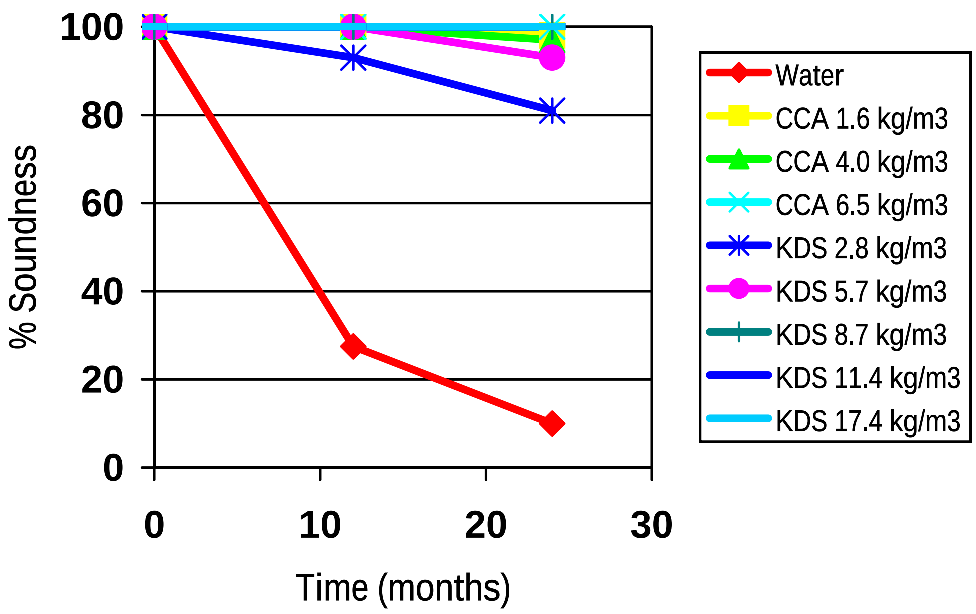
Hylotrupes bajulus	< 0.22 kg/m^3
Reticulitermes santonensis	< 2.89 kg/m^3
Mastotermes darwiniensis	< 1.83 kg/m3
Coptotermes acinaciformis	< 1.83 kg/m3

Toxic thresholds for termites found in Europe (Reticulitermes santonensis) and Australia (Coptotermes acinaciformis , Mastotermes darwiniensis) are similar.

Field Tests:

In conjunction with the field tests against decay a termite examination has been carried out.

Graph 19: Field Stake termite Test Saucier

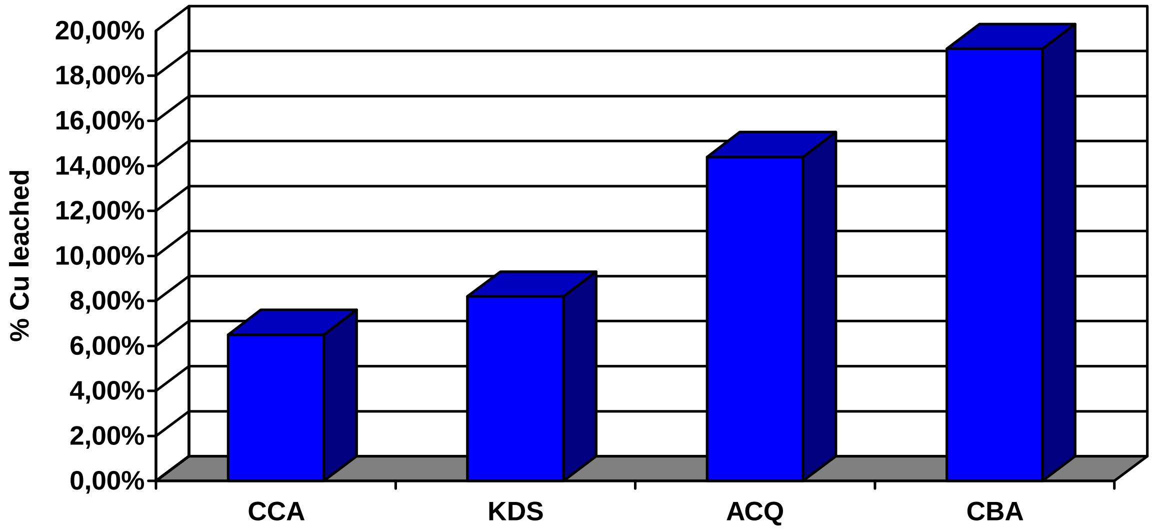


After 24 month untreated control stakes were nearly destroyed. At the lowest retention rate stakes treated with impralit KDS were slightly attacked. Stakes with higher loadings perform well.

Leaching Characteristics:

The impact of water borne wood preservatives for ground contact use to the environment is mostly determined by the leaching behavior. Due the fact that Polymer Betaine fixes excellent to wood and can be degraded in soil and Boron is a low toxic substance in the environment the leaching behavior of impralit KDS is defined by leaching of Copper. In a comparative leaching test with wood preservatives on the market it is demonstrated that the Copper in impralit KDS is fixed at the same stage as Copper of normally used CCA.

Graph 20: Cu leaching from different preservatives (Univ. FL method)



Conclusion

Impralit KDS is a unique preservative system, with an active – Polymeric Betaine - especially designed for the use in wood preservatives.

The excellent decay and insect efficacy is combined with a low toxicity and an acceptable influence to the environment.

Literature

Barth, V.; Härtner, H., A new type of biocide suitable for use in different fields of wood preservation, IRG/WP 93-30014

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