COST ACTION E22: Environmental optimisation of wood protection

Proceedings of the Final Conference
22 – 23 March 2004, Estoril, Portugal

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COST ACTION E22 - ENVIRONMENTAL OPTIMISATION OF WOOD PROTECTION

PROCEEDINGS OF THE FINAL CONFERENCE
22 – 23 MARCH 2004, ESTORIL, PORTUGAL

FOREWORD

In the Memorandum of Understanding for COST Action E22 a series of highly demanding objectives were adopted and technical benefits anticipated. The main overall objective was 'to improve and consequently to increase the cost-effective use of European wood by means of optimising the environmental factors relating to its durability'. Perhaps the most important technical benefit to be achieved was 'improved guidance in specifying durability requirements to reduce reliance on traditional biocidal wood preservatives'. COST Action E22 has concentrated on developing the scientific and technical basis necessary to achieve these aims. Independent evaluation of the work of COST Action E22 at its completion has commended the Action very highly in the achievement of its objectives.

Organised under three Working Groups of :
- Performance
- Impacts, and
- Innovations,

the programme of the Action addressed each of its specific scientific and technical topics through a series of Workshops culminating in the Final Workshop on 22-23 March 2003 in Estoril, Portugal. The outputs from all the various Workshops are available through the Action Website http://www.bfafh.de/cost22.htm and specific Workshops have been published as hard copy booklets listed at the end of these Proceedings. The Action has also published a 'State of the Art Review' of research capacity and activity in the field of environmental optimisation of wood protection throughout the 21 participating member States..

The Final Workshop set out to address a series of specific themes being an industry perspective on the changing face of wood protection, issues of biotransformation of traditional biocides and updates in the adoption of wood modification and heat treatments. A Special Session was devoted to developments in the control of termite invasion using modern baiting techniques. Finally two sessions provided updates on treated wood waste management and environmental risk management.

The Final Workshop was organised by Dr Lina Nunes and Dr Tania Nobre of the Laboatorio nacional de Engenharia Civil, Lisbon, to whom the Management Committee of COST Action E22 is most grateful. The MC would also like to acknowledge the support of the LNEC Institute and of Günter Siegel, the COST Scientific Secretary to the Action.

The Chairman and Management Committee of COST Action E22 wish to express their great gratitude to Vice-Chairman Prof Dr Rolf-Dieter Peek who has personally complied these Proceedings for publication.

Dr A F Bravery
Chairman COST Action E22

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Professor Dr R D Peek	Vice-Chairman
Dr D J Dickinson	Convenor WG1
Dr G Deroubaix	Convenor WG2
Professor Dr H Militz	Convenor WG3

16 October 2000

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Working Group 1 : *Performance*

WORKING GROUP 1 : *PERFORMANCE*

WG1 has been concerned with the performance of wood modified to extend its service life in relation to biological hazards. The main objectives were defined in the MOU as;

- Classifying hazard in relation to geographical location
- Efficacy prediction
- Improved guidance in durability performance
- Optimising preservative performance
- Determining bio-effective life
- Establishing realistic basis for predictive testing

During the Action particular attention was given to measures to reduce the scale of use of conventional preservatives by addressing the problems of specifying non-biocidal treatments and attempting to predict bio-effective life of products. This was specifically addressed in relation to field testing in Use Class 3 (formerly 'Hazard Class') and in the scientific basis behind testing in un-sterile soil in Use Class 4. The variation in performance in relation to geographical location and the effects of macro and micro-environment played an important role in the work.

In the Final Conference attention was given to the role of the newer biocide treatments where the currently available biocides were reviewed , together with the challenges facing the industry in relation to the Biocidal Products Directive. The problems of bio-transformation and distribution in wood of the newer organic preservatives was emphasised. The latest results of the European decay index were summarised together with those from Germany on the effects of micro and macro environmental influences in Use Class 3.

Dr David J Dickinson
Chairman Working Group 1 '*Performance*'

ISSUES FACING PRESERVATIVE SUPPLIERS IN A CHANGING MARKET FOR TREATED WOOD

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Keywords: wood protection, market, regulatory

Abstract: *This paper considers the changing market for wood protection and the challenges facing suppliers, in particular with respect to an increasing regulatory burden.*

1 Introduction

The modern wood protection industry was born in 1838 following the development of pressure impregnation of timber by Bethell. The treatment of sleepers and poles applying creosote via pressure impregnation grew rapidly throughout Europe to meet the rapid growth of the railways and telecommunications markets.

During the early 1900’s, research focused on the development of water-based systems mainly based on fluorides, chromates, nitrophenols and arsenates. In 1933 the first chromated-copper-arsenate (CCA) was invented by an Indian Government research worker, Dr Sonti Kamesan. CCA demonstrated excellent performance in the field and developments throughout the 50’s and 60’s led to the conclusion that a “Type C” product was superior when compared to other types.

CCA and creosote are broad spectrum preservatives which perform well in almost all end-uses and have been the mainstay in a market which has enjoyed relative stability right up to the 1990s.

One other significant development to address the incidence of decay in untreated joinery from the early 1960’s, was the use of organic solvent (OS) preservatives for double vacuum impregnation or immersion. The preservatives are carried in a light oil such as petroleum distillate or by means of a heavy oil. The double vacuum process was introduced in 1961 and has mainly utilised fungicides such as tributyltin oxide (TBTO), pentachlorophenol (PCP), and insecticides such as dieldrin and lindane. As with the waterbornes, the market was relatively stable until the mid-1990’s.

This paper will consider the drivers for change since 1990 and look at the issues and trends for wood protection in the future.

2 The Market

Within the European Union approx. 18 million m^3 of timber are pressure treated for a variety of end-uses as shown in Table 1.

Table 1 : European Pressure Treated Timber Volumes

End-Use	m^3 x million
Construction Timbers (HC1/2)	10.3
Joinery (HC 3)	0.6
Exterior out-of-ground (HC 3)	6.0
In-ground (HC 4)	1.8

Significantly, less than 5% of the total volume relates to poles, sleepers, and marine end-uses. However, this total volume has almost doubled over the past 15 years and reflects, in particular, the preference for treated wood in the construction and leisure areas. It is this changing market for treated timber, away from the main emphasis on heavy duty end-uses, which is providing opportunities for new treatment products providing “fitness for purpose” performance. This trend will likely continue for the foreseeable future.

Alongside the changing market needs, one other significant factor is the changing nature of timber sourcing. Following the changes in the political climate from the late 1980’s, the opening up and the development of the timber industry in Eastern Europe is providing enormous supplies of good quality timber. Alongside these developments we are witnessing the growth of a preservation industry in the Baltic States, Poland, Bulgaria, and Russia.

A number of these countries will shortly accede to the European Union and, therefore, be subject to the same legislative burden as the current 15 Member States. At the moment, the vast majority of timber treated at source in Eastern Europe is exported to other European countries and therefore market needs and regulatory restrictions in the EU have to be addressed.

3 The Political Climate

The Member States of the EU agreed to the Amsterdam Treaty which entered into force 1st May 1999. This Treaty extended the role for the EU in matters relating to health and safety and protection of the environment. Importantly it introduced a much wider role in terms of consumer protection and it is in this area alongside the growth in construction and leisure uses for treated timber which will provide the main challenges in the future for the wood protection industry.

The removal of the old political barriers is leading to the development of a single internal market. The accession of the Northern European countries in 1996 has led to ‘national’ problems and priorities being ‘exported’ throughout Europe.

At a recent conference, Environment Commissioner Margot Wallstrőm (Wallstrőm, 2003) set out her vision for regulation of the chemicals industry. She emphasises the EU focus on consumer and in particular child protection and quotes “EU strategy will focus on childhood diseases and the environmental factors with which they are associated, including air pollution, dioxins, heavy metals and endocrine disruptors.”

It is clear, that in a market which is dominated by construction and leisure end-uses, this message must be addressed by the supply industry.

4 Regulatory Issues

4.1 Products

The main focus of EU strategy is on products and consumer protection and it is in this area where Regulation will have most impact on industry. The main instruments for regulation are as follows :

4.1.1 Existing Substances Regulations (793/93/EEC, ESR)

In 1993, the Council adopted Council Regulation 793/93/EEC as the Existing Substances Regulations. These Regulations were intended to provide a framework for the evaluation and control of “existing chemicals substances" i.e. any substance listed in the European Inventory of Existing Commercial Substances (EINECS), an inventory containing 100, 195 substances. These substances were deemed to be on the Community market between 1st January 1971 and 18th September 1981. The Regulations set out the programme for evaluation and control of risks in four stages :

Stage 1 - Data collection;
Stage 2 - Priority setting;
Stage 3 - Risk assessment;
Stage 4 - Risk reduction.

Any chemical substance marketed after 18th September 1981 is called a new chemical, and before introduction in to the EC has to be notified and subjected to a risk assessment in accordance with the seventh amendment of the Dangerous Substances Directive (67/548/EEC). Generally, for substances introduced after 1981 the process has been handled well, but for existing substances, the legislation has not worked and only 17 substances have been evaluated in spite of the focus being placed on High Production Volume Chemicals.

The failure of these Regulations has led to new initiatives on EU chemical policy but until these are finalised, ESR will rumble on.

Of particular significance to the wood protection industry is the ESR evaluation of chromium compounds, currently being undertaken by the UK's Health and Safety Executive (HSE). The evaluation has proceeded through the first three stages and exposure to Cr(VI) through inhalation and dermal routes are identified as important pathways. The assessment also states that based on available data and the genotoxicity, a dose-response relationship or threshold level cannot be deduced. This opinion has been agreed by the EC's experts, the Scientific Committee on Toxicity, Ecotoxicity, and the Environment (CSTEE).

Consequently, the HSE is now responsible for developing a risk reduction strategy as in Stage 4 and wood protection is in the forefront of this evaluation.

Any recommendations regarding restrictions on use are likely to be imposed through the Marketing and Use Directive.

4.1.2 REACH

Intended to replace the existing flawed system for the regulation of chemicals in the EU, is the new framework of REACH (Registration, Evaluation, and Authorisation of Chemicals).

The Commission intend for REACH to allow a sustainable chemicals policy by protecting human health and the environment from the risks of chemicals, and to encourage innovation and competitiveness.

Industry fully support these objectives but have expressed serious concerns over the practicalities of the proposals and in particular the impact on competitiveness. The proposals do not address how to deal effectively with the vast volumes of chemicals, preparations, articles containing restricted chemicals, which are imported into the EU. As it stands, there is a real risk of increasing the number of dangerous and unauthorised substances which find their way into Europe if it causes relocation of manufacturing away from Europe.

Whilst biocides will be assessed under the Biocidal Products Directive (98/8/EC), substances used in wood protection formulations or as additives will be subject to REACH. Low volume or specialist chemicals used as such may not warrant the investment to keep them on the market thereby restricting options for the supplier.

4.1.3 Biocidal Products Directive (98/8/EC)

Detailed rules for a ten-year programme to review the safety of biocides have been set out in Commission Regulation 2032/2003/EC. The Regulations require the environmental and health hazards of all existing biocidal active substances (23 product types including wood preservatives) to be reviewed at EU level. Biocidal products e.g. wood preservative formulations, containing approved active substances, will then need to be approved at national level.

A total number of 360 active substances have been successfully notified, some 37% of the total number of existing substances. This means that the remainder will disappear from the European market in less than 3 years, a process which has already occurred in the pesticides regime. Of these notifications, 81 are as wood preservatives (Product Type 8) but already we are witnessing withdrawal of support from a number of these substances including TBTO, ethanol, lactic acid, cetyl/pyridinium chloride, deltamethrin, cyfluthrin, organotin, and fipronil.

The final date for submission of dossiers for wood preservatives is 28 March, 2004 and only then will the full picture of supported active substances be clear. However, the greatest uncertainty is how Member States will conduct the assessments. For wood preservatives a number of issues around environmental risk assessment have still to be resolved.

There are many outstanding issues regarding the implementation of these Regulations, principally around data protection and scope. However, the costs of compiling and assessing dossiers is huge with estimates around 4 million euros for one active substance. This clearly will impact on the availability of new active substances for wood preservative formulations and focus research more onto improving performance of existing products.

4.1.4 Marketing and Use Directive (76/769/EC)

This Directive has been used to regulate the marketing and use of dangerous substances. The scope of this Directive has extended to include treated wood articles and major restrictions on wood treated with creosote, pentachlorophenol, and more recently chromated-copper-arsenate have been introduced. Often, this Directive has reflected political and economic interests of certain Member States and will continue to implement restrictions on products and articles in combination with the other main Directives regulating chemicals.

4.1.5 The Consumer

One of the trends driving corporate interests is growing consumer concern about themselves and their own health. Environmental Group such as Greenpeace and Friends of the Earth are taking advantage of such concerns by focusing their attention more and more onto the large retail groups. FOE ranked retailers' performance by creating red, amber, or green lists, and those retailers on the red list were threatened with disruptive action and boycotts of their stores.

These pressures have seen major retailers adopt "chemicals policies" across a wide range of consumer products including coatings and timber-based materials. Such policies can conflict with regulation and must also be addressed by the suppliers.

4.2 Application

Over the past 20 years or so, the implementation of European environmental legislation at national level has resulted in major improvements in application design and techniques in the timber treatment industry. Together, with the rationalisation and new investment in timber treatment operations, most modern sites operate on a full containment basis.

The framework legislation is the Integrated Pollution Prevention and Control Directive, (IPPC, 96/61/EC) and this Directive considers emissions to land, air and water.

Within Europe, products containing "red list" substances such as lindane, pentachlorophenol, or TBTO, are barely used. Solvent-containing formulations for construction have been largely replaced by water-based products. Where solvent-based products are used for some joinery applications, formulations and application techniques have been developed to minimise emissions outside of the requirements of the Solvents Emissions Directive (1999/13/EC).

Future issues likely to impact on the wood protection industry relate to energy consumption and optimisation and, therefore, affect those processes which are high energy intensive.

5 Performance

The Construction Products Directive (89/106/EC) requires the establishment of harmonised product standards. The wood protection industry has actively participated in the standards development programme and the current programme of work involves more than 50 European standards. At the European level (CEN/TC38), 8 Working Groups have been established to develop standard test methods relating to durability classification, performance, assessment and specifications, fungal testing, insect testing, external factors, physical/chemical factors, exposure aspects and creosote, to underpin product standards.

Once adopted, the product standards take on a regulatory nature which is radically different from the voluntary regime which prevailed in the past and will effectively define what is possible in the market in the future.

These standards take on board environmental issues in accordance with EU strategy recognising that it is no longer acceptable to treat wood with inappropriate and excessive quantities of chemicals. The standards emphasise the use of optimum quantities of preservative taking into account the end-use to which the treated wood is being placed.

However, European Standard test methods are mainly based on laboratory tests which compare the performance of the product against fungi and insects in artificial conditions. Whilst these tests provide a good indication of the preservative efficacy, they are not always accurate indicators of performance and service life in actual use. Current developments to include relevant preconditioning regimes will improve the validity of these test methods. Appropriate field tests alongside such laboratory tests are essential tools in determining the likely true long-term performance.

At the Scandinavian Wood Reservation Conference (Edlund, 1998) discusses the need for field tests concluding that field tests provided a guarantee that products which are approved are of high quality, which in the long term will benefit the consumers, the timber industry, and last but not least, the wood preservation industry.

Butcher (Butcher, 1985) reviews the disastrous experience of quaternary alkyl ammonium compounds (AAC's) in New Zealand when introduced in the late 1970's. Failures on a huge scale led to liabilities of over NZD 9 million. Products were approved based almost solely on laboratory performance, and he concludes that a laboratory evaluation of a new preventive system, however, detailed, is inadequate to predict likely performance in service.

Also, standards development is based on tried and trusted custom and practice and current standard test methods are very much based on comparative performance against traditional preservatives such as CCA. This works well for protection systems with similar mechanisms but may not be suitable in predicting performance for new systems which rely on non-biocidal methods of protection.

An example of where changes away from chemical treatment to more "environmentally friendly" non-chemical means has resulted in disastrous consequences has been witnessed in New Zealand in wall frames. The compulsory use of treated timber in wall frames was dropped about 6 years ago and replaced by heat-treated timbers. Evidence of "leaky homes" has created a crisis in New Zealand with some estimates of bills of around NZ$ 1 billion to counter rot and leak damage.
The Building Industry Authority (BIA) in New Zealand has announced a programme to address the leaky buildings problem. This includes design improvements coupled with additional chemical treatments.

In order to protect the future integrity of the wood protection industry, development of appropriate standards for newer systems with the emphasis on "fitness for purpose" and "desired service life" are essential. Field tests and appropriate preconditioning in laboratory tests should be an integral part of this process.

6 Waste Management

Currently most timber wastes are disposed of in landfill sites but under the Landfill Directive (1999/31/EEC), Member States are required to reduce the quantity of biodegradable waste over the next 20 years. In the UK, for example, the government are targeting a reduction to around 1/3 of current levels.

The hierarchy for preventing waste where possible and making more sustainable use of any waste which is produced, is recognized in EU strategy, namely;

- Reduce/minimise waste generation;
- Re-use waste materials;
- Recycle waste materials;
- Recovery (e.g. energy recovery, composting);
- Disposal (incineration without energy recovery, landfill).

Disposal is the least favoured of the above options.

Under EU classification requirements, CCA and creosote – treated wood waste are classified as hazardous and therefore disposal on recycling options are further limited.

7 The Challenge

The chemical wood protection industry has been subject to tremendous change over the past 20 years following almost 200 years of conservatism. As with most industries, and in particular those linked to the chemical and forestry industries, environmental factors play a huge part in the public and regulatory acceptability of treated timber products.

The environmental credentials of timber as a constructional material are well documented; it is the only true renewable building material and it takes far less energy to produce timber than any other building material.

The environmental impact of decisions around the selection of materials for construction has been considered in some detail at the Building Research Establishment (BRE) in the UK and these are discussed at the Annual Convention of the Institute of Wood Science, 2002 (Ewbank, 2003). From the BRE work it is concluded:

- The most significant impact factors overall are energy consumption in manufacture and in transporting materials to site (i.e. embodied energy) and energy efficiency in use.
- Timber forms of construction tend to score well in most applications.
- Timber treatment is so insignificant a factor overall as to not alter a functional unit's impact rating, regardless of whether it is treated or not.
- The use of treated softwood in structural or non-structural elements does not undermine the strong environmental case for the use of wood components in buildings.

Indeed, in many situations, treatment improves the overall rating of timber by extending the life of a component and so minimising the environmental impact of early disposal and replacement.

These conclusions are reinforced by the application of Life Cycle Assessment (LCA). Imperial College in the UK has developed LCA of forest products and conclude that under such assessments, forest timber products fare well. (Hillier and Murphy, 2000). Their studies, which bring in elements of forest growth and disposal and recycling at end-of life, provides a positive context within which to make environmental assessments of forest products. Treated timber products compete extremely favourably when compared to alternative materials.

With such a good story and with increasing supplies of timber from Eastern Europe the opportunities for chemical wood protection are boundless. The aesthetic properties of wood are well accepted and the challenge for the industry is to continue to supply products which provide the required performance using acceptable biocides and chemicals (to the regulator and consumer), and importantly are competitive against alternative materials.

Over the past 20 years, the industry has been both proactive and responsive to these drivers for change. Whilst traditional preservatives such as CCA, creosote, and PCP are now severely restricted or banned a whole range of new products are available for the treater. These can be broadly categorised according to either the defined end-use of the treated wood, or by product type.

i) Water-based organic biocide preservatives which are designed mainly for internal construction timbers such as roof trusses or framing. These products can also be used for joinery (usually in organic solvents) or in external out of ground situations when a coating is applied. They are not suitable for unprotected out of ground or in ground applications. The biocides which are commonly used are triazoles combined with synthetic pyrethoids. All the components are being supported under the Biocidal Products Directive.

ii) Copper-chromium based preservatives mainly designed for external in-ground and unprotected out-of-ground applications. A number of products are available including CCB, (chromated-copper-boron), CC, (chromated-copper), and CCP, (chromated-copper-phosphate).

These products have been around for many years and to a greater or less extent, have been used commercially in Northern and Central European markets. Chromium fixation in all three products is not as rapid or complete as with CCA although the differences between CCA and CCP are relative small. In Northern European markets the use of chromium based preservatives is restricted in a similar way to CCA and these products are not allowed in Denmark.

The future of these products which contain chromium is under almost immediate threat by the Commission's Risk Reduction Programme.

iii) The third and the most popular group of alternative products are those based on copper in conjunction with an organic biocide. These products are less generic than the traditional chromated preservatives and performance and ancillary features depend on the composition and treatment parameters. TANALITH E from Arch Timber Protection is based on a unique blend of copper and triazole fungicides and has been in successful use on the European market since 1993 and is now sold in over 20 countries worldwide. Wood treated with TANALITH E can be used in internal and external constructions and in ground contact exterior situations. Favourable long term field test data are available from 23 sites around the world to underpin the products performance.

ACQ's are based on copper plus a quaternary ammonium chloride organic biocide and is available in ammoniacal or amine form with different quaternary compounds. In the UK, they are marketed by Rockwood and Osmose for the same end-uses as TANALITH E.

Another copper-based product is supplied in Europe by Dr. Wolman and based on Cu-HDO.

All of these compounds are being supported under the BPD and if authorised will be around until at least 2010.

And so, the industry has developed alternatives to the traditional products which have proven performance and are not only acceptable to the regulator but provide the consumer with a real choice.

Whilst costs of treatment may be slightly higher for some end-uses, for many end-uses they are similar or even lower. When the Danish government stopped the approvals of CCA and then chromium-based products in the early to mid-nineties, the industry expressed concern about its future. However, companies embraced the challenge and through the change to copper based preservatives aggressively marketed own brands and unique features of their products. Contrary to many forecasts, the industry has flourished and benefited from the changes as treated wood continues to be the material of choice.

The challenge for the suppliers is to pre-empt the regulatory and market drivers and look to next generation products beyond 2008-2010. New organic biocides such as Bethogard, and which are effective against soft-rot are already well developed and such biocides will form the basis of future protection systems. Current standard test methods, developed for low hazard or metal-based systems are unlikely to be appropriate for these systems and new test methods will need to be developed. Ancillary properties will need to be addressed if the treated wood is to remain attractive in use. The natural colours and weathering properties of metal-based systems will need to be matched as growth in the leisure and garden wood markets continues.

The key to success will be formulation skills and the delivery and protection systems for these new organic biocides. A fundamental understanding of the mechanics of degradation with these systems is essential if this challenge is to be successful.

The market for treated wood has almost doubled over the past 15 years, mainly due to growth in the construction and leisure areas.

As long as the specifier and consumers are provided with a first class durable and attractive product, at the right price, there is no reason as to why this growth should not continue with treatment of wood with alternative, non-restricted chemical protection products.

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CHEMICAL WOOD PROTECTION: IMPROVEMENT OF BIOCIDES IN USE

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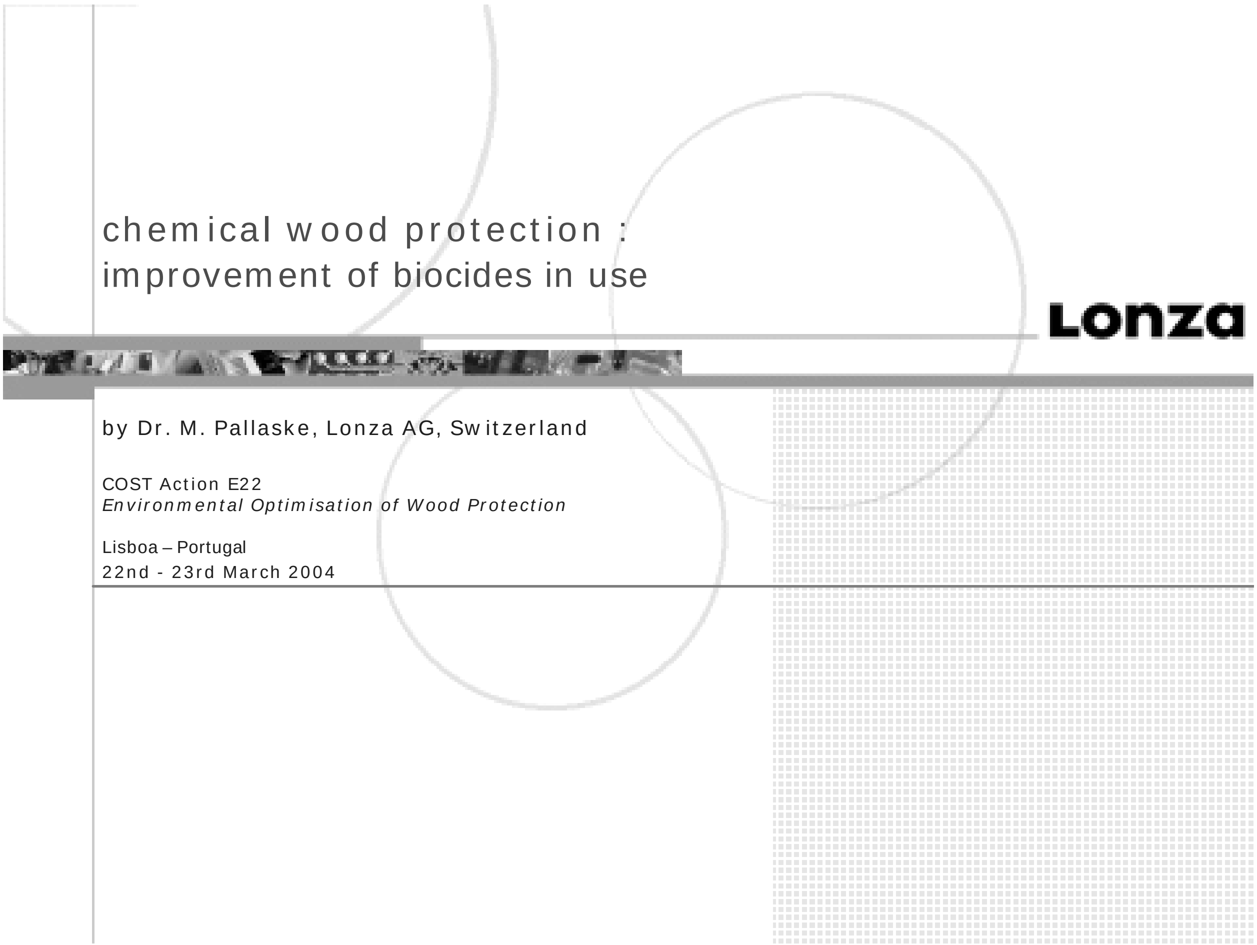
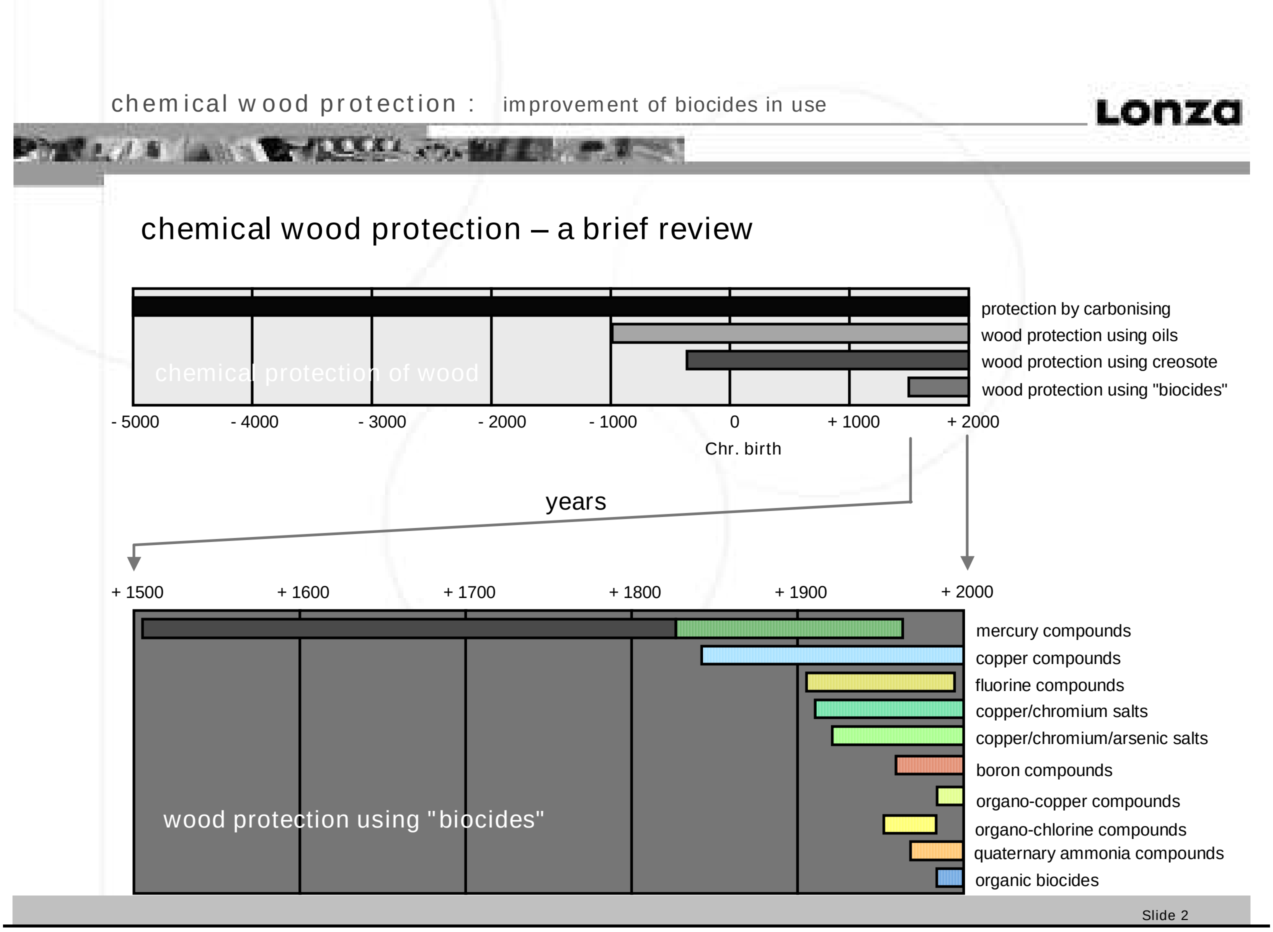


chemical wood protection :
improvement of biocides in use

by Dr. M. Pallaske, Lonza AG, Switzerland

COST Action E22
Environmental Optimisation of Wood Protection

Lisboa – Portugal
22nd - 23rd March 2004



chemical wood protection : improvement of biocides in use

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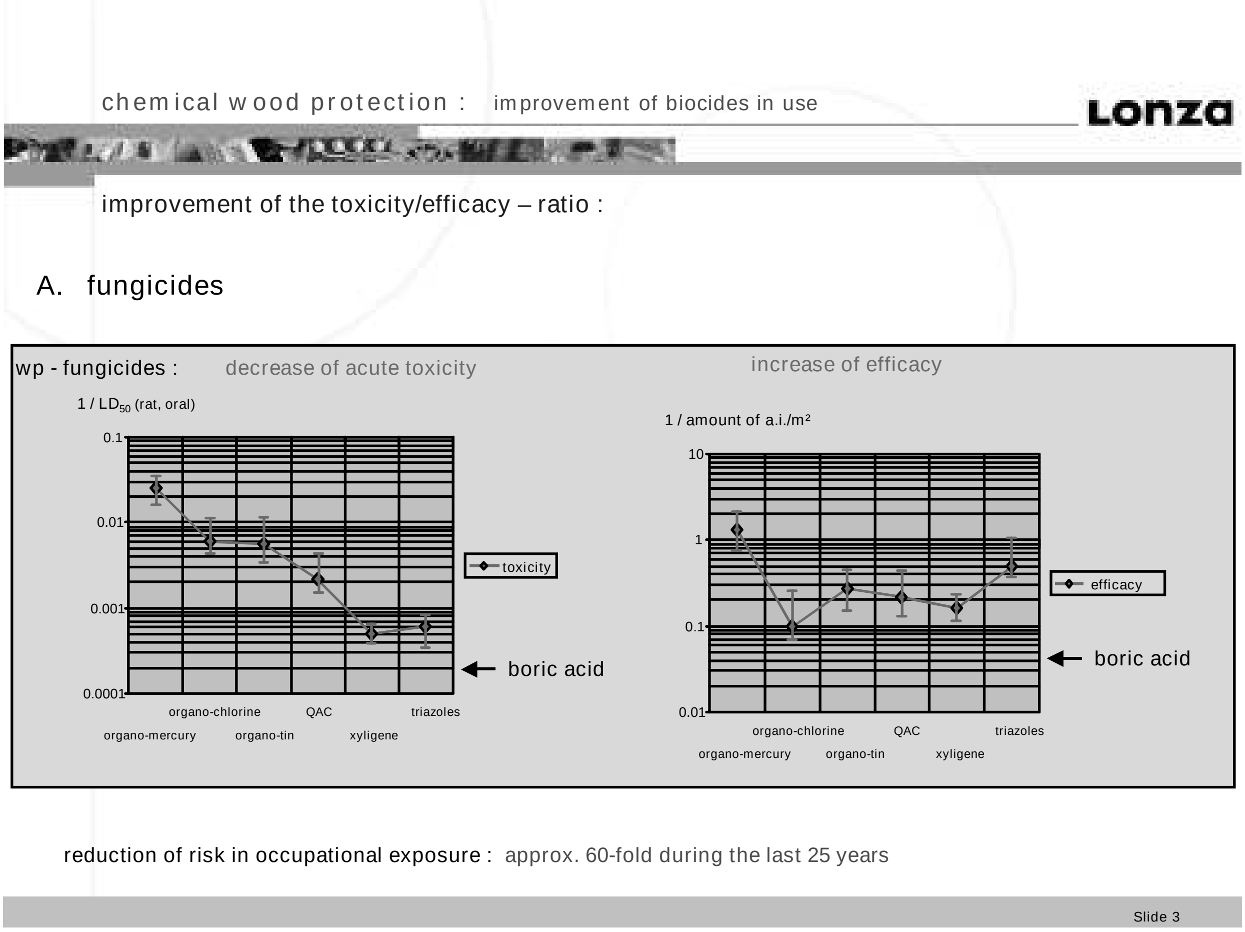
chemical wood protection – a brief review



chemical wood protection : improvement of biocides in use

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improvement of the toxicity/efficacy – ratio :

A. fungicides

reduction of risk in occupational exposure : approx. 60-fold during the last 25 years

chemical wood protection : improvement of biocides in use

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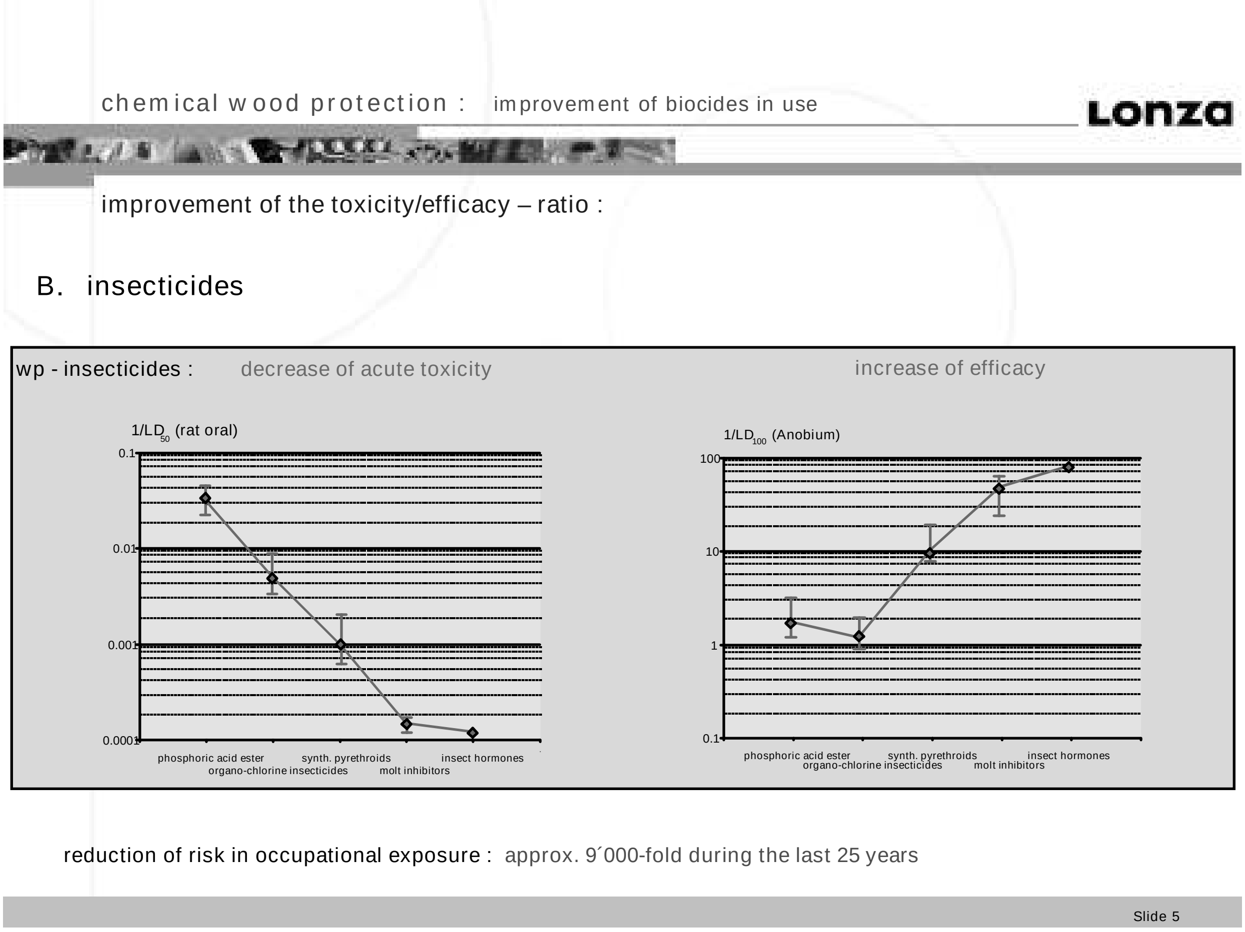
improvement of the environmental impact :

A. fungicides



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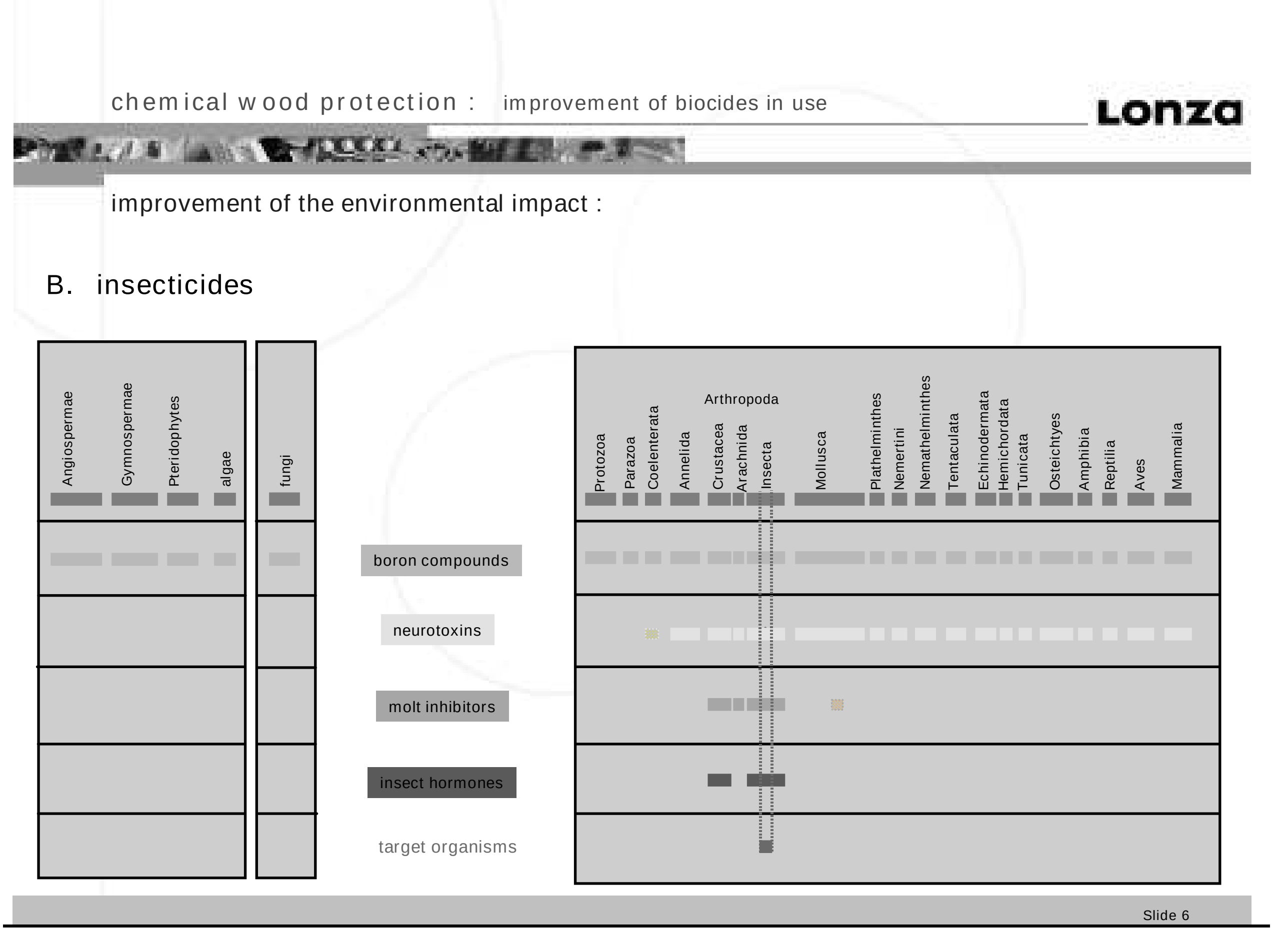
improvement of the toxicity/efficacy – ratio :

B. insecticides

wp - insecticides : decrease of acute toxicity increase of efficacy

reduction of risk in occupational exposure : approx. 9´000-fold during the last 25 years



chemical wood protection : improvement of biocides in use

improvement of the environmental impact :

B. insecticides

	Angiospermae	Gymnospermae	Pteridophytes	algae	fungi	Protozoa	Parazoa	Coelenterata	Annelida	Crustacea	Arachnida	Insecta	Mollusca	Plathelminthes	Nemertini	Nemathelminthes	Tentaculata	Echinodermata	Hemichordata	Tunicata	Osteichtyes	Amphibia	Reptilia	Aves	Mammalia
										Arthropoda															
boron compounds	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■
neurotoxins								▨	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■
molt inhibitors										■	■	■	▨												
insect hormones										■		■													
target organisms												■													

Slide 6

chemical wood protection : improvement of biocides in use

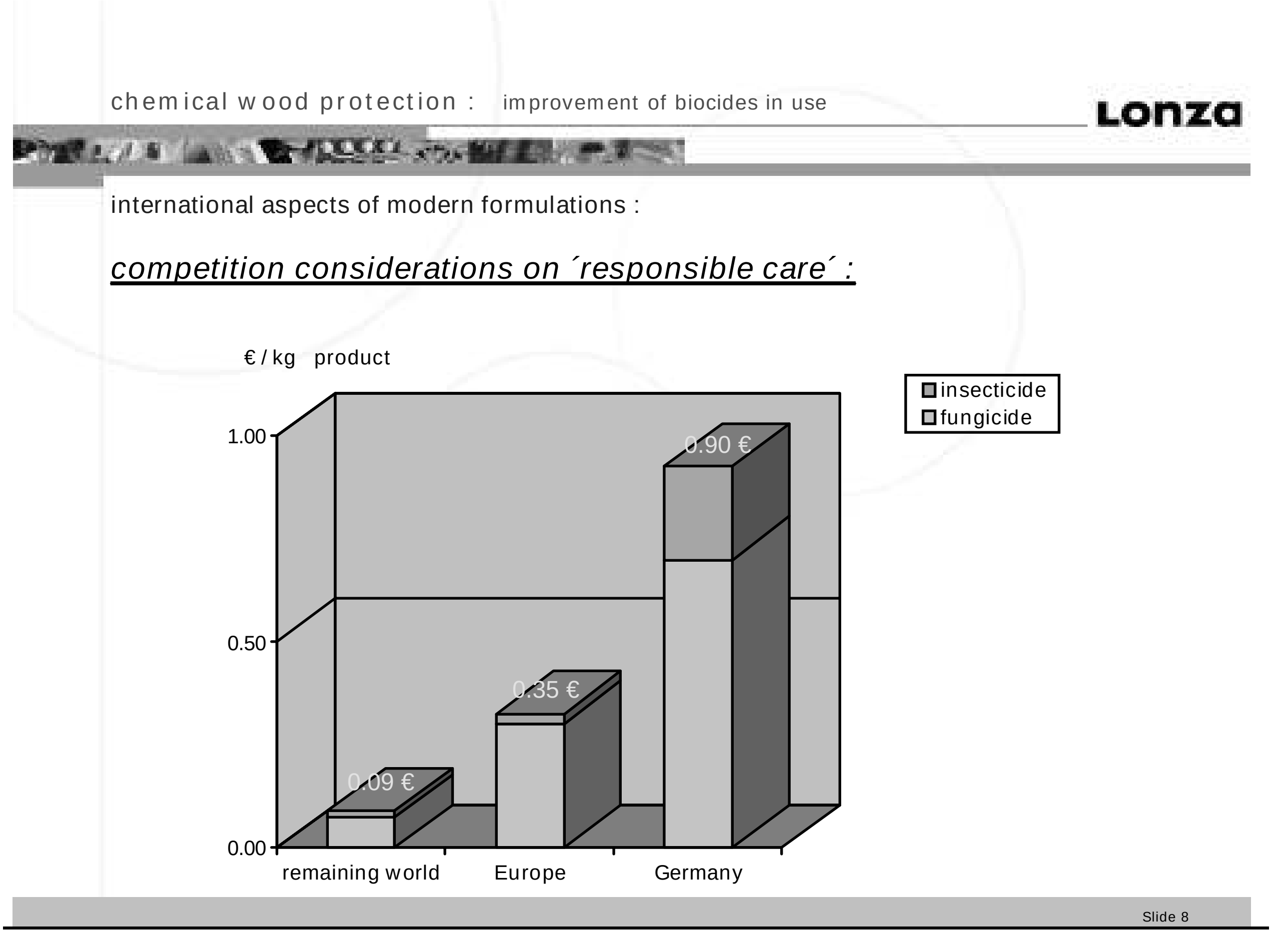
Holzschutz : Konsequenzen der "responsible care" - Verpflichtung

international aspects of modern formulations :

do a good job and talk about it -

- but ...

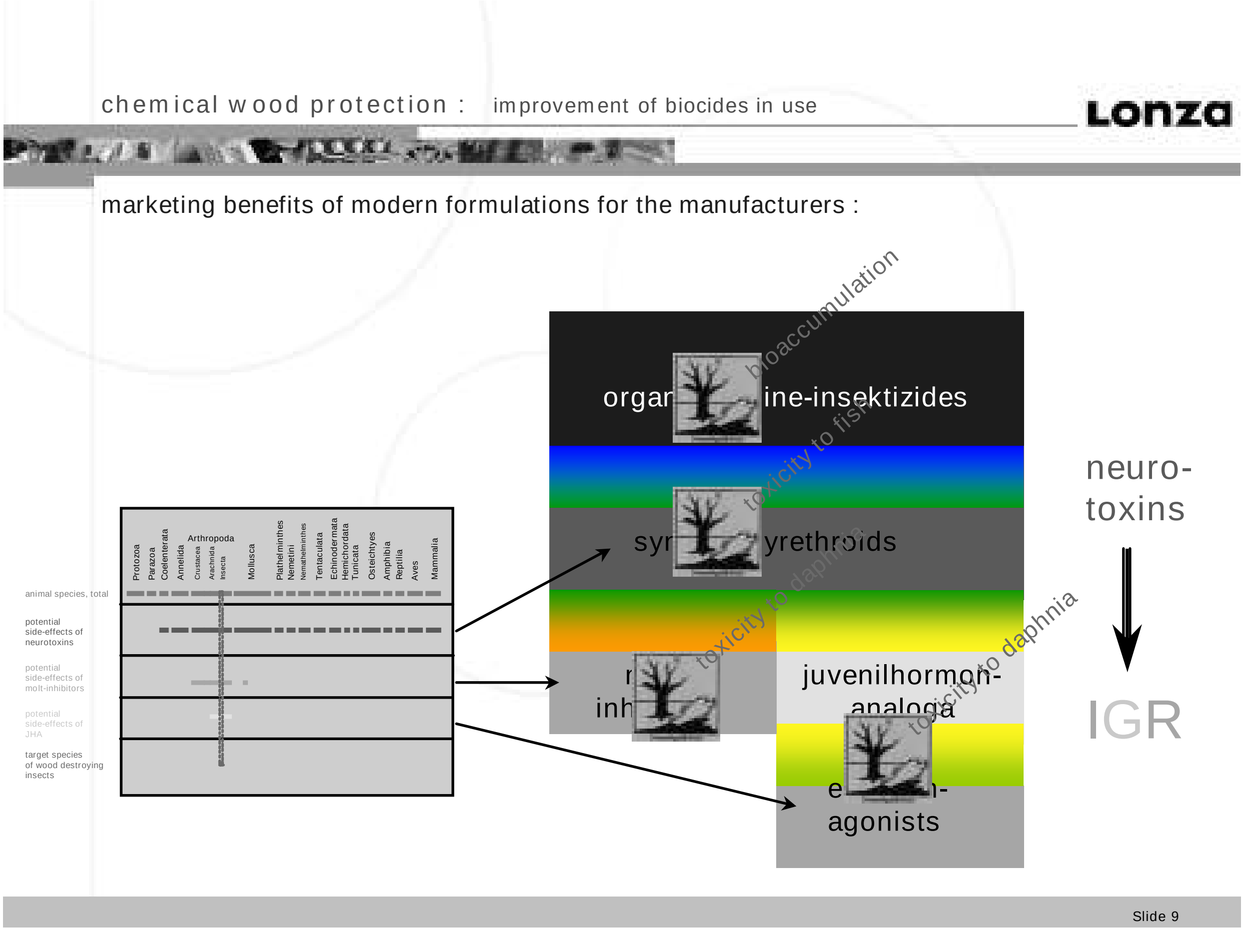
Slide 7



chemical wood protection : improvement of biocides in use

international aspects of modern formulations :

competition considerations on ´responsible care´ :



chemical wood protection : improvement of biocides in use

marketing benefits of modern formulations for the manufacturers :

chemical wood protection : improvement of biocides in use

registration cost aspects of a single formulation :

+ 45´000.- BPD registration-€ for each formulation in Germany

+ 15´000.- BPD-€ for national re-registrations

+ 25´000.- BPD testing-€ for each formulation

chemical wood protection : improvement of biocides in use

consequences :

BPD does not help improving performance because of extreme overhead costs

BPD will promote the use of old biocides

BPD will delay innovation because of extreme long return-of-investments

BPD will cause a severe reduction of R&D-activities

BPD will cause a very reduced number of different formulations

BPD will remove smaller player from the wood protection market

THE TOOLS AT OUR DISPOSAL

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Keywords: wood protection, active substances, performance, standards

Abstract: *This paper discusses the recent product changes in the industrial pre-treatment preservation industry, the current challenges facing the chemical manufacturers and formulators, and the implications for product development and performance.*

1 Introduction

Timber preservation has a long history, but has only developed as an industry over the last two centuries. With the industrial revolution and developments like Stephenson’s locomotive, pressure vessels were developed. In 1831 a Frenchman Jean Robert Breant patented a method for applying wood preservative by pressure in a sealed iron vessel. In this process a vacuum was first applied to the timber to remove the air followed by pressure injection of the preservative. Prior to this time most preservatives were applied by simple brush on methods.

The year 1838 marked the beginning of modern industrial wood preservation. During this year John Bethell invented a practical process for treating wood with preservative. The process involved the use of an initial vacuum, a pressure period and final vacuum. At the end of the process the wood cells are full of preservative, hence the name full cell method. One of the preservatives mentioned in Bethell’s patent was “dead oil of coal tar” or creosote. In his description of the equipment required, Bethell says “an air or water tight vessel or tank must be made of strong metal, of any form that may be preferred “one made like a circular wrought iron boiler for high pressure steam engines answers very well, strong enough to withstand an internal pressure of 200 pounds per square inch”. Bethel began operating a treatment plant using creosote oil and soon the process became so popular as to become known as the “Bethell process”. The creosoting industry grew quickly, a mere 15 years after Bethell patented his process one timber preserver was treating 10,000 railway sleepers a week.

Since these early days, the pre-treatment timber preservation industry has become a major global industry but has undergone many changes. Today it is less reliant on the treatment of utility timbers, with construction timbers, fencing and more recently leisure wood being the major growth markets. Leisure wood gives an added challenge with customers placing greater emphasis on the aesthetic properties with the need for water repellency/dimension stability, coloured timber and timbers which have a resistance to staining, thereby minimising maintenance.

Another important trend is the move away from a single product being used for a number of commodities, for example CCA which is commonly used for the treatment of timbers in hazard class 2, 3, 4 and 5. This trend is likely to continue as new metal-free active substances have a narrower spectrum of activity and need to be targeted to a specific biological hazard.

Whilst many of the industry changes have been regulatory driven, the preservation industry has embraced the challenge and today, there is a range of modern products with proven performance which provide the market with a real choice.

This paper reviews some of the more recent changes in terms of how the industrial pre-treatment chemical manufacturers have adapted their product ranges and looks at the current challenges facing the chemical manufacturers and formulators and the implications for product development and performance.

2 Past and present and future trends for the European timber preservation market

Irrespective of the type of active substance used, a wood preservative system must be designed to meet a series of requirements. These include permanence in the wood structure (from water-leaching, evaporative ageing, biological degradation or chemical degradation), spectrum of activity, overall performance, handling characteristics, health, safety and environmental characteristics and of course, cost.

Wood preservative formulations are often classified into three categories based on end-use:

- Industrial pre-treatment of timber
- Professional, remedial and preventive of timber in-situ
- DIY and amateur application

Only the industrial pre-treatment of timber sector is being considered within this paper, although many of the market trends and regulations apply to all three categories.

Historically, wood preservative compositions for the industrial pre-treatment of timber sector have been distinguished based on the type of carrier and three distinct groups are generally cited, namely:

- Tar oils, for example creosote
- Light organic solvent based preservatives
- Metal containing water-based formulations

With the new developments over the recent years and the reduced reliance on heavy metals, it is now appropriate to consider a fourth group, namely:

- Metal-free water-based formulations

Each of the above formulation types is discussed below by end use class (hazard class) in terms of how a particular sector has been influenced by regulations, the types of products which have evolved and the expected performance levels.

The hazard class system (EN335-1, 1992) is based on 5 classes and is a convenient way of describing the requirements wood preservatives as summarised below:

Table 1 – Hazard class system according to EN335-1.

Hazard Class	Service Conditions	Typical uses	Biological Agents
1	Interior, dry	Framing, roof timbers	Beetles and Termites (Insects)
2	Interior, damp	Framing, roof timbers	Insects and Decay Fungi
3	a) Protected exterior	Exterior joinery	Insects, decay fungi and disfiguring (bluestain) fungi
	b) Unprotected exterior	Deck boards	
4	a) In-ground	Fence posts	As HC-3 plus soft rot fungi
	b) Fresh Water	Cooling tower	
5	Marine	Piles	As HC-4 plus marine borers

2.1 Hazard class 1

The only risk in this hazard class, but one which is applicable to all hazard classes is insect and where applicable termite attack.

Organic insecticides based on chlorinated hydrocarbons dissolved in a light oil such as white spirit have been used successfully for many years. Lindane, dieldrin and aldrin were commonly used throughout the 1960's through to the 1980's but have since been replaced with more modern insecticides of lower mammalian toxicity and improved eco-tox profiles.

A number of new insecticides have been introduced over the last 10 years or so and the trend continues even today. Today's insecticides include pyrethroids, chloronicotinyl and neonicotinoids, pyroles and phenylpyrozoles, insect growth regulators (IGR), and chitin synthesis inhibitors (CSI's).

The other major trend is the move away from solvent-based formulations to water-based formulations for environmental, regulatory and cost reasons. In the UK, the move away from solvent-based preservatives coincided with the introduction of the Construction Products Directive and the 'results based' specifications in accordance with European standards such as EN351-1 and EN599-1. These standards gave the formulator more freedom and a vast number of water-based formulations were introduced in the 1990's, so much so that except for the joinery market, the majority of the interior construction market is now using water-based micro emulsions.

The Solvent Emissions Directive (EC Directive 1999/13/EC) accelerated this transition with treaters consuming more than 25T of solvent per year having to treat to less than 11kg/m^3 solvent or look to implement solvent recovery systems.

The most widely used organic insecticides in wood preservatives in Europe today where protection against woodborers and termites is required are the pyrethroids, permethrin and cypermethrin. These are highly active and are typically used at 0.05-0.1 g/L. They are used in both water-based (as micro-emulsions) and solvent based formulations.

Where protection against just woodborers is required, the IGR flufenoxuron (Flurox) is commonly used, this recently replacing another IGR, fenoxycarb (Farox). Some concerns have recently been expressed around the slow acting nature of these ingredients and performance.

Borates have also been widely used for many years in water-based systems to protect against both wood borers and termites.

2.2 Hazard class 2

The trends for this hazard class mirror those in hazard class 1 with solvent-based preservatives being largely replaced with water-based formulations. In addition, there has also been a significant move away from heavy metals to organic only biocides.

Other than chromated copper arsenate (CCA) and borates, preservative formulations used in the UK prior to the introduction of the CPD for the treatment of interior or coated exterior timbers, complied with BS5707. Table 2 lists the fungicides whose efficacy had been assessed by BSI for use in solvent-based formulations and were included in the 1997 version of this standard.

Table 2 - Minimum concentration of fungicides ex BS5707, 1997

Fungicide(s)	Minimum concentration *% (m/m)*
Pentachlorophenol	5.0
Tri-*n*-butyltin oxide	1.0
Pentachlorophenol/Tri-*n*-butyltin oxide	1.75 pentachlorophenol/0.44 tri-*n*-butyltin oxide
Zinc naphthenate/pentachlorophenol	1.8 zinc/2.0 pentachlorophenol
Zinc naphthenate/pentachlorophenol	1.0zinc/5.0 pentachlorophenol
Pentachlorophenyl laurate	6.25
Copper naphthenate	2.0 copper
Copper naphthenate	2.75 copper
Acypetacs zinc	14.5
Zinc versatate	2.9 zinc
Triexylene glycol biborate	3.5
Tri-*n*-butyltin naphthenate	1.8
Tebuconazole	0.75
TCMTB	2.25 TCMTB
Propiconazole	1.0

It is clear to see that the use of tin, copper and zinc, in various forms, were prevalent at the time. Interestingly, two of the latest entries were tebuconazole and propiconazole.

Although initially developed as fungicides for seeds and plants, the value of azoles as wood preservatives was quickly recognised and this sector of the market is now dominated by the triazole fungicides in the form of micro-emulsions. Their mode of action is primarily the inhibition of ergosterol biosynthesis (Kato *et al.*, 1986).

Tebuconazole alone formulations are very effective against *Gleophyllum Trabeum* (Exner, 1991) but relatively poor against *Poria Placenta* whereas propiconazole alone formulations (Valcke, 1989) are the opposite. Mixtures of azoles were identified as offering an improved spectrum of activity (see Table 3) and a 1:1 mix of tebuconazole and propiconazole is widely used due to its improved spectrum of activity.

Table 3 - Toxic values as kg tebuconazole plus propiconazole (1:1) /m^3 wood (Buschhaus and Valcke, 1995).

Test fungus	Unaged	After EN73	After EN84
Coniophora puteana	< 0.050	< 0.050	< 0.050
Gleophyllum trabeum	< 0.050	< 0.050	< 0.050
Poria placenta	0.050 – 0.100	0.050 – 0.100	0.100 – 0.150
Coriolus versicolor	0.170 – 0.230	0.170 – 0.230	0.320 – 0.420

Azaconazole is no longer used but new azoles such as cyproconazole are being used successfully outside of Europe. Although protection against microfungi is less important in this hazard class than hazard classes 3 and 4, azoles used in combination with IPBC (3-iodo-2-propynylbutyl carbamate) are growing in popularity.

Quaternary ammonium compounds such as BAC (alkylbenzyl ammonium chloride) and DDAC (didecyldimethyl ammonium chloride) and borates are also used often in combination with azoles.

Within the last couple of years, a number of patents have been granted claiming improved efficacy of biocides when used with adjuvants, for example amine oxides (Tseng and Walker, 2003 and Walker, 2003). The mechanism of the adjuvants is unclear but these non-biocides may affect the micro-distribution of the biocide in the treated timber or their interaction with fungi.

Preservatives based on copper in conjunction with organics biocides, such as copper-azole and copper-quat and copper-organometallics such as copper-HDO are the other major preservative groups used for the protection of construction timbers and are discussed in more detail under hazard class 3.

2.3 Hazard class 3

There are two distinct hazard class 3 categories depending upon whether the timber is to be coated or uncoated.

i) Uncoated

In uncoated end uses, CCA and creosote have been the dominant preservatives for the last 60 years except in the Germanic regions where CCB (copper chrome borate) has dominated until recently. Where CCA restrictions were imposed, which was as early as 10 years ago in Denmark, the markets experimented with other chromium-based preservatives such as CC (chromated copper) and CCP (copper chrome phosphate). Experiences were mixed, with numerous reported premature failures of CCP treated timber.

In a number of European countries, the copper/organic biocide, chromium-free preservatives have become the dominant preservative type, either through market choice or due to national restriction on the use of chromium in timber preservation, for example Denmark.

Countries which continued to use CCA throughout the 1990's and to the present, are now having to consider alternatives due to the imminent imposed restrictions on arsenic (Marketing and Use Directive on arsenic, EC Directive 2003/02/EC). It could be argued that the decision making process for a treater today is an easier one than that faced by the Scandinavians in the 1990's; the copper/organic biocide preservatives have now been able to demonstrate their performance through long-term field tests and 10-15 years in use service, and it is now becoming apparent that future restrictions on the use of chromium are likely to be only a couple of years away. Consequently, the majority of CCA treaters having to make the move away from CCA are making the transition to the copper-organic range of preservatives. The situation in France is even clearer, with CTBA now only approving arsenic free, copper-chromium containing formulations for the treatment of vineyard stakes and agricultural buildings following the reclassification of Cr(VI).

All three major product types are currently approved by the Nordic Wood Preservation Council (NWPC) for use in Class AB (hazard class 3), their current approved retentions being:

Table 4 - Major product types currently approved by the Nordic Wood Preservation Council (NWPC) for use in Class AB (hazard class 3), and their current approved retentions.

Approved sapwood product retention / kg/m^3	
Copper-azole, 25 Cu : 1 azole (Tanalith E 3492)	9
Copper-quat, 2 Cu : 1 quat (ACQ 2200 and Celcure AC 500)	12
Copper-HDO, 2.6 Cu : 1 Cu HDO (Wolmanit CX-8)	12

Whilst all three product types have had no reported in service failures worthy of note, Edlund and Jermer (2002) highlighted the potential for some formulations to lose a significant degree of performance when exposed for two years outdoors before being subjected to a decay fungi in an EN113 test. Research undertaken in the FACT (field and accelerated conditioning tests) programme and reported as prCEN/TR 14723 again highlighted the role of primary colonisers in preservative detoxification. Both soil preconditioning and natural weathering were shown to be far more severe for copper-quat than copper-azole. It is understood that CEN TC38 WG25 is currently considering new pre-exposure test methods for EN113, something which has the potential to significantly increase product retentions for some formulations or result in the need for modified or new formulations.

One of the conclusions of the FACT programme was that the range of test fungi in EN113 has to be reconsidered for this method to be relevant for modern biocidal formulations where only climax decay fungi tend to be used which are often less tolerant to biocides than other fungi and bacteria.

The situation for metal-free formulations which are beginning to emerge in some countries is even more confused. Most of the formulations used or approved for use are based on tebuconazole/propiconazole, tebuconazole/propiconazole/IPBC, propiconazole or IPBC which are all inherently biodegradable being mostly derived from the agrochemical industries. Warburton and Hughes (2002) highlighted the potential for bacteria, in the absence of copper, to degrade these active ingredients at potentially an alarming rate but demonstrated that this can be minimised through a greater understanding of formulation chemistry. However, for such products, the NWPC is currently applying large multiplication factors to EN113 test results and in the absence of the preservative manufacturer being able to demonstrate limited depletion/biodegradation for their formulation, they are being approved only for use under a coating. Manufacturers are already addressing this through increased application of formulation chemistry and complexity of formulation design.

ii) Coated

The types of timber generally used in a coated hazard class 3 environment are cladding and joinery, these requiring a strict dimensional tolerance and a good machined finish. Solvent based preservatives are therefore ideal for these applications and the majority of the remaining solvent-based preservatives are used in this end use. Having said that, water-based preservatives are gaining in popularity especially when applied using a superficial process such as controlled immersion and flow coater. These later application methods offer the benefit of the process being a continuous one rather than a batch process.

TBTO in a fast drying solvent was and still is used for this application although this is not being supported through the Biocidal Products Directive (BPD). Hydrocarbon resins and paraffin wax are usually included to provide improved water repellence and paint adhesion. In the UK, zinc soap based preservatives (zinc acepytacs and zinc versatate) were commonly used in the late 1980’s and 1990’s but retarded drying due to the high solids contents. With the increasing use of azoles, it did not take long for these to become the most commonly used actives, either in solvent or water-based emulsions. Whereas azoles provide excellent protection against brown rot fungi, increased retentions are required to protect against white rot (see Table 3). They also have limited protection against bluestain fungi, such as *Aureobasidium pullulans*, and combinations with IPBC or dichlorfluanid are therefore commonly used. IPBC tends to be favoured due to its dual role of providing activity towards staining and basidiomycetes fungi and the fact that it can be formulated in water-based preservatives.

2.4 Hazard class 4

CCA and creosote have dominated this market for many years but as for uncoated hazard class 3, the existing CCA markets are faced with a choice when deciding upon how to respond to the restrictions around arsenic.

In practice, most treaters have or are in the process of converting to copper/organic biocide preservatives. Despite there being derogated end uses for CCA, most treaters have a single treatment cylinder and treat a variety of timbers. Consequently, many of the derogated end uses will be treated with only copper/organic biocide preservatives.

A relatively small number of UK treaters have decided to use CCP and even fewer CC. In the case of CCP, a number of customers have stated that they have made this decision because of the lack of available colour and water repellent additives from their incumbent supplier. The situation is different with copper-chrome, with customers wanting to treat timbers for the treatment of cooling tower timbers. With the likelihood of chromium not being approved for use from 2006, any such moves are short term measures.

All three major product types are currently approved by the Nordic Wood Preservation Council (NWPC) for use in Class A (hazard class 4), their current approved retentions based on a minimum 5 years field testing in accordance with EN252 in two sites being:

Table 5 - Major product types currently approved by the Nordic Wood Preservation Council (NWPC) for use in Class A (hazard class 4), and their current approved retentions.

Approved sapwood product retention / kg/m^3	
Copper-azole, 25 Cu : 1 azole (Tanalith E 3492)	18
Copper-quat, 2 Cu : 1 quat (ACQ 2200 and Celcure AC 500)	36
Copper-HDO, 2.6 Cu : 1 Cu HDO (Wolmanit CX-E 87%)	24

The issue of bio-detoxification is even more important for timbers in-ground than out of ground. This is highlighted by the fact that some preservatives which are approved on only laboratory tests are approved at retentions significantly less (by as much as half) than those above. A number of product standards (e.g. BS EN 12465:2001 for telegraph poles and BS EN 13145:2001 for sleepers) and specifications (e.g. UK Highways Agency Clause 311 specification, November 2002) for utility timbers, where long-term, proven performance is a requirement, specifically make reference to field tests.

Whereas metal-free preservatives are approved and used in uncoated hazard class 3, albeit on a limited scale and in some cases with certain constraints, the task of formulating an in-ground preservative in the absence of copper is extremely challenging especially as very few organic molecules, other than creosote, possess activity towards soft rot fungi.

Forster *et al.* (2002) reviewed work on a new active substance belonging to the group known as oxathiazines, bethoxazin. Research has shown this active ingredient to have a broad spectrum of activity towards both higher and lower fungi, its main activity towards microfungi being towards the bluestain, sapstain and soft-rot group of wood degraders.

Whilst ENV807 and EN252 are accepted test methods for metal-based preservatives, the WOODPOLE (FAIR-CT98-3933) project has highlighted that ENV807 is not an appropriate test method for creosotes, “because of detoxification”. A critical value of creosote of more than 350kg/m^3 would be required when calculated against the CC reference. A modified test method has therefore been proposed. Despite its failure to pass ENV807 at even close to a commercial retention level, creosote is used in HC4 and has had a long history of successful use. However, the fact that a highly effective preservative such as this cannot pass current European standards should act as a warning that such standards require ongoing consideration and review if they are not to hinder, rather than help, necessary development within the wood protection industry.

2.5 Hazard class 5

This is a relatively small market sector representing around only 0.15% of the total timber treated in Europe (Connell, 2004). CCA has been widely used for this end use but this is not a derogated end use and CCA cannot be used for this end use after June 30th 2004. The NWPC have approved the use of CC, CCB and creosote for use in the marine environment. Even if a number of treaters continue to treat with chromium based preservatives after the June 2004 CCA deadline, it is likely that chromium will not be available for use as a wood preservative after 2006.

3 Biocidal Products Directive (BPD), 98/8/EC and its implications

The definition of a wood preservative under the BPD is “Products used for the preservation of wood, from and including the saw-mill stage, or wood products by the control of wood-destroying or wood-disfiguring organisms. This product type includes both preventive and curative products”

Whilst there are still many uncertainties around the BPD, one thing for certain is that there will be fewer active substances available to the wood preserving formulator after 2006. A number of actives on the market today with many years of proven use as wood preservatives, are not being supported. All national product authorisations for these active substances which are not being supported through the BPD have to be withdrawn by September 2006.

Eighty one existing active substances have been notified as wood preservatives (product type 8) to the EU Commission as listed in Annex 5 of the second review regulations (see Appendix 1). The Commission has since received requests for voluntary withdrawal of eight existing active substances, tributyl tin oxide (TBTO), deltamethrin, cyfluthrin, fipronil, ethanol, lactic acid, cetyl pyridinium chloride and tributyl tin napthenate (TBTN) derivatives, and it is likely that there will be several others. Out of the remaining seventy three, it is not known how many will go on to submit dossiers to their Rapporteur member state by the 28th March 2004 deadline and how many are likely to be successful. Assessment of the dossiers is anticipated to be completed by 2006.

Active substances which were not on the European market prior to 14th May 2000 are deemed to be new active substances. The number of applications made for new active substances is not public but as for existing active substances, dossiers will be required. Whilst not public, a number of new actives are being supported and will greatly assist in the development of the next generation of products.

4 Summary

The European timber preservation market has undergone many changes over its history, particularly in the last 15 years. Despite product life cycles getting shorter, a trend which is likely to continue in the future, the timber preservative manufacturers have invested heavily in research and development and embraced this change.

With increasing regulations against metals, greater emphasis is now being placed on the use of organics molecules and new active ingredient suppliers have emerged. Greater emphasis is now being placed on fitness for purpose and aesthetic properties with preservatives often being developed for a single hazard class.

For interior and coated exterior applications, metal-free preservatives offer fit for purpose, cost effective treatments. Preservatives based on copper in conjunction with organics biocides remain the dominant preservative types for exterior, unprotected end uses, either in-ground or above ground and have a proven track record with 10-15 years in use service and similar field test data. The challenge for the industry over the coming years is to reduce its reliance on copper whilst maintaining performance levels with a reduced number of available active ingredients, something which will require a greater understanding and application of formulation chemistry and ecology. The challenge for those who work within standardisation is to consider and review emerging technologies, and produce standards which continue to be fit for purpose.

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Wood preservatives approved by the Nordic Wood Preservation Council 12 January 2004, No. 70.

Appendix 1 - Existing active substances notified as wood preservatives (product type 8) to the EU Commission as listed in Annex 5 of the second review regulations.

Name (EINECS and/or others)	EC Number	CAS Number
Cyclohexylhydroxydiazene 1-oxide, potassium salt	--	66603-10-9
Bis[1-cyclohexyl-1,2-di(hydroxy-.kappa.O)diazeniumato(2-)]-copper	--	312600-89-8
Etofenprox	407-980-2	80844-07-1
Formic acid	200-579-1	64-18-6
Dazomet	208-576-7	533-74-4
Zinc oxide	215-222-5	1314-13-2
Cypermethrin	257-842-9	52315-07-8
Alpha-cypermethrin	Plant protection product	67375-30-8
Propan-2-ol	200-661-7	67-63-0
L-(+)-lactic acid	201-196-2	79-33-4
Hexa-2,4-dienoic acid / Sorbic acid	203-768-7	110-44-1
Calcium dihexa-2,4-dienoate	231-321-6	7492-55-9
Trimagnesium diphosphide	235-023-7	12057-74-8
Aluminium phosphide	244-088-0	20859-73-8
Potassium (E,E)-hexa-2,4-dienoate	246-376-1	24634-61-5
Cyfluthrin	269-855-7	68359-37-5
Margosa ext.	283-644-7	84696-25-3
(E)-1-(2-Chloro-1,3-thiazol-5-ylmethyl)-3-methyl-2-nitro-guanidine	433-460-1	210880-92-5
3-lodo-2-propynyl butylcarbamate	259-627-5	55406-53-6
Tebuconazole	403-640-2	107534-96-3
Thiabendazole	205-725-8	148-79-8
Zinc borate	235-804-2	12767-90-7
Dodecylguanidine monohydrochloride	237-030-0	13590-97-1
Hexafluorosilicic acid	241-034-8	16961-83-4
cis-4-[3-(p-tert-butylphenyl)-2-methylpropyl]-2,6-dimethylmorpholine	266-719-9	67564-91-4
Thiamethoxam	428-650-4	153719-23-4
Naphthenic acids, copper salts	215-657-0	1338-02-9
Lignin	232-682-2	9005-53-2
Ethanol	200-578-6	64-17-5

N-Didecyl-N-dipolyethoxyammonium borate / Didecyl-polyoxethylammonium borate	Polymer	214710-34-6
Copper oxide	215-269-1	1317-38-0
Dicopper oxide	215-270-7	1317-39-1
Copper sulphate	231-847-6	7758-98-7
Oxine-copper	233-841-9	10380-28-6
Copper(II) carbonate-copper(II) hydroxide (1:1)	235-113-6	12069-69-1
Copper dihydroxide	243-815-9	20427-59-2
Flufenoxuron	417-680-3	101463-69-8
Fipronil	424-610-5	120068-37-3
Bifenthrin / Biphenate	Plant protection product	82657-04-3
Tolyfluanid	211-986-9	731-27-1
Propiconazole	262-104-4	60207-90-1
Didecyldimethylammonium chloride	230-525-2	7173-51-5
Quaternary ammonium compounds, benzyl-C12-18-alkyldimethyl, chlorides	269-919-4	68391-01-5
Quaternary ammonium compounds, benzyl-C12-16-alkyldimethyl, chlorides	270-325-2	68424-85-1
Quaternary ammonium compounds, di-C8-10-alkyldimethyl, chlorides	270-331-5	68424-95-3
Quaternary ammonium compounds, benzyl-C12-14-alkyldimethyl, chlorides	287-089-1	85409-22-9
Quaternary ammonium compounds, benzyl-C12-14-alkyl[(ethylphenyl)methyl]dimenthyl, chlorides	287-090-7	85409-23-0
Poly(oxy-1,2-ethanediyl), .alpha.-[2-(didecylmethylammonio)ethyl]- .omega.-hydroxy-, propanoate (salt)	Polymer	94667-33-1
Quaternary ammonium compounds (benzylalkyldimethyl (alkyl from C8-22, saturated and unsaturated, tallow alkyl, coco alkyl, and soya alkyl) chlorides, bromides, or hydroxides) / BKC	Mixture of EINECS listed substances	
Quaternary ammonium compounds (dialkyldimethyl (alkyl from C6-18, saturated and unsaturated, and tallow alkyl, coco alkyl, and soya alkyl) chlorides, bromides, or methylsuphates) / DDAC	Mixture of EINECS listed substances	
Quaternary ammonium compounds (alkyltrimethyl (alkyl from C8-18, saturated and unsaturated, and tallow alkyl, coco alkyl, and soya alkyl) chlorides, bromides, or methylsuphates) / TMAC	Mixture of EINECS listed substances	
Permethrin	258-067-9	52645-53-1
Sodium pentachlorophenolate	205-025-2	131-52-2
Imazalil	252-615-0	35554-44-0

Technical grade imazalil	Plant protection product	73790-28-0
Diboron trioxide	215-125-8	1303-86-2
Disodium tetraborate, anhydrous	215-540-4	1330-43-4
Boric acid	233-139-2	10043-35-3
Disodium octaborate tetrahydrate	234-541-0	12280-03-4
Chlorothalonil	217-588-1	1897-45-6
Fenoxycarb	276-696-7	72490-01-8
N-(3-aminopropyl)-N-dodecylpropane-1,3-diamine	219-145-8	2372-82-9
3-benzo(b)thien-2-yl-5,6-dihydro-1,4,2-oxathiazine,4-oxide	431-030-6	163269-30-5
Esfenvalerate / (S)-.alpha.-Cyano-3-phenoxybenzyl (S)-2-(4-chlorophenyl)-3-methylbutyrate	Plant protection product	66230-04-4
Chlorfenapyr	Plant protection product	122453-73-0
Sulphuryl difluoride	220-281-5	2699-79-8
Creosote	232-287-5	8001-58-9
Deltamethrin	258-256-6	52918-63-5
Iodine	231-442-4	7553-56-2
Bis(tributyltin) oxide	200-268-0	56-35-9
Fenitrothion	204-524-2	122-14-5
Cetylpyridinium chloride	204-593-9	123-03-5
Dichlofluanid	214-118-7	1085-98-9
Diarsenic pentaoxide	215-116-9	1303-28-2
Chromium trioxide	215-607-8	1333-82-0
Sodium dichromate	234-190-3	10588-01-9
2-otcyl-2H-isothiazol-3-one	247-761-7	26530-20-1
Stannane, tributyl-, mono(naphthenoyloxy) derivs.	287-083-9	85409-17-2
Guazatine triacetate	Plant protection product	115044-19-4
Homopolymer of 2-tert-butylaminoethyl methacrylate (EINECS 223-228-44)	Polymer	26716-20-1
Benzothiazole-2-thiol	205-736-8	149-30-4

BIOTRANSFORMATION OF ORGANIC BIOCIDES – LONGEVITY VERSUS ENVIRONMENTAL ACCEPTANCE

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Keywords: Bethoguard, biocide, biotransformation, bacterium

Summary: *The bacterially mediated degradation and detoxification of the new anti-fungal biocide, bethoguard, was studied in vitro, via experiments specifically designed to encourage bacterial biotransformation. Woodblocks, impregnated with sub-toxic levels of bethoguard, were incubated in soil, and the microorganisms associated with detoxification isolated on selective media. It was found that exceptionally high bacterial numbers were present in the wood blocks, and that the associated bacteria were capable of mediating bethoguard degradation. Molecular analyses of the bacterial isolates using 16S rDNA PCR amplification, ARDRA and molecular sequencing, established the identities of the associated bacteria. A large number of Pseudomonas, Alcaligenes and Bordetella strains were found to be dominant in the samples and are thought to play a key role in biocide degradation within the wood. There is therefore a need to study the biotransformation of organic biocides, with view to their stabilisation in wood, to develop the next generation of environmentally benign wood preservatives.*

1 Introduction

In recent years the timber preservation industry has responded to environmental, legislative and economic pressures to utilise more environmentally sensitive formulations. The potential hazards posed by wood preservatives have long been highlighted by regulatory bodies and the Biocidal Products Directive (BPD), introduced by the European Parliament in 1998, is intended to monitor the broad-spectrum toxicity and persistence of biocidal compounds. The directive requires manufacturers to detail the environmental hazards of the active chemicals with particular attention being paid to the ultimate fate of the compounds. In an ideal situation, a candidate biocide should be completely biodegradable, leaving no toxic residues and having zero environmental impact. It is clear that a number of the chemicals used in preservatives need to be replaced and this represents a significant challenge and opportunity to the industry.

The main problem associated with the development of environmentally friendly preservatives based on the newer organic biocides, is the observed biotransformation of these compounds in the wood in service. Activity against the causal decay organisms and resistance to physical losses are no longer the sole performance criteria necessary to understand when developing a new wood preservative. There are several examples of modern organic biocides, currently in use by the wood preservation industry, which are subject to biotransfromation.

IPBC is an organoiodine carbamate fungicide commonly used to control the defacement of softwood timber by sap-staining fungi, in place of the environmentally persistent pentachlorophenol (Xiao & Kreber, 1999). It has been reported that IPBC is degraded when exposed to soils, with members of the Enterobacteriaceae and the proteobacteria mediating IPBC dehalogenation, resulting in loss of fungal toxicity (Cook and Dickinson, 2002; Cook *et al.*, 2002). Bacterially mediated degradation has also been reported for propiconazole in treated wood exposed to soil (Herring, 1999). The problem is to balance the environmental advantages of biotransformation with the required life of the wood product. Understanding the bacterial groups involved, and the mechanisms by which this degradation occurs, is essential if we are to take advantage of the new, environmentally acceptable biocides available to the industry.

Bethoguard is a new metal-free and halogen-free fungicide of a new chemical class. It is being developed for the control of stains, moulds and algae in paints, woodstains, wood preservatives and coatings. A study conducted by Forster *et al.* (2002) revealed the effectiveness of Bethoguard against a variety of soft rot fungi and indicated the potential of this biocide to protect wood exposed in ground contact in the absence of heavy metals. This study is being undertaken to provide detailed information regarding the potential for the microbial degradation of bethoguard, and to provide the basis for understanding and controlling its stability in wood. The initial stages of work consisted of the isolation, and molecular characterisation of the key organisms involved in bethoguard degradation and is the subject of this paper.

2 Materials and Methods

Wood block impregnation, conditioning and soil exposure

The procedures used for the treatment, conditioning and exposure of the wood blocks to soil, were derived from those described in the European standard for determining the toxic effectiveness against soft rotting fungi and other soil inhabiting micro-organisms (prENV807, 2001). Small blocks of scots pine sapwood (15 x10x5 mm) were treated with 500 μM bethoguard (0.2 Kg/m^3), placed on 50 g of John Innes No. 2 compost in 500 ml glass test vessels and covered with a further 50 g of compost. The moisture content was raised to 95% of the water holding capacity, the jars closed with a lid containing a cotton-wool plug, to allow for aeration, and incubated at 25 ˚C and 70% relative humidity. These were deemed to be the conditions that would promote optimal bacterial colonisation and growth.

Wood block analyses

Wood blocks were harvested at weekly intervals and the soil removed from the surface using a small brush. Sawdust was produced from woodblocks using a flame-sterilised saw blade and 1g of the sawdust was suspended in 9 ml of TE buffer (pH 8.0; 10mM Tris-HCl, 1mM EDTA) and shaken at 50 rpm for 12 hours. A dilution series was produced and 100 μl samples were spread, in triplicate, onto a range of selective media.

Selective media and bacterial isolation

The microbiological media were selected to specifically culture a wide range of bacterial groups including those that have been previously implicated in preservative degradation (Cook & Dickinson, 2002; Cook *et al.*, 2002). The groups selected for included actinomycetes, enterics, and the pseudomonads. The media utilised were:

R_2A general heterotrophic medium (Reasoner & Geldreich, 1985)
Starch casein medium for actinomycetes (10 g, soluble starch, 2 g KNO_3, 0.3 g casein, 2 g NaCl, 2 g K_2HPO_4, 0.05 g $MgSO_4$, 0.02 g $CaCO_3$, 0.01 g $FeSO_4$, 1.5% agar, volume to 1 L)
Pseudomonas isolation agar (Difco)
Luria Bertani general heterotrophic medium (1% tryptone, 0.5% yeast extract, 0.5% NaCl, 1.5% agar)

All of the media were supplemented with 200 mM bethoguard in DMSO to give a final concentration of 100 μM. After incubation at 25˚C for 2 weeks, the plates were analysed and the number of bacterial colony forming units per gram of wet wood determined. From these plates, unique bacterial colonies were isolated and developed as pure cultures.

DNA extraction

The DNA extraction was performed using the CTAB/Proteinase K DNA extraction method, as described by Stewart & Via (1993). The extraction is essentially composed of three basic steps: (A) bacterial cell lysis, (B) purification and (C) DNA precipitation.

A loopful of bacterial culture was suspended in 567 μl TE, 30 μl 10% sodium dodecyl sulphate (SDS) and 3 μl 20 mg/ml proteinase K, and incubated at 37^{o}C for 1 hour to rupture the bacterial cells.

100 μl 5 M NaCl and 80 μl CTAB/NaCl solution were added to the eppendorf and incubated at 65^{o}C for 10 minutes to precipitate polysaccharide materials present in the sample. 500 μl of phenol/chloroform/isoamyl alcohol (25:24:1) was then added to the eppendorf, mixed and then

centrifuged at maximum speed for 5 minutes, to precipitate contaminating proteins. The upper aqueous phase was then removed and treated with 500 µl of chloroform:isoamyl alcohol (24:1) to remove any traces of phenol.

The DNA was precipitated overnight at -20°C, through the addition of a 0.1 volume of 3 M sodium acetate and a 0.6 volume of absolute ethanol. The DNA was pelleted by centrifugation at maximum speed for 10 minutes, washed in 200 µl 70% ice-cold ethanol. The DNA pellet was then aspirated and dried under laminar flow, to remove all traces of ethanol. The DNA was then dissolved in 25 µl of TE buffer and a 5 µl sample visualised on a 1.2% agarose gel containing ethidium bromide in 1X TBE buffer (10.8 g Tris (hydroxymethyl) methylamine, 5.5 g boric acid, 0.93 g EDTA) for 30 minutes at 100 V. The gels were visualised under UV transillumination using a Gel Doc 2000 (BIORAD).

16S rDNA PCR amplification

The polymerase chain reaction (PCR) amplification of the 16S rDNA fragment was directed by a primer set which correlated to *E. coli* positions 27-48 (5'-GAG AGT TTG ATC CTG GCT CAG-3'; Sigma-Genosys) and 1495-1477 (5'-CTA CGG CTA CCT TGT GA-3'). The 50 µl reaction mixture for the PCR amplification contained approximately 20 ng template genomic DNA, 25 pmol of each primer, 1U PCR buffer (0.05 M KCl, 0.01 M Tris.HCl pH 9.0, 0.1% Triton-X), 2.5 mM $MgCl_2$, 200 µM dNTP's and 1U of *Taq* DNA polymerase.

The PCR amplification consisted of an initial denaturation step at 95°C for 5 minutes, followed by 25 cycles of 1 minute at 95°C, 1 minute at 50°C and 1 minute at 72°C, with a final extension step at 72°C for 10 minutes in a MJ Research PTC-200 Peltier thermal cycler (Genetic Research Instrumentation). The products were then analysed on 1.2% agarose gels to confirm the presence of a single product of the desired size.

Amplified ribosomal DNA restriction analysis (ARDRA)

The 16S rDNA was digested separately with *Taq*I and *Dde*I restriction endonuclease enzymes (Sigma) using the conditions outlined by the manufacturer. Approximately 40 ng of the PCR products were digested for 2 hours using 1 U of restriction enzyme, at 37°C for *Dde*I and 65°C for *Taq*I in a MJ Research PTC-200 Peltier thermal cycler. The digested PCR products were visualised on 1.2% agarose gels as outlined, the band sizes determined by comparison against a 1 Kb DNA ladder (Promega) and used to produce a binary matrix, representing the bands present in each sample. This "matrix" was then used to determine sequence diversity using the TREECON phylogenetic software package (Van de Peer and de Wachter, 1994).

Sequence analysis

Representative isolates were selected from the ARDRA phylogenetic tree, the PCR products purified using the Minielute™ PCR purification kit (QIAGEN), as described by the manufacturer, and sequenced by ABC sequencing, Imperial College, using the Big Dye dideoxy terminal sequencing kit (ABI; Rosenblum *et al.*, 1997). Sequence chromatographs were analysed for quality using CHROMAS v1.45 and species identification using the BLASTn search software of the National Centre of Biotechnology Information (NCBI; http://www.ncbi.nlm.nih.gov/BLAST/). The sequences were then aligned using CLUSTALX v1.81 (Thompson *et al.*, 1997) and a phylogenetic tree was produced using the TREECON software.

Antibiotic assay test

Three 13mm Whatman antibiotic assay discs were placed onto 2% malt agar plates, in triplicate, for use in a biological assay. 50 µl of bethoguard-containing LB broth, which had been exposed to the bacterial isolates for 48 hours, were applied to each of the discs and allowed to air-dry. Addition of 25 µl of a 1% tetracycline sulfate solution (VWR) to each of the assay discs prevented the growth of the test bacteria, which would distort the bioassay. A spore suspension of *Aspergillus niger* in 10 ml of a 2% sodium dioctyl sulfosuccinate was sprayed onto each test plate. The plates were incubated at 25°C for 14 days, and the diameter of any zone of inhibition in the fungal lawn measured through the centre of the assay discs (Cook *et al.*, 2002).

3 Results

Very large bacterial numbers were cultured from the bethoguard impregnated wood exposed to whole soil, as shown in figure 1. The pattern of colonisation was characterised by very high initial bacterial numbers, which increased gradually over time. It is thought that the large initial population was a consequence of water being absorbed into the wood, carrying with it a microbial inoculum from the soil. A number of selective media were utilised to estimate the bethoguard degrading population and it should be remembered that the growth of a specific bacterial group was not restricted to a single selective medium. Pure cultures of all bacterial colonies were established for further analysis (see table 1 and figure 2 for identities).

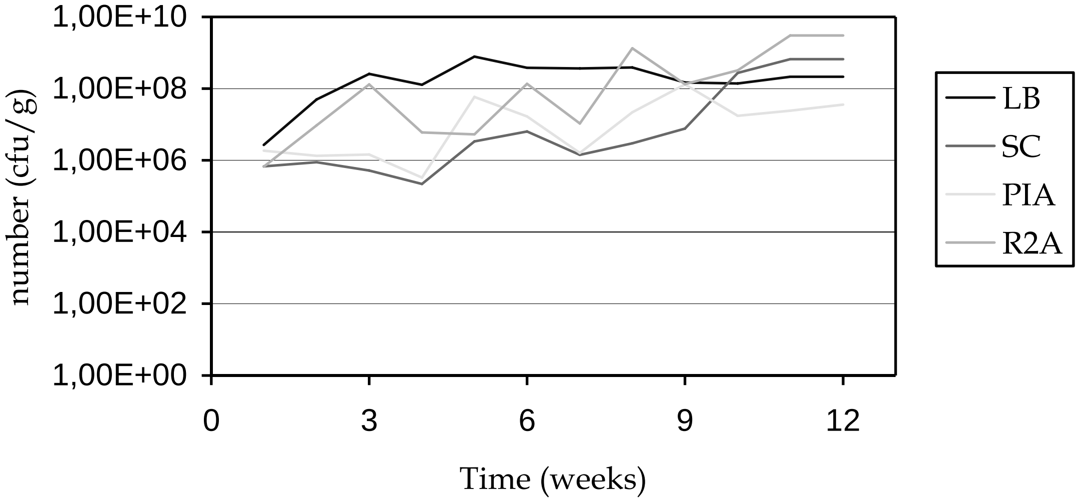

Figure 1: Observed bacterial numbers cultivated from bethoguard impregnated wood exposed to soil

Luria Bertani Agar

Luria Bertani agar (LBA) is a nutrient rich heterotrophic growth medium, and use of this medium estimated that the microbial load of bethoguard impregnated wood after 1 week was 3 x 10^6 cfu/g wet wood. Microbial numbers were found to increase until a maximum was reached at week 5, of 8 x 10^8 cfu/g wet wood. After this, it was found that bacterial numbers decreased slowly with time, giving a count of 2 x10^8 cfu/g wet wood at week 12.

Of all the media that were used in this study, LBA resulted in the greatest diversity of microorganisms. Bacteria isolated from this medium included *Bordetella petrii*, a number of pseudomonad species, *Denitrobacter permanens, Variovorax paradoxus* and *Alcaligenes xylosoxidans*.

R_2A

R_2A is an oligotrophic growth medium, differing from LBA in that it contains low levels of carbon and nitrogen, and will thus support a different microbial population. R_2A agar gave an initial estimate of bacterial numbers of 7 x 10^5 cfu/g wet wood. The number of bethoguard resistant bacteria then increased steadily with time and was at a maximum at week 12. At this time, the bacterial population was found to be 3 x 10^9 cfu/g wet wood and increasing. Organisms isolated from this medium were found to belong to the pseudomonads.

Table 1: Phylogenetic identities of the bethoguard-degrading bacterial isolates

Culture	Identity	Similarity (%)
F15	*Pseudomonas fluorescens*	99
F7	*Pseudomonas putida*	99
F8	*Bordetella petrii*	98
C1	*Denitrobacter permanens*	97
B2	*Pseudomonas citronellolis*	99
F11	*Pseudomonas putida*	95
A2	*Alcaligenes*	97
F14	*Pseudomonas putida*	97
A9-1A	*Ralstonia taiwanensis*	98
C5-2	*Alcaligenes*	96
F4	*Bordetella petrii*	99
B4	*Alcaligenes xylosoxidans*	100
D1	*Achromobacter xylosoxidans*	93
F20	*Variovorax paradoxus*	99

Pseudomonas Isolation Agar

Use of *Pseudomonas* isolation agar (PIA) allowed enumeration of this genus of bacteria, which is typically found in large numbers in soil (Holt *et al.*, 1994) and also implicated in biocide degradation (Cook *et al.*, 2002). Initial pseudomonad numbers were estimated at 2 x 10^6 cfu/g wet wood, and this population gradually increased with time to a maximum of 1.3 x 10^8 cfu/g wet wood at week 9, after which the population begins to fall. Phylogenetic analysis of the bacterial isolates identified three different *Pseudomonas* species: *Pseudomonas fluorescens*, *Pseudomonas putida* and *Pseudomonas citronellolis*. Notably, *Pseudomonas fluorescens* and *Pseudomonas putida* had been isolated using LBA and R_2A.

Starch Casein Agar

The final medium utilised was starch casein agar (SCA), commonly used for the cultivation of actinomycetes. Bacterial numbers were initially estimated at 7 x 10^5 cfu/g wet wood, with this number staying constant until week 7, after which numbers increased rapidly to a maximum of 6.7 x 10^8 at week 12. Colonies observed on this medium were deduced not to be actinomycetes on the basis of microscopic examination. Colonies were selected for pure culture and then analysed by means of 16S rDNA phylogenetic analysis, and all isolates were identified as being *Pseudomonas fluorescens*.

Bethoguard detoxification

The identified bacterial isolates were exposed to low-levels of bethoguard (100 µM) in LB broth to determine the extent and rate of biocide degradation. The antibiotic disc assay was then used to determine the loss of fungal toxicity of the bethoguard solution after 48 hours, as described. In all cases, the *Aspergillus niger* was uninhibited in growth and grew as a lawn over the entire plate and discs. This assay shows that the bacteria isolated in this study are capable of mediating the biological detoxification of bethoguard.

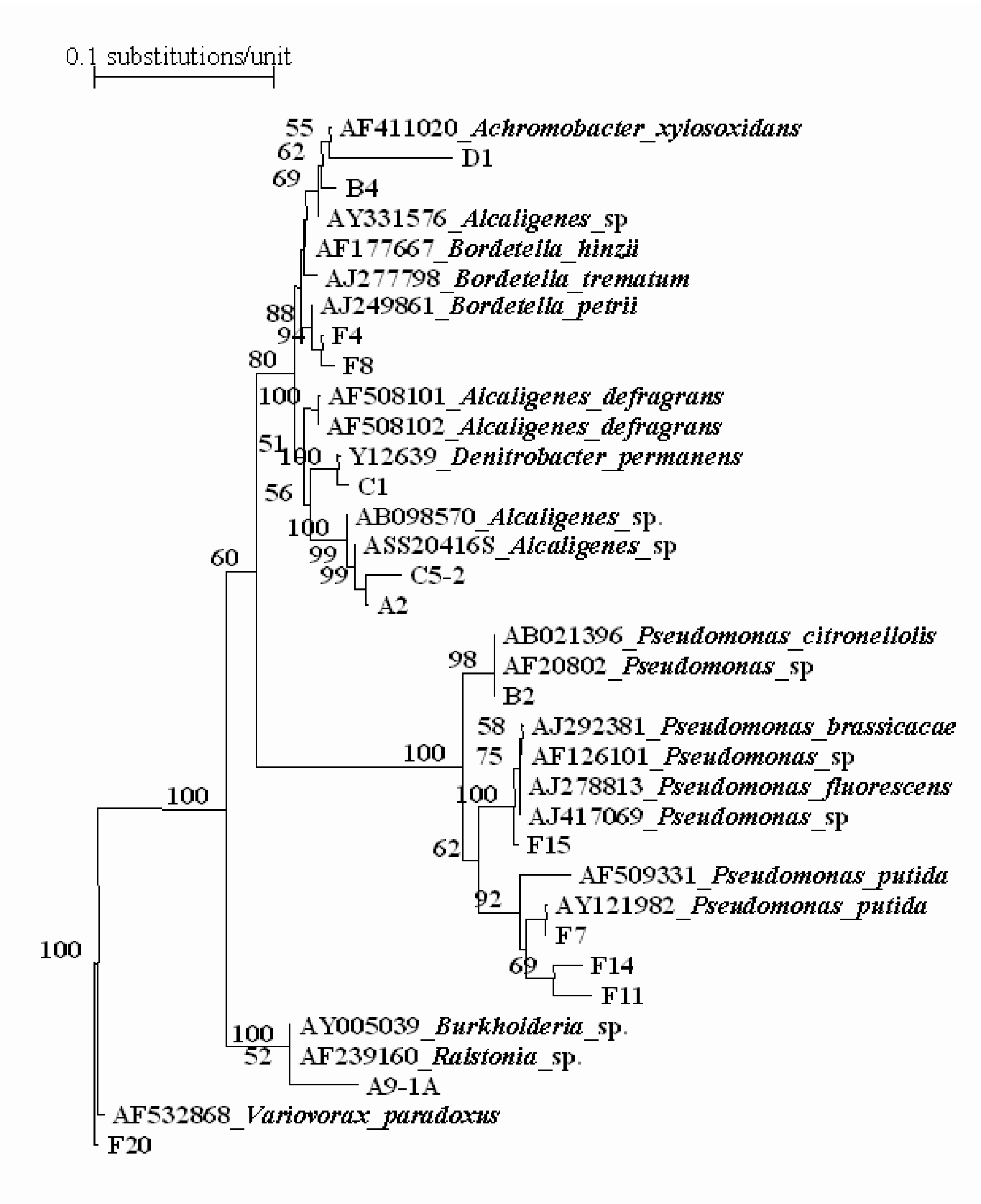

Figure 2: Phylogenetic inference of the identity of the studied bethoguard-degrading bacterial isolates

4 Discussion

It was the aim of this study to isolate and characterise the micro-organisms which showed an ability to degrade the anti-fungal compound bethoguard. The study utilised an aqueous based bethoguard formulation, and did not include co-biocides or co-formulants that would improve preservative performance (Forster *et al.*, 2002). To promote the colonisation of the wood blocks by bacteria, subtoxic levels of bethoguard were utilised, representing wood at the end of service-life, along with an incubation temperature of 25ºC. This study does not therefore provide an evaluation of the performance of bethoguard as a wood preservative (see Forster *et al.*, 2002), but provides a comprehensive analysis of the degrading potential of soil-inhabiting bacteria.

A very large bacterial population capable of bethoguard detoxification was found to be associated with the bethoguard impregnated miniblocks exposed to soil. Use of a number of different selective media were utilised to culture bacterial isolates from as wide a range of microbial genera as possible. In particular, attempts were made to culture the bacterial genera previously identified as being involved in biocide degradation, such as those bacteria involved in the degradation of propiconazole and IPBC (Cook & Dickinson, 2002). As such, a number of selective media were employed to culture the actinomycetes (SCA) and the pseudomonads (PIA), alongside two different heterotrophic media (LBA and R_2A), which should be suited to the culturing of bacteria from all genera. The inclusion of 100 µM bethoguard in these media ensured that only biocide tolerant or degrading bacteria could be cultured, as the bethoguard exerts a selective pressure.

From these plates, unique bacterial colonies were selected and developed as pure cultures, for use in bethoguard degradation studies. In total, around 60 pure cultures were established and a means by which the diversity of these isolates could be established was required. Instead of using classic microbiological techniques, which are extremely time consuming and labour intensive, a molecular approach was utilised. This approach consisted of the amplification of the 16S rDNA from each of the pure cultures, and the subsequent analysis of the nucleotide sequences. The analytical technique adopted was amplified ribosomal DNA restriction analysis (ARDRA) a powerful technique for differentiating between large numbers of nucleotide sequences, based on the cutting of the DNA strand by restriction endonuclease enzymes, in this case *Taq*I and *Dde*I.

Sequence analysis revealed that the bacterial isolates were dominated by the pseudomonads (α-proteobacteria), with *Pseudomonas citronellolis*, *Pseudomonas putida* (two strains) and *Pseudomonas fluorescens* identified. These pseudomonad strains were cultured from a number of the different selective media, including PIA, LB and SC, and thus contributed to the estimated bacterial populations obtained from these media. This has resulted in repeated isolation of these bacteria, with the pure culture library thought to contain 30 pseudomonad isolates, which represents 54 % of the total isolates. The use of PIA estimated that there was 1.3×10^8 pseudomonad cfu/g wet wood, illustrating the abundance of these metabolically diverse organisms.

Pure culture studies revealed that these pseudomonad isolates showed a great capacity for detoxification of low levels of bethoguard, with degradation of the biocide within 48 hours. This is further evidence to the capacity of the pseudomonads to degrade biocides, as *Pseudomonas fluorescens* was also identified as being capable of mediating the dehalogenation of IPBC (Cook & Dickinson, 2004; Cook & Dickinson, 2002; Cook *et al.*, 2002). The capacity of the pseudomonad isolates to degrade higher concentrations of bethoguard has not yet been evaluated.

Sequence analysis of the 16S rDNA PCR products also showed that a number of the isolates were very closely related to *Bordetella petrii*, a β-proteobacterium. This was the first time that *Bordetella* has been implicated in biocide degradation and was a significant observation. This microbe was found only to grow on LBA, but contributed significantly to the total number of bacteria observed on this medium. Experimentation using the pure culture of this organism showed that this isolate was capable of mediating bethoguard degradation and detoxification, although the mechanism is unknown and is subject to further study.

A number of *Alcaligenes* species (β-proteobacterium, *Bordetella* group) were also identified from the pure culture isolates. Of these, two did not align with any certainty to a defined *Alcaligenes* species and may represent a new species, a third aligned with *Alcaligenes xylosoxidans* and a fourth aligned with *Achromobacter xylosoxidans*, a species which represents a further classification of *Alcaligenes xyloxidans*. This group of organisms were isolated from a number of different selective media, and it is thought that these bacteria may make up a significant percentage of the bethoguard degrading population. Pure culture analysis showed that these bacteria were able to mediate bethoguard degradation and detoxification in liquid media, determined using the antibiotic assay. *Alcaligenes* was also implicated in the degradation of IPBC, showing that these microbes are capable of mediating the detoxification of a number of organic biocides (Cook & Dickinson, 2002). The property of biocide degradation appears to be widespread amongst the proteobacteria, specifically the *Bordetella* group of the β-proteobacteria. This was further exemplified by the identification of *Ralstonia taiwanensis, Variovorax paradoxans* and *Denitrobacter permanens* amongst the bacterial isolates.

From the phylogenetic studies, it was observed that all of the identified bacterial isolates are members of the proteobacteria. This suggests that the property of bethoguard degradation is wide-spread in this grouping of microbes, and may represent a property of the common ancestor. If a single mode of degradation is employed by all of the isolates, then there is a great chance that the preservative can be stabilised by interference of this pathway. Studies of the mode of bethoguard degradation by the isolates are currently being carried out.

5 Conclusion

This study has established that soil-inhabiting bacteria have the potential to degrade and detoxify the anti-fungal biocide bethoguard. Use of conventional microbiological techniques identified that the α- and β-proteobacteria are implicated in this degradation, in particular the pseudomonads, *Alcaligenes* and *Bordetella*. Pure culture analyses have established that these isolates were capable of detoxifying a 100 µM bethoguard solution *in vitro* within 48 hours. Only through the study of bacterial degradation can we predict and manipulate the bioeffective life of bethoguard, leading to the most efficient use of new organic biocides.

6 Acknowledgements

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PENETRATION AS KEY FACTOR FOR DETERMINING THE PERFORMANCE OF WOOD PRESERVATIVES

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Abstract: *Stakes of Scots pine sapwood with a dimension of 4 x 4 x 75 cm³ were impregnated with preservatives containing 2 % of propiconazole which had passed the EN 113 test criteria. The penetration of the colourless preservative was determined by a fungal screening test described by Sutter (1978). The test revealed an insufficient penetration behaviour within the given cross section (4 x 4 cm²) although the preservative uptake of approx. 610 kg/m³ indicated a full penetration.*

1 Introduction

A number of methods are available for the efficacy testing of preservatives. Most of them pretend an even distribution of the preservative within the entire test specimen and in most cases this can be seemingly achieved in the specimens used e.g. in screening tests or in EN 113. Therefore, it is unusual to check the penetration and distribution of the test preservative in detail as long as there is no indication that the penetration might be insufficient. However, the preservative may perform well in the laboratory tests if a minimum penetration of 8 mm (half of the width of a specimen) or a perfect shell treatment is achieved. This might happen when a vacuum impregnation is applied and if the active ingredients are fixed immediately to wood substances or if the nature of the wood causes a chromatographic effect during impregnation.

The penetration of preservative formulations with a distinct colour (like those containing chromium) can be checked easily at cross or longitudinal sections of a specimen. However, the visual assessment cannot be applied to colourless preservatives nor does it provide any information about the efficacy of the preservative in a distinct depth. Spot test on the surfaces of freshly cut specimens might help in many cases, however, most of the organic active ingredients are colourless and are applied in low concentrations that spot test might fail to detect the active ingredient.

Therefore, a test was performed to investigate the suitability of a simple biological test proposed by Sutter (1978) to explore the penetration behaviour of colourless active applied by a surface treatment.

2 Test methods

A number of water based preservative formulations which are known to perform well in screening tests and likewise in EN113 tests were applied to Scots pine sapwood (end sealed sticks of 4 x 4 x 75_{axial} cm^3) in a vacuum-pressure treatment process (2% preservative in tap water, 1h pre-vacuum <5kPa, 6h pressure 0,8 Mpa, 0,25h post-vacuum <5kPa, see also prCEN/TC 14734). Prior to end-sealing a block of 5 cm in length was cut from each stick which was used to determine the wood moisture content (by oven drying) and the individual density (at 12% wood moisture content). The sticks were weighed before and after impregnation for calculating the uptake of treatment solution. The treated sticks were stored one week for fixation, then cut to slices of 4 x 4 x $0{,}5_{axial}$ cm^3 and subsequently dried for another week in the laboratory. The specimens were soaked with demineralised water according to EN 84, leached for 4 days (4 times replacement of the water) and dried for 2 weeks in the laboratory. After sterilisation (gamma radiation, (Co_{60}) approx. 26kGy) the specimens were exposed to an attack by the following wood destroying fungi: *Coniophora puteana* (BAM Ebw. 15), *Gloeophyllum trabeum* (BAM Ebw. 109), *Oligoporus placenta* (FPRL 280), *Coriolus versicolor* (CTB 863 A).

The specimens were transferred into Petri dishes (diameter 89 mm) containing 20 ml malt agar (2% agar agar, 4 % malt extract powder) which was completely overgrown by the test fungi. A plastic support (height: approx. 2 mm) was placed between specimen and the mycelium mat.

The penetration of the preservative was determined by assessing the presence of surface mycelium on the specimens as well as by the visible decay on the wood surface after oven drying.

3 Results

The mean preservative uptake was approx. 610 kg/m^3 corresponding to a mean preservative retention of 12,2 kg/m^3 (min: 10,7 kg/m^3, max: 12,9 kg/m^3, see Table 1). Considering the density of the stakes used (0,52 g/cm^3 - 0,65 g/cm^3), the retention indicated a full penetration of the whole stake. This assumption was supported by the observation that all specimens cut after one week were still wet in the centre.

The growth behaviour of the test fungi as well as the decay pattern showed that the preservative was not evenly distributed over the cross section. In many cases the fungi were able to grow through the centre of the specimens also causing mass loss. This was observed not only for the most tolerant test fungus *G. trabeum* but also for the less agressive fungi *O. placenta* and *C. versicolor* (see figures 1 - 10).

4 Conclusions

The test proposed by Sutter (1978) was easy to handle and performed well even with the pressure treated timber. However, an evaluation based only on the assessment of the surface mycelium might cause misleading results. However, this can easily be compensated by evaluating the visible decay pattern as well as the mass loss data. It should be mentioned that *Coriolus versicolor* was less suitable for this method, since its white rot decay pattern was hardly visible on the surface of the specimens. Thus, for *C. versicolor* it was possible to distinguish between full penetration (no mass loss detectable and no surface mycelium) and penetration less than 2 cm (mass loss detectable and break out of mycelium in the middle of the specimens), but it was impossible to determine a distinct depth of penetrationof the active ingredient.

Table 1: Density, preservative uptake and retention of the test stakes.

Test preservative	wood species	stake no.	Density [g/cm³]	preservative uptake [kg/m³]	Retention [kg/m³]
010	Scots pine	4-0	0,54	584	11,7
		4-1	0,52	621	12,4
		4-2	0,57	603	12,1
		4-3	0,52	628	12,6
		4-4	0,52	622	12,4
		mean	0,53	612	12,2
047	Scots pine	6-0	0,58	608	12,2
		6-1	0,52	644	12,9
		6-2	0,57	631	12,6
		6-3	0,54	633	12,7
		6-4	0,65	533	10,7
		mean	0,57	610	12,2

Although the preservatives used in these experiments had shown good effectiveness in screening tests as well as in an EN 113 test (see Table 2), the penetration pattern in slightly larger specimens seems to reveal a weak spot of these formulations. The penetration from the surface into the wood was often less than 2 cm and consequently the centre of the specimens (cross section: 4 x 4 cm²) could be deteriorated. This was observed not only for the tolerant fungus *G. trabeum* for which the EN 113-test indicated an effective retention between 12 kg/m³ and 19 kg/m³, but it was also true for the less tolerant fungi *O. placenta* and *C. versicolor* which were not able to deteriorate specimens in the EN 113-test with a preservative retention ≥ 7 kg/m³.

It can be concluded from the resultsthat the small EN 113-specimens were either perfectly shell treated or fully penetrated. A full penetration of small specimens is suitable for determiningthe effective retention. However, testing cross sections of larger stakes focuses on the penetration performance under more realistic conditions. The results provide important information whether a non-durable sapwood is still chemically protected when the surface layers are damaged e.g. in cases of severe cracking.

In practice a minimum penetration of 20 mm or a full penetration of the sapwood is required predominantly (e.g. specification acc. EN 351-1: P7, P8, P9).

In the test described here, the indirect assessment of uptake and penetration (determination of the preservative uptake and visual assessment that the whole specimen was soaked with liquid), did not provide sufficient information about the real penetration of the active ingredient. This observation supports the requirement outlined in EN 351-1 7.2.2: *Indirect testing: " … When these features are used for routine quality assessment, the correctness of the relationship used shall be examined at certain intervals."* at least for such preservatives which contain colourless active ingredients.

Table 2: Effective retention of the formulations 10 and 47 determined in EN 113 tests after ageing acc. to EN 84 and EN 73

Test preservative	Test applied	effective retention [kg/m³] (mass loss of the virulence controls)				Brv
		C. puteana	*G. trabeum*	*O. placenta*	*C. versicolor*	
10	EN 113	11,9 – 18,9 (42%)	7,7 – 12,0 (47%)	4,8 – 7,6 (31%)	3,0 – 4,8 (25%)	11,9 – 18,9
	EN 84/EN 113	7,4 – 12,2 (37%)	12,1 – 19,1 (49%)	4,9 – 7,5 (37%)	4,8 – 7,5 (17%)	12,1 – 19,1
	EN 73/EN 113	7,7 – 12,2 (47%)	7,7 – 12,3 (39%)	4,8 – 7,6 (33%)	3,1 – 4,8 (23%)	7,7 – 12,3
47	EN 113	7,5 – 11,7 (34%)	7,4 – 11,5 (41%)	4,7 – 7,4 (25%)	< 1,6 (20%)	7,5 – 11,7
	EN 84/EN 113	7,3 – 11,3 (39%)	11,3 – 18,4 (31%)	4,6 – 7,3 (33%)	2,5 – 4,0 (31%)	11,3 – 18,4
	EN 73/EN 113	7,4 – 11,5 (52%)	7,5 – 11,6 (29%)	4,5 – 7,4 (27%)	-	7,5 – 11,6

The test proposed by Sutter gave a good indication of the penetration of the preservative tested and may be a suitable tool to check the correlation between preservative uptake and penetration behaviour of the active ingredients at least for those preservatives which are very difficult or expensive to analyse in treated timber.

5 Acknowledgement

We would like to thank Marie-Therese Lenz (BFH), Kerstin Klutzny (BAM) and Wilma Mierke (BAM) for their practical work and their ideas to improve the experimental design. The study has been carried out with financial support from the Federal Ministry of Education and Research of Germany (BMBF).

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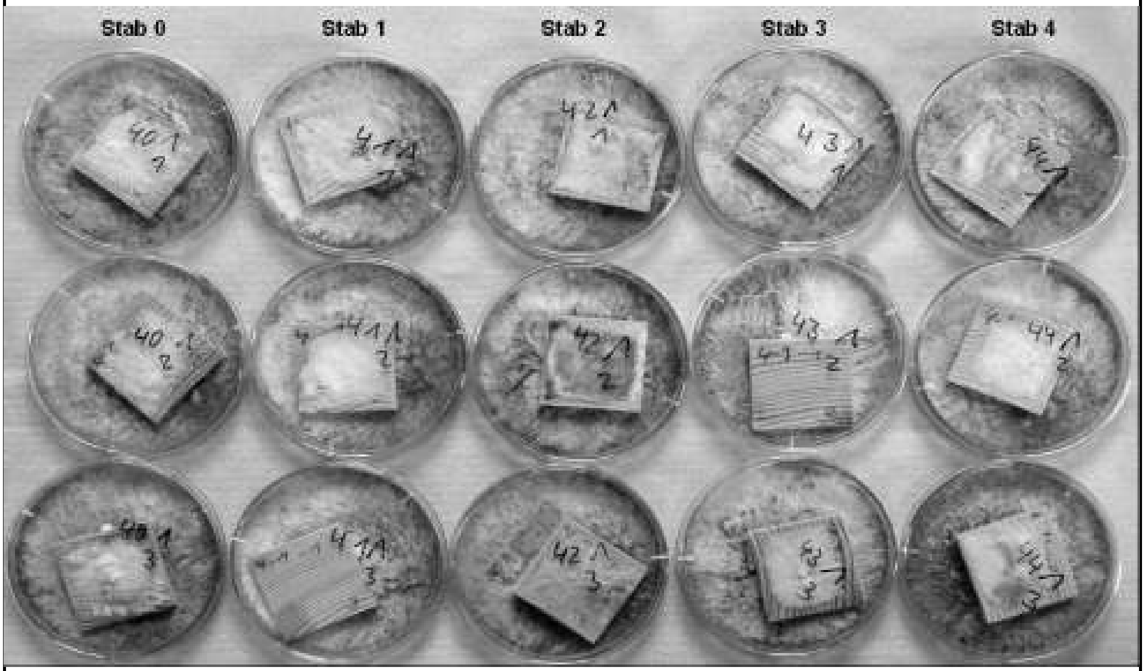

Figure 1: Surface mycelium of *C. puteana* after 6 weeks of incubation on crosscut section of stakes treated with preservative 10 (5 stakes, 3 replicates per stake)

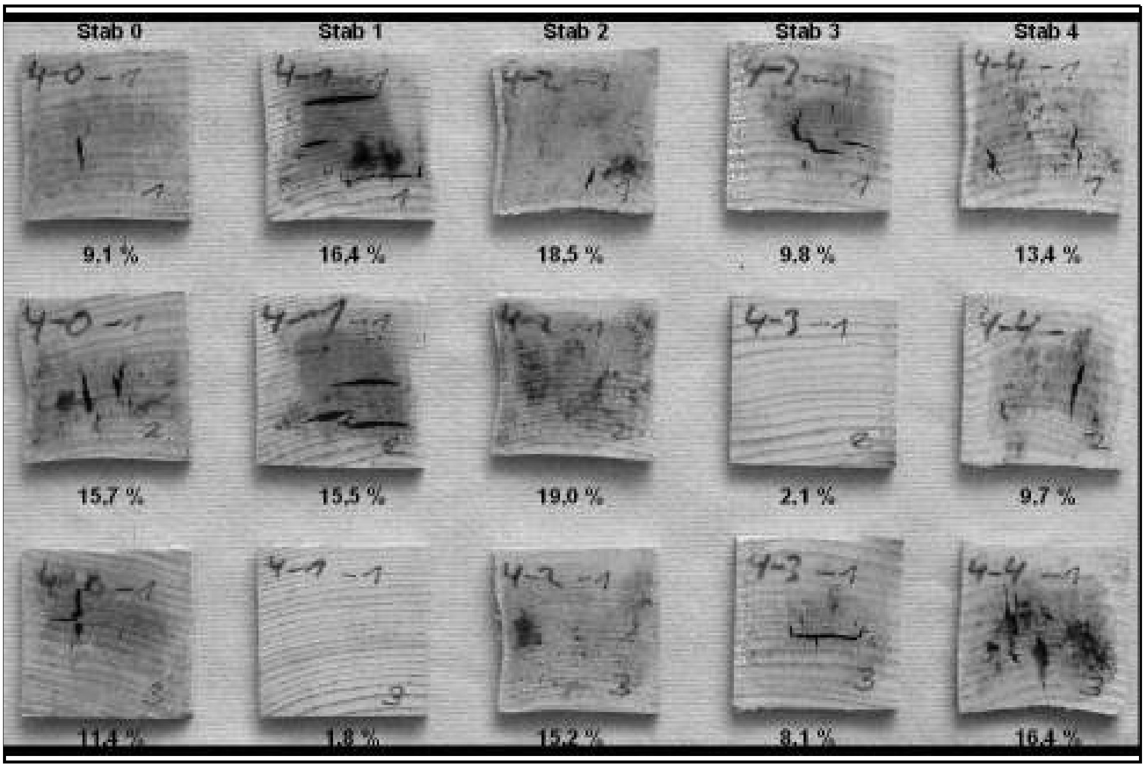

Figure 2: Pattern of decay on cross-cut sections of stakes treated with preservative 10 (5 stakes, 3 replicates) after incubation with *C. puteana* (mass loss given below the specimen).

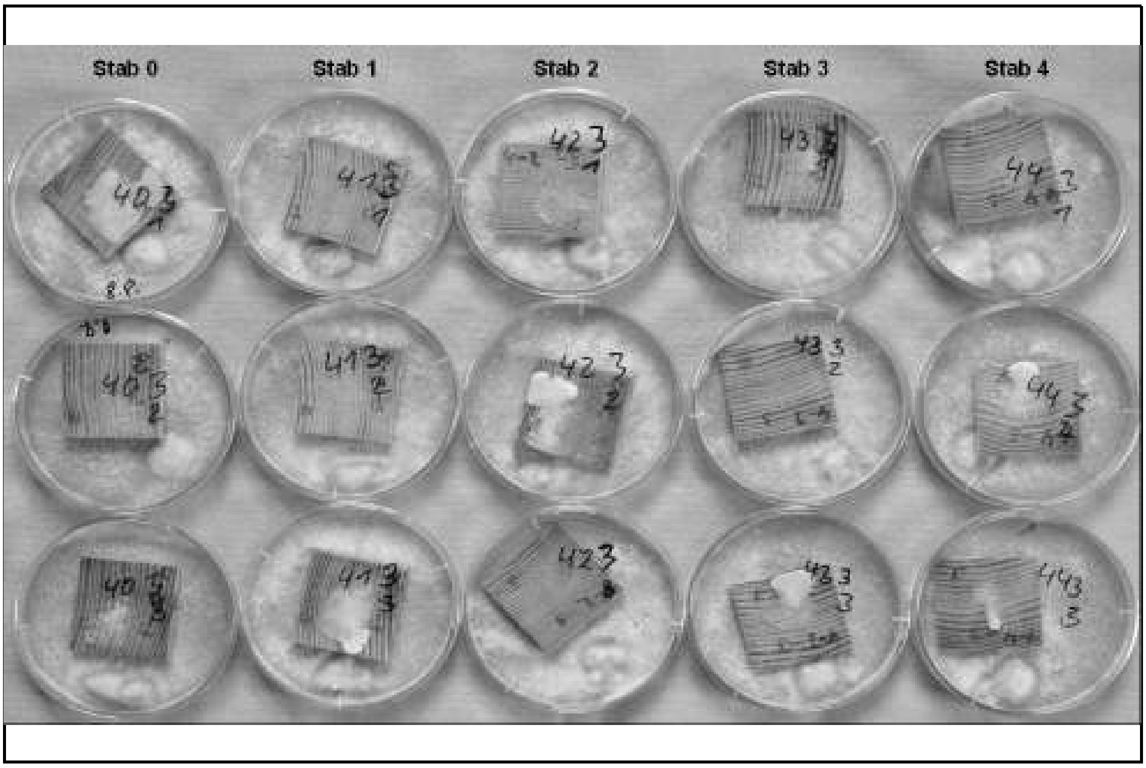

Figure 3: Surface mycelium of *G. trabeum* after 6 weeks of incubation on cross-cut sections of stakes treated with preservative 10 (5 stakes, 3 replicates per stake)

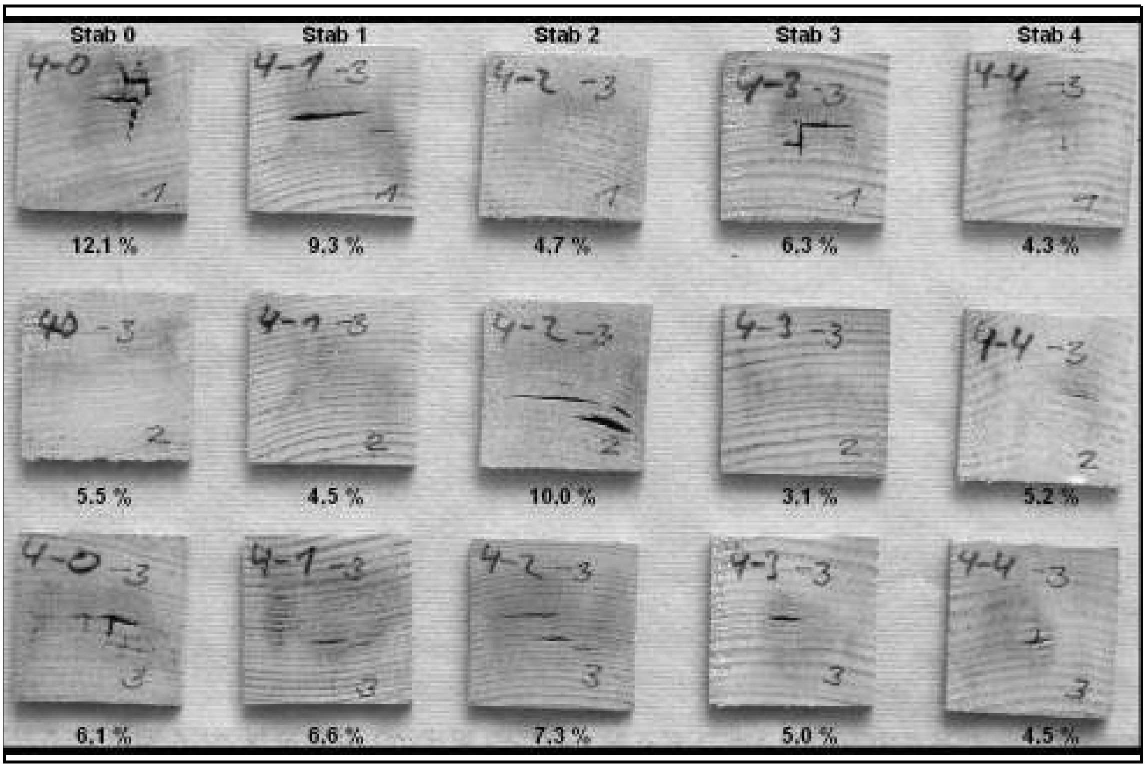

Figure 4: Pattern of decay on cross-cut sections of stakes treated with preservative 10 (5 stakes, 3 replicates) after incubation with *G. trabeum* (mass loss given below the specimen).

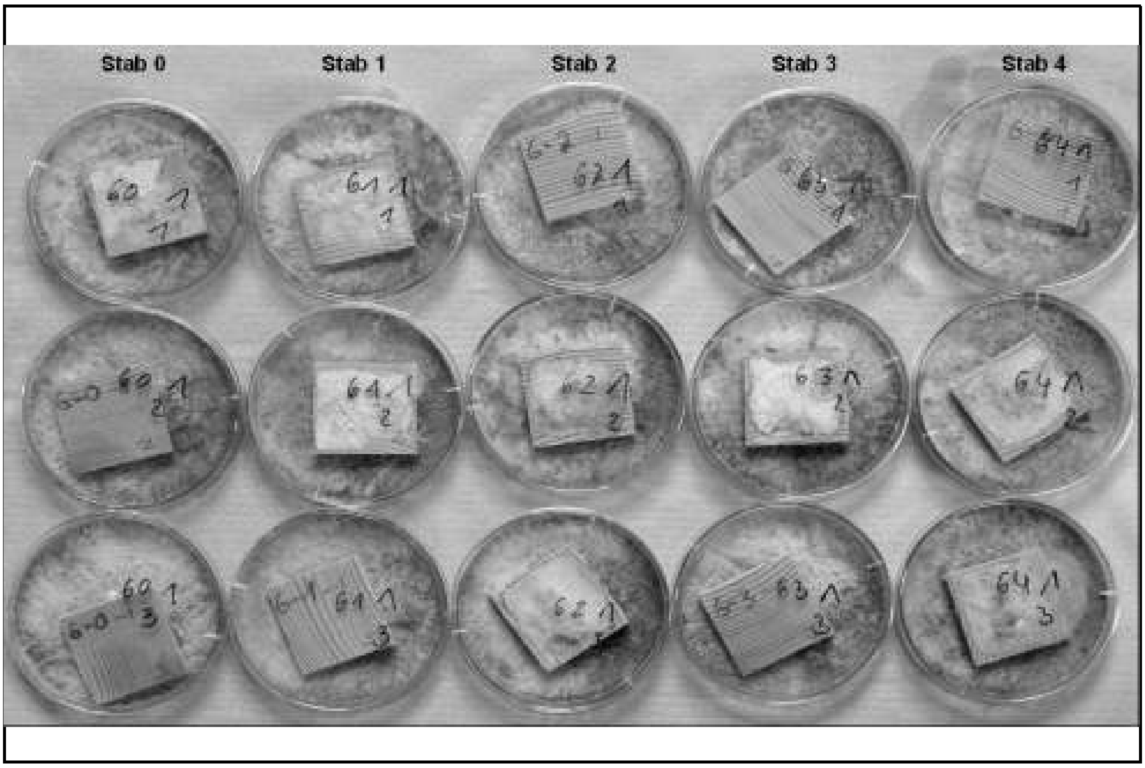

Figure 5: Surface mycelium of *C. puteana* after 6 weeks of incubation on cross-cut sections of stakes treated with preservative 47 (5 stakes, 3 replicates per stake)

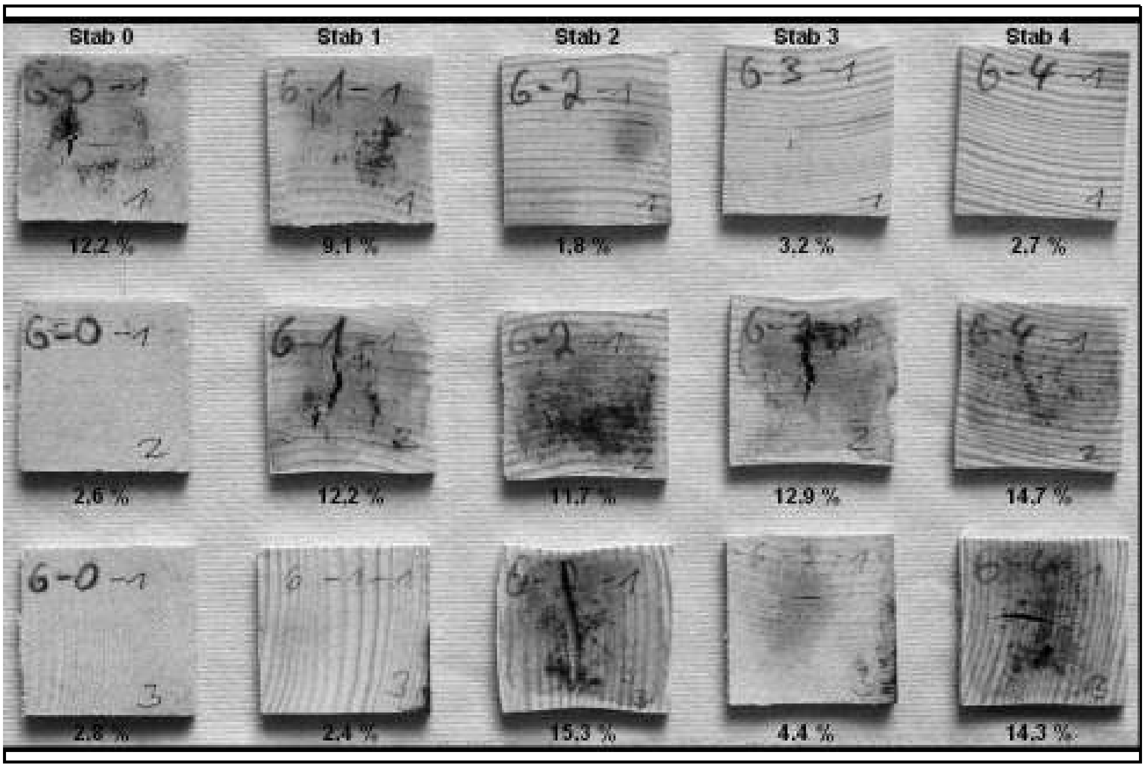

Figure 6: Pattern of decay on cross-cut sections of stakes treated with preservative 47 (5 stakes, 3 replicates) after incubation with *C. puteana* (mass loss given below the specimen).

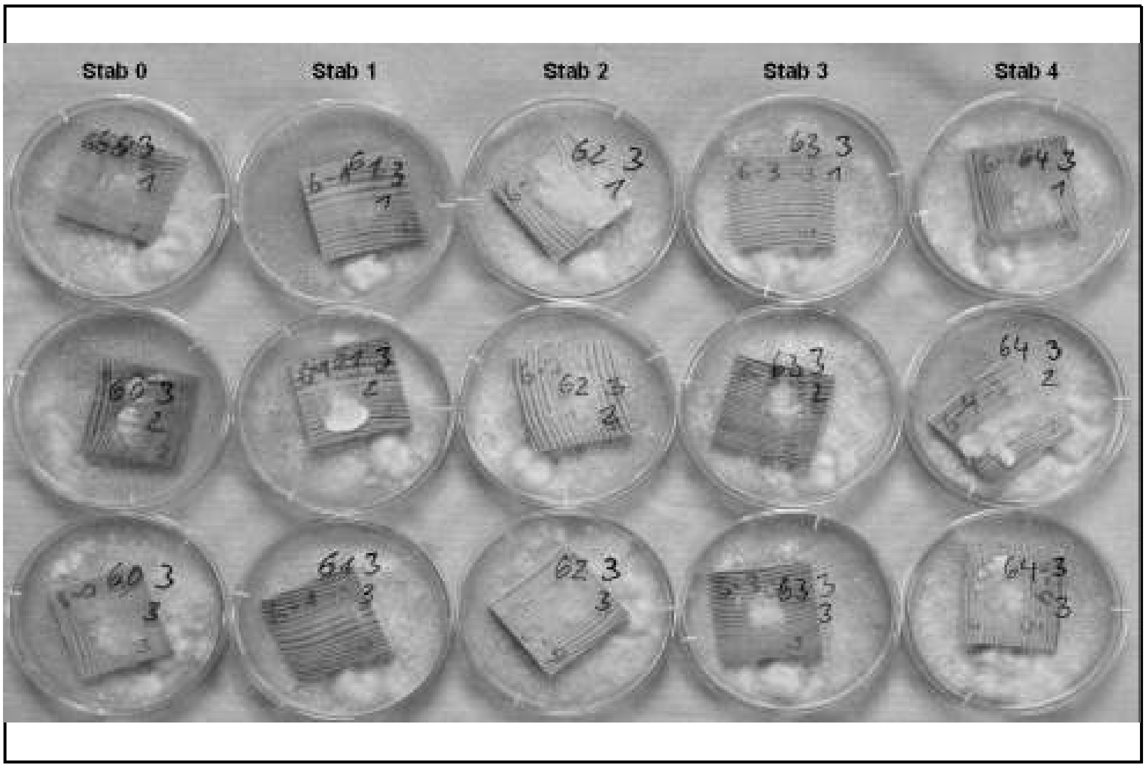

Figure 7: Surface mycelium of *G. trabeum* after 6 weeks of incubation on cross-cut sections of stakes treated with preservative 47 (5 stakes, 3 replicates per stake)

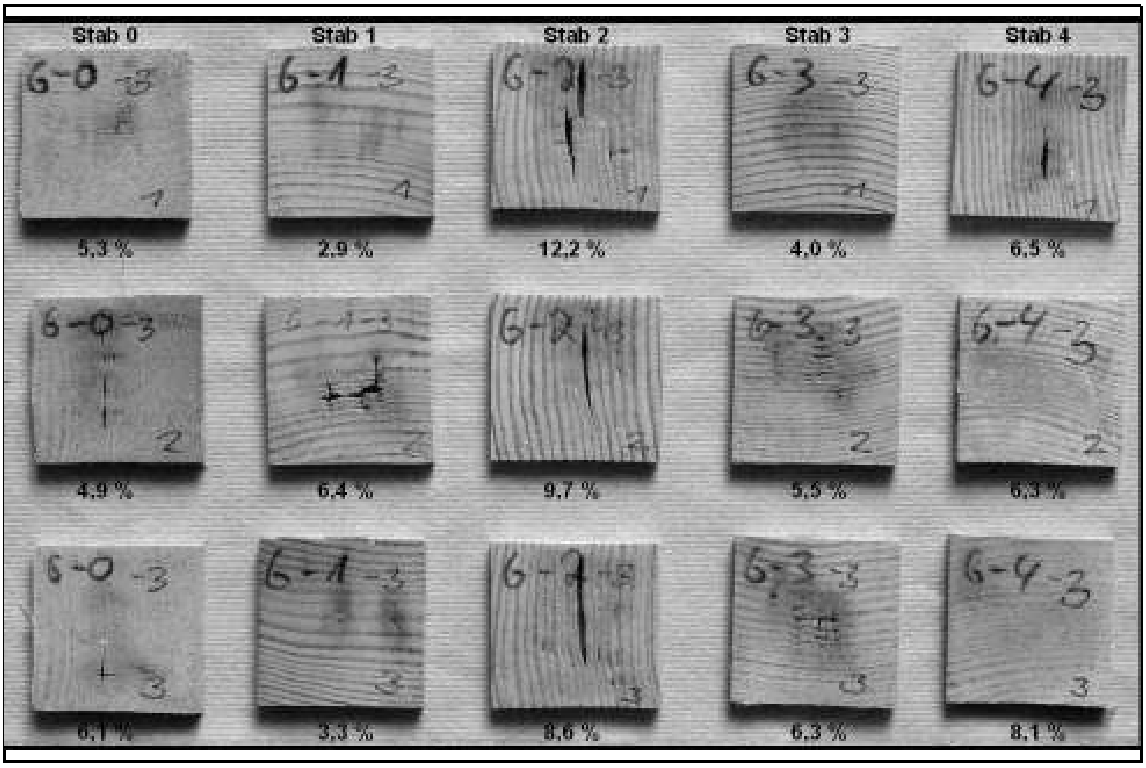

Figure 8: Pattern of decay on cross-cut sections of stakes treated with preservative 47 (5 stakes, 3 replicates) after incubation with *G. trabeum* (mass loss given below the specimen).

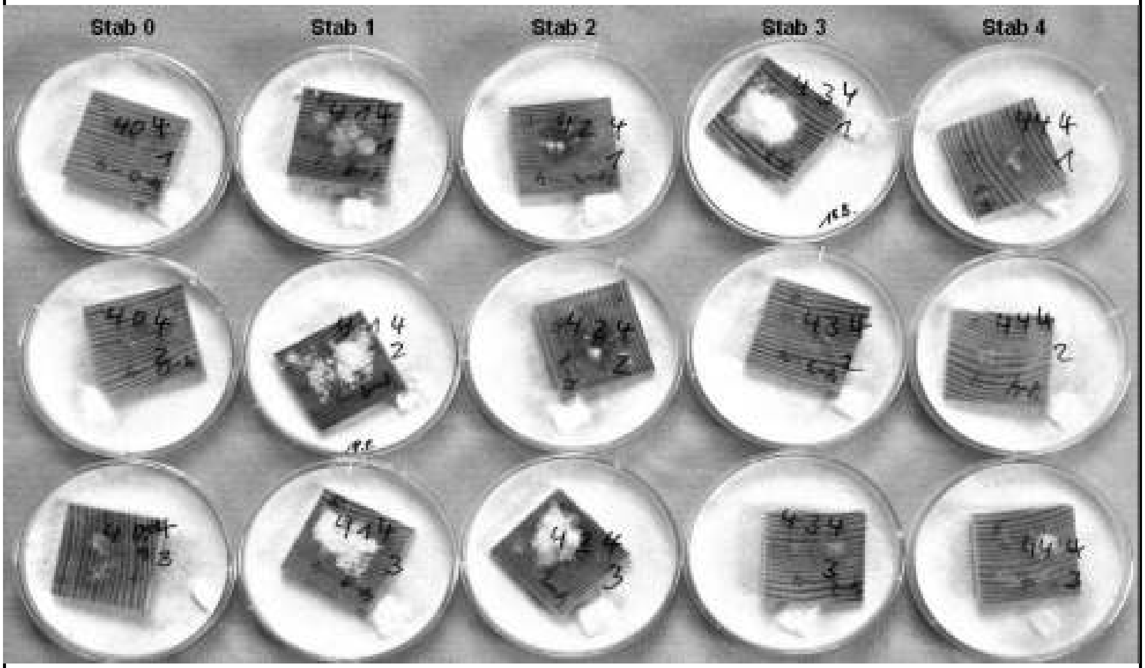

Figure 9: Surface mycelium of *C. versicolor* after 6 weeks of incubation on cross-cut sections of stakes treated with preservative 10 (5 stakes, 3 replicates per stake)

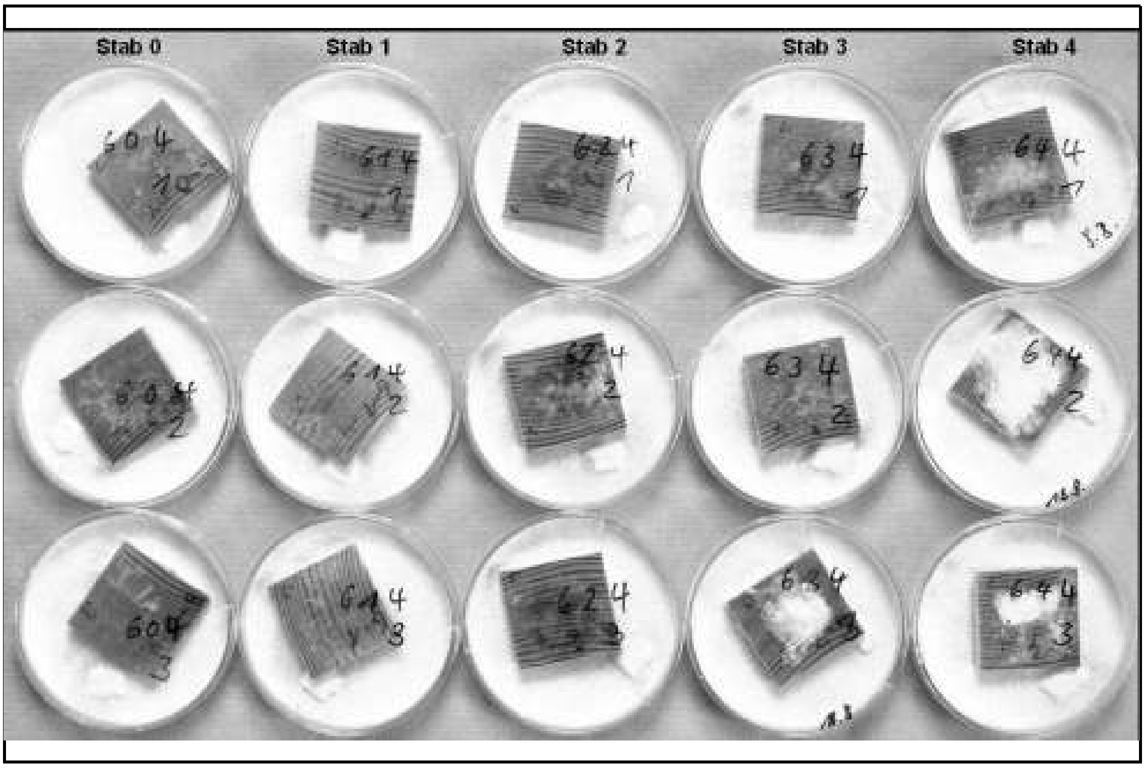

Figure 10: Surface mycelium of *C. versicolor* after 6 weeks of incubation on cross-cut sections of stakes treated with preservative 47 (5 stakes, 3 replicates per stake).

THE COST EURO INDEX FOR FUNGAL DECAY - FIVE YEARS RESULTS

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1 Introduction

In 1971 Theodore C. Scheffer published his “Climate index for estimating potential for decay in wood structures above ground” for the mainland of the USA. Although more or less rough even for the US with their huge relatively uniform climate zones, this index is regarded as a useful tool for both architects and producers of wooden constructions and outdoor commodities which helps them determine for their products adequate protection by design and preservation by chemical means against wood destroying fungi. Well aware of the difficulties arising from the need for long-term observations and a high amount of funding necessary to complete such a project, a group of European experts on wood preservation, gathering in the COST E2 action, nevertheless made a first step to develop something like a European equivalent to the “Scheffer index”. The framework of the European SMT Project FACT “Co-normative research on field tests for wood preservatives out of ground contact in conjunction with methods for preconditioning test specimens prior to test” (SMT4 – CT96 – 2135) provided the opportunity for funding at least the production and the distribution of wood specimens from one source as a starting point of the COST Euro index trial. Periodical assessment and evaluation of the results could be realized only on the basis of national funding.

2 Objectives

As outlined in the first progress report (Carey & Molnar 2000), the aim of the project is to develop a European index for fungal decay which can be used to estimate the severity of the (regional) hazard experienced by wood used in constructions out of ground contact and exposed to situations described in use class 3 (formally hazard class 3) of EN335-1.

Therefore it was and still is necessary to correlate climatic conditions with the rate of fungal decay:

- to be able to predict rates of decay or “hazard”, and
- to assess the suitability of field trial sites for achieving valid results with European standard methods of test (EN 330 and ENV 12037).

Another objective was: to reconsider the rating schemes used for rating the condition of the exposed test specimens. Results of tests in the FACT project, running parallel to this project, have already led to the preliminary amendment of the rating scheme of ENV 12037 and to discussions on the amendment of the scheme of EN 330.

Other secondary objectives were:

- to assess the need for specimens for destructive assessment in ENV 12037, and
- to develop guidance on the procedure of rating the specimens so as to provide better conformity between different assessors.

3 Work done to date

In July 1998 sets of 20 untreated, coated L-joints according to EN 330, and 20 untreated lap-joints, as described in ENV 12037, were distributed by the UK partners to 17 participants in Europe and one in the USA who all expressed interest in the trial. The test site in the US was Hilo, Hawaii. A second test site outside mainland Europe, run by CTBA, France, included in the trial is at Guadeloupe. These two sites, both well established for conducting field trials and very rapid fungal decay, provide the most valuable opportunity to compare necessary European durations of exposure with tropical conditions. Unfortunately, due to leaving of colleagues and lack of national funding, two of the participants could either not start or not continue their part in the project. The list of remaining participants who provided data is given in Table 1 together with the dates of exposure of specimens. CSI (Laporte) and BAM did not expose L-joints. BAM and HFA exposed lap-joints from a different source. The participants provided racking appropriate for the types of joint.

In the beginning most of the participants assessed their specimens in about half year intervals; later about one year intervals prevailed. Not in all cases was it possible to assess the specimens every year at the same month. Accidental corruption of electronic data led to gaps in the continuation of data of two of the participants. Nevertheless, at least data for the four to five years assessments could be submitted. The assessment scales for stain and decay are given in Table 3. Some of the partners already used the amended decay rating scale of ENV 12037. In co-operation with the partners attempts have been made to convert these data to line up with the original rating scale for the project.

The number of joints that had been distributed allowed for destructive assessment after periods of three and five years. So far only BFA and Janssen Pharmaceutica made use of this possibility (both after about 5 years). This procedure is part of the assessment process in EN 330 but not in ENV 12037 at the present time. Considering the extraordinary long periods until the beginning of decay at some of the test sites and the overall considerable variety in the number of specimens attacked, it seems to be better to save the existing specimens for further visual assessments during the years to come.

Not all the participants have climate recording facilities at their sites. But data were collected from the nearest weather stations. The monthly mean temperatures and the accumulated monthly precipitation were regarded as minimum data for the equation of climatic conditions with the rates of decay. Participants who could provide climate data are listed in Table 2.

4 Results to date

The results on fungal staining and decay received to date are summarized in Table 4 for the lap-joints and in Table 5 for the L-joints. In tables 6 and 7 the data are grouped around the approximate periods of assessment. In order to provide a better overview, the decay data recorded up to 21 months and the staining data are excluded from these tables, the latter because on the lap-joint outer surfaces the staining reached rating 3 within one year and was at least complete within three years in the joint area of both types of specimens.

A compilation of the climate data received from the participants is given in Table 8. Assuming that the precipitation falling in times with temperatures below 0 °C does not contribute to the moistening of the specimens (at least until melting) and that at temperatures lower than 5 °C there is no decay activity, the number of months with temperatures in steps of 5 °C are given together with the accumulated precipitation during these months. Where available three years mean values of these numbers of months and of the corresponding precipitations are given in Table 9.

5 Discussion

Although an overall increase in the speed and the rate of decay from the north to the south of Europe is obvious as is for instance illustrated by the data from Ultuna versus Lisbon . It is also obvious that the rate of decay can differ remarkably between sites located at or near to the same northern latitude. For this Ultuna versus Espoo and Birkenes are clear examples (see also Table 10 for names and locations of test sites and approximate ranking according to the degree of fungal decay).

The local climate plays an important role even from year to year which can be seen from the lap-joint results of BAM (see Table 6). No increase of the rate of decay took place between years 5 and 6 of the exposure in Horstwalde. The reason was a locally very hot and (as can be seen from Table 8) dry summer in 2003.
By comparing the climate data of Ultuna and the data of the weather station near Birkenes which show nearly identical long periods of temperatures below 0°C but a huge difference in the amount of precipitation, the influence of the precipitation on the decay process becomes visible. There is not much difference in the altitude of the location of these sites (see Tables 11a and 11b). Unfortunately no precipitation data have been reported for Espoo which is located on the same altitude as Ultuna but shows a much higher rate of decay in the exposed lap-joints.

On the other hand differences in the amount of precipitation alone cannot explain the differences in the rate of decay in lap-joints exposed in Bois le Roi and in Vienna. Although in Vienna the three years mean of precipitation was lower than in Bois le Roi and despite the fact that Vienna has also minus degrees in the winter, the lap-joints of Vienna suffered a much higher rate of decay than those of Bois le Roi (Tables 11a and 11b). The amount of precipitation also does not explain the differences between the rates of decay of the lap-joints exposed in Garston and Sunningdale on the one hand and the rates of decay of the lap-joints exposed in Bois le Roi on the other. The three years means of precipitation are nearly identical at the UK sites and the France site. One possible explanation may be the form of the precipitation and the relative humidity. It can make a remarkable difference, if the precipitation falls down over a long period in very small drops like in the frequent mist of the British Isles, or pours down in short but strong showers. The much higher rate of decay in the upper surface of the UK lap-joints may be an indication of the influence of a typical weather (of at least parts) of England.

Except at the UK sites, the Portuguese site and the tropical Guadeloupe site the joint of the lap-joint was (as expected) the main entrance for the start of decay. To achieve experiences with the lap-joint it is most valuable to assess the decay in each single part of the specimen. But it is still an open question how to summarize the single data and to formulate an overall rating of decay for the whole of the lap-joint specimen.

The L-joint results are much more uniform for the sites in central Europe than those of the lap-joints. Only two of the Nordic sites (Ultuna and Birkenes) and the Portuguese site make an exception. Nearly nothing happened at the Nordic sites, whereas the decay was rapid and most severe in Lisbon. Not in line with the other Nordic sites was Espoo. The results of Espoo were comparable to those of the sites of central Europe.

As the differences between the results of some of the test sites are in most cases not so pronounced, the ranking of some of the sites can only be approximate and preliminary. As stated in the first progress report, the influence of different interpretations of the rating scheme on the results cannot be estimated. The secondary objective “to develop guidance on the procedure of rating the specimens so as to provide better conformity between different assessors” is still valid and should be fulfilled. Now that the decay progresses and different rates of decay are shown in the same set of specimens, it seems to be the best time for this task.

6 Conclusions

- The data achieved within five to six years of exposure allow an approximate ranking or grouping of the existing European test sites for above ground tests according to the rates of decay (or “hazard”) in lap-joints and (to a lesser extent) in L-joints.
- Both the lap-joint and the L-joint methods are no accelerated methods of test.
- These methods may provide valuable performance data for wood preservatives for different regions of Europe, but
- they do not provide reliable efficacy data for approvals of wood preservatives as required by EN599.
- First results of destructive assessment of lap-joints are not sufficient for the decision on whether or not destructive assessment should be part of ENV12037.

- Guidance on the procedure of rating of specimens is desirable.
- The rating scheme for the assessment of lap-joints (ENV 12037) has already been amended, taking into account the experiences from the FACT project and those described by Carey & Suttie (2001). The rating scheme of the L-joint method (EN 330) should be amended likewise. Nevertheless the new scheme of ENV 12037 seems to be relatively rough when compared to the scheme used by Scheffer (1971) for his decay index (see also Scheffer et al: (1963)).
- A summarized rating of decay for the whole of the lap-joint specimen seems to be necessary as a basis for the calculation of a decay index. This needs further discussion.
- Mean monthly temperatures and monthly precipitations are not enough for the interpretation of differences in the decay rates of lap-joints exposed in different European regions. Mean monthly precipitations do not really reflect the course of the moisture content in the specimens which is the basis for any fungal activity; it may be necessary to take into consideration also the number of days with rainfall and even the course of the relative humidity as to provide more meaningful information.
- As annual climate differences may be very pronounced mean climate data of a number of years are necessary. The means of three years considered in this report may not be sufficient.
- Whenever possible, the project should be continued. At least additional climate data available from relevant weather stations should be taken into consideration, and a further workshop on assessment should be held.

Table 1. Remaining participants and dates of exposure

Participant	Country	Date exposed
IMP COL	UK	08/98
BRE	UK	01/99
SHR	Netherlands	09/98
BAM	Germany	09/97 (lap-joints only)
BFH	Germany	07/98
Dr. Wolman GmbH	Germany	? (L-joints not reported)
CTBA	France	10/98 (lap-joints) 11/99 (L-joints)
	Guadeloupe (France overseas department)	09/98 (lap-joints) 10/99 (L-joints)
SLU	Sweden	07/98
Janssen Pharmaceutica	Belgium	09/98
RUG	Belgium	08/98
HFA	Austria	07/98 (lap-j. 13, L-j. 20 specim.)
EMPA	Switzerland	07/98
VTT	Finland	09/98
NTI	Norway	09/98
LNEC	Portugal	01/99
CSI (Laporte)	USA (Hilo, Hawaii)	09/98

Table 2. Minimum climate data reported (monthly mean temperature and accumulated monthly precipitation)

Participant	Data reported	Weather station	Data complete yes + / no -
IMP COL	-		
BRE	January 1998 to February 2004	Garston	+
SHR	-		
BAM	January 1998 to December 2003	Baruth	+
BFH	June 2000 to November 2003	Hamburg	+
Dr Wolman GmbH	-		
CTBA Bois le Roi	October 1997 to August 2003	Fontainebleau	nearly
CTBA Guad.	October 1997 to September 2003	Guadeloupe	-
SLU	July 1998 to December 2003	Ultuna	+
Janssen Pharmaceutica	January 2000 to December 2003	KleineBrogel	+
RUG	January 2000 to December 2003	Semmerzake	+
HFA	January 2000 to December 2003	Vienna	+
EMPA	January to December 1998	St. Gallen	+
VTT	August 1998 to August 2002	Espoo	+ (temp. only)
NTI	October 1998 to August 2003	Birkenes	+
LNEC	January 1999 to December 2003	Lisbon	-
CSI (Laporte)	-		

Table 3. Rating scale

Rating type	Rating	Description	Definition
Stain	S0	No discoloration	No evidence of discoloration caused by micro-organisms
	S1	Slight discoloration	Slight discoloration, often dark and in streaks and/or individual stained spots; in total no more than 3 cm^2 affected
	S2	Distinct discoloration	Distinct discoloration, but in discrete patches and streaks with over 3 cm^2 affected or patches of continuous staining making up no more than 1/3 of the total surface area
	S3	Total discoloration	Complete or near complete discoloration of the entire surface area
Decay	D0	Sound	No softening or weakening of the wood
	D1	Moderate attack	Small areas of decay (softened weakened wood); typically not more than 3 cm^2 affected.
	D2	Severe attack	Marked softening and weakening of the wood typical of fungal decay and in extensive patches and streaks; distinctly more than 3 cm^2 affected
	D3	Failure	Very severe and extensive rot; joint members often capable of being easily broken.

Table 4. Summary of lap-joint data received until April 16, 2004 (nominal mean rating)

Participant	Exposure months	Upper surface		Lower surface		Within the joint	
		stain	decay	stain	decay	stain	decay
IMP COL	6	1.9	0	2.4	0	1.2	0
	14	3	0	3	0	2.3	0
	20	3	0	3	0	2.4	0.2
	52	3	2	3	1.8		1.7
	66	3	2.3	3	2.1	3	1.9
BRE	16	1.4	0	2.7	0.8	2.4	0.1
	60		2.1		1.9		2.3
SHR	6	1.5	-	2.5	-	1.2	0
	61	3	1	2.4	1	2.9	1.5
BAM	6	3	0	3	0	2	0
	13	3	0	3	0	3	0
	18	3	0	3	0	3	0
	25	3	0	3	0	3	0
	30	3	0	3	0	3	0.6
	37	3	0	3	0	3	0.6
	50	3	0	3	0.3	3	1.1
	63	3	0.2	3	0.7	3	1.5
	73	3	0.2	3	0.7	3	1.5
BFH	8	2.8	0	3	0	2.7	0
	14	3	0	3	0	3	0
	27	3	0	3	0	3	0.1
	34	3	0	3	0.2	3	0.5
	40	3	0	3	0.1	3	0.5
	51	3	0.1	3	0.1	3	0.9
	57	3	0.1	3	0	3	0.6
	68	3	0.6	3	0.6	3	1.1

Table 4. Continued

Participant	Exposure months	Upper surface		Lower surface		Within the joint	
		stain	decay	stain	decay	stain	decay
Dr.Wolman GmbH	24		0		0		0.1
	48		0.3		0.1		1.1
	60		0.3		0.1		1.6
CTBA Bois le Roi	6	3	0	3	0	1.8	0
	12	3	0	3	0.1	3	0.2
	18	3	0.1	3	0.1	3	0.2
	24	3	0.1	3	0.1	3	0.2
	30	3	0.1	3	0.1	3	0.3
	36	3	0.1	3	0.1	n. a.	n. a.
	48	3	0.1	3	0.2	3	1.1
	60	3	0.1	3	0.3	3	1.4
CTBA Guadeloupe	6	2.3	0.2	2.8	0.5	2.1	0.3
	12	3	0.9	3	1.2	2.9	1.7
	18	3	1.3	3	1.6	3	1.7
	24	3	1.6	3	2	3	1.9
	30	3	3	3	2.9	3	2.9
	42	3	3	3	3	3	3
	54	3	3	3	3	3	3
SLU	3	2.1	0	2.1	0	1	0
	10	3	0	3	0	1.6	0
	15	3	0	3	0	2	0
	22	3	0	3	0	2.4	0
	28	3	0	3	0	2.9	0.1
	39	3	0.1	3	0	3	0.2
	52	3	0.2	3	0.2	3	0.8
	62	3	0.2	3	0.3	3	1
Janssen	37	3	0	3	0	3	1
	48	3	0.7	3	0.4	3	1.3
	61	3	1.8	3	1.1	3	2
RUG	66					3	2.2

Table 4. Further continued

Participant	Exposure months	Upper surface		Lower surface		Within the joint	
		stain	decay	stain	decay	stain	decay
HFA	6	3	0	2.9	0	3	0
	12	3	0	3	0	3	0
	18	3	0	3	0	3	0
	24	3	0	3	0	3	0.2
	30	3	0	3	0	3	1
	37	3	0	3	0	3	1
	50	3	1.4	3	1.4	3	2.2
	64	3	1.6	3	1.4	3	2.3
EMPA	5	3	0	2.8	0	3	0
	14	3	0	3	0	3	0
	22	3	0	3	0	3	0
	35	3	0	3	0	3	0.1
	46	3	0.3	3	0	3	1
	58	3	0.8	3	0.1	3	1.4
VTT	24?	3	0.4	3	0.8	2.9	0.7
	36?	3	0.6	3	1.3	3	1.1
	49	3	0.7	3	1.4	3	1.4
NTI	13	3	0	2	0	1	0
	19	3	0	3	0	2	0
	36	3	0.1	2.9	0.1	2.9	0.3
	45	3	0	2	0.3	2.1	0.4
	60	3	1.1	2.2	1.5	2	1.4
LNEC	6	1.6	0	2.3	0	3	0
	12	2.1	0	3	0.1	3	0
	42	3	2.7	3	2.2	3	2.7
	48	3	2.9	3	2.7	3	3
CSI (Laporte)	6	3	0	2.5	0.3	1.4	0.3

Table 5. Summary of L-joint data received until April 16. 2004 (nominal mean rating)

Participant	Exposure months	External surfaces		Within the joint	
		stain	decay	stain	decay
IMP COL	6			1.6	0
	14			1.8	0
	20			2.1	0.1
	52				1.3
	66				1.8
BRE	12				0
	60				1.9
SHR	6			0.2	0
	61			2.9	1.7
BAM	Not exposed	-	-	-	-
BFH	8	0.2		2.7	0
	14	0.6		3	0.1
	20	0.7		3	0.3
	27	1.1		3	0.5
	34	1.5		2.9	0.4
	39	1.2		3	0.5
	51	2.1		3	1.9
	57	1.9		3	1.3
	64	?		3	2.0 ?
Dr. Wolman GmbH	No report	-	-	-	-
CTBA Bois le Roi	6			2.3	0
	12			2.3	0
	18			2.4	0.1
	36			3.0	1.4
	48			3.0	1.7
CTBA Guadeloupe	6			0.4	0.2
	12			0.6	0.1
	30			2.3	2.3
	42			2.3	2.5
CSI (Laporte)					

Table 6. Summary of lap-joint decay data (nominal mean rating, number of lap-joints with decay rating 3 in parenthesis): Numbers in front of the participant's acronyms reflect the approximate ranking of the sites according to the degree of the decay hazard (max 1, min 16).

Participant (Northern latitude)	Exposure period - months																							
	22 … 25			27 … 30			34 … 39			40 … 42			45 … 54			58 … 64			66 … 68			73		
	Us	Ls	Jt	Us	Ls	Jt	Us	Ls	Jt	Us	Ls	Jt	Us	Ls	Jt	Us	Ls	Jt	Us	Ls	Jt	Us	Ls	Jt
16 SLU (little below 60°)	0	0	0	0	0	0.1	0.1	0	0.2				0.2	0.2	0.8	0.3	0.3	1						
15 BFH (about 53.5)				0	0	0.1	0	0.2	0.5	0	0.1	0.5	0.1	0.1	0.9	0.1	0	0.6	0.6 (1)	0.6	1.1			
14 BAM (about 52°)	0	0	0	0	0	0.6	0	0	0.6				0	0.3	1.1	0.2	0.7	1.5				0.2	0.7	1.5
13 CTBA BLR (about 48.5°)	0.1	0.1	0.2	0.1	0.1	0.3	0.1	0.1	-				0.1	0.2	1.1	0.1	0.3	1.4						
12 EMPA (about 47.5°)	0	0	0				0	0	0.1				0.3	0	1	0.8	0.1	1.4						
11 SHR (about 52°)																			1	1	1.5			
10 NTI (about 58°)							0.1	0.1	0.3				0	0.3	0.4	1.1	1.5	1.4						
9 Wolman (about 49.5°)	0	0	0.1										0.3	0.1	1.1 (5)	0.3	0.1	1.6 (5)						
8 VTT (little above 60°)	0.4	0.8	0.7				0.6	1.3	1.1				0.7	1.4	1.4									
7 RUG (about 51°)																					2.2			
6 Janssen (about 51°)							0	0	1				0.7	0.4	1.3	1.8 (4)	1.1	2 (7)						
5 HFA (little above 48°)	0	0	0.2	0	0	1	0	0	1				1.4 (2)	1.4 (2)	2.2 (2)	1.6 (3)	1.4 (2)	2.3 (4)						

Us: upper surface; Ls: lower surface; Jt: within the joint

Table 6. Continued.

Participant (Northern latitude)	Exposure period - months																							
	22 … 25			27 … 30			34 … 39			40 … 42			45 … 54			58 … 64			66 … 68			73		
	Us	Ls	Jt	Us	Ls	Jt	Us	Ls	Jt	Us	Ls	Jt	Us	Ls	Jt	Us	Ls	Jt	Us	Ls	Jt	Us	Ls	Jt
4 IMP COL (about 51.5°)													2	1.8	1.7				2.3 (6)	2.1 (2)	1.9 (1)			
3 BRE (about 51.5°)																2.1 (1)	1.9 (1)	2.3 (2)						
2 LNEC (little below 39°)										2.7 (13)	2.2 (3)	2.7 (14)	2.9 (17)	2.7 (13)	3 (20)									
1 CTBA Guad. (little above 16°)	1.7 (1)	2 (1)	1.9 (1)	3 (20)	3 (18)	3 (17)				3 (20)	3 (20)	3 (20)	3 (20)	3 (20)	3 (20)									

Us: upper surface; Ls: lower surface; Jt: within the joint

Table 7. Summary of L-joint decay data (nominal mean rating, number of L-joints with decay rating 3 in parenthesis): Numbers in front of the participant's acronyms reflect the approximate ranking of the sites according to the degree of the decay hazard (max 1, min 14).

Participant (Northern latitude)	Exposure period – months						
	20 … 25 Joint	27 … 30 Joint	34 … 39 Joint	40 … 42 Joint	45 … 54 Joint	58 … 64 Joint	65 … 68 Joint
14 SLU (12)* 60°	0	0	0		0	0.2	
13 NTI (10) (little above 58°)			0		0.1	0.1	
12 EMPA (12) (about 47.5°)	1		1.1		1.1	1.5	
11 BFH (15) (about 53.5)	0.3	0.5	0.4	0.5	1.9	1.3 (2)	2.0 ? (5)
10 HFA (5) (little above 48°)	0.2	0.2	0.2		0.7	1.3 (4)	
9 IMP COL (4) (about 51.5°)	0.1				1.3		1.8 (8)
8 SHR (11) (about 52°)						1.7	
7 RUG (7) (about 51°)						1.7 (5)	
6 VTT (8) (little above 60°)			1.2		1.8		
5 CTBA BLR (13) (about 48.5°)			1.4 (2)		1.7 (3)		
4 BRE (3) (about 51.5°)						1.9	
3 Janssen Ph. (6) (about 51°)			1.1		1.5 (1)	2.2 (7)	
2 LNEC (2) (little below 39°)				2.1 (1)	2.2 (7)		
1 CTBA Gu. (1) (little above 16°)		2.3 (3)		2.5 (12)			

* Ranking of lap-joint data according to the degree of decay at the same site

Table 8. Summary of relevant weather data received

Participant (weather station)	Year	Range of mean monthly temperatures [°C] 1st line: N° of months; 2nd line: corresp. Accumulated precipit. [mm]						Remarks
		< 0	> 0…5	> 5..10	> 10..15	> 15..20	> 20	
BRE (Garston)	1998			6	4	2		
				429	322	139		
	1999		1	5	3	3		
			88	259	178	202		
	2000		1	5	3	3		
			18	453	341	95		
	2001		1	5	2	4		
			67	364	217	192		
	2002			5	3	4		
				498	200	224		
	2003			4	5	3		
				190	268	82		
BAM (Baruth)	1998		4	3	1	4		
			173	147	75	252		
	1999		4	2	2	3	1	
			156	75	64	111	40	
	2000		4	1	3	4		
			184	27	107	144		
	2001	1	4	1	4	2		
		48	173	54	251	79		
	2002	1	2	4	1	4		
		30	107	228	38	336		
	2003	2	2	3	1	4		
		51	51	107	29	151		
BFH (Hamburg)	2000		1	1	2	3		June to Dec only
			62	35	137	224		
	2001		4	2	4	2		
			256	151	419	181		
	2002		2	5	1	3	1	
			121	498	92	316	189	
	2003		2	3	2	2	2	Jan to Nov only
			101	135	133	130	97	
CTBA (Fontainebleau)	1998		3	2	2	3		No temp. of Feb, Aug
			158	96	210	273		No precip. of Feb, Aug
	1999		1	3	2	4		No temp. of March, Sept
			44	290	168	255		No precip. of March, Sept
	2000		1	4	2	5		
			23	301	236	327		
	2001		2	4	2	4		
			131	315	150	344		
	2002			6	3	3		
				356	158	170		
	2003		2	1	2	2	1	No temp. of Sept to Dec
			100	19	127	101	30	No precip. of Sept to Dec

Table 8. Continued

Participant (weather station)	Year	Range of mean monthly temperatures [°C] 1st line: N° of months; 2nd line: corresp. Accumulated precipit. [mm]						Remarks
		< 0	> 0…5	> 5…10	> 10..15	> 15...20	> 20	
CTBA (Guadeloupe)	1998						9	No temp. of May to July
							4381	No precip. of Feb.
	1999						8	No t. Jan, Feb, July, Aug
							2175	No p. Jan, Feb, May - Aug
	2000						10	No temp of. Aug , Sept
							2866	
	2001						9	No t. of Jan, Feb, Sept
							2837	No precip. of Jan, Sept
	2002						12	
							3777	
	2003						9	No temp. of Oct to Dec
							3892	No precip. of Dec
SLU (Ultuna)	1998	2	1		2	1		July to Dec. Only
		18	14		224	18		
	1999	3	2	3	2	2		
		45	12	32	21	11		
	2000	2	2	3	3	2		
		7	17	54	24	36		
	2001	4	1	2	3	2		
		32	13	20	22	47		
	2002	3	3	1	2	3		
		27	33	2	15	38		
	2003	3	4		2	3		
		24	30		20	33		
Janssen Pharmaceutica (KleineBrogel)	2000		1	4	3	4		
			55	307	248	378		
	2001		3	4	3	2		
			192	325	225	190		
	2002		2	5	3	2		
			240	415	234	160		
	2003		3	3	3	3		
			191	203	151	150		
RUG (Semmerzake)	2000		1	5	2	4		
			32	330	197	363		
	2001		2	4	3	3		
			135	353	282	183		
	2002		1	4	4	3		
			112	374	193	319		
	2003	1	2	3	2	4		
		24	158	126	85	74		

Table 8. Continued further

Participant (weather station)	Year	Range of mean monthly temperatures [°C] 1st line: Nº of months; 2nd line: corresp. Accumulated precipit. [mm]						Remarks
		< 0	> 0…5	> 5…10	> 10...15	> 15...20	> 20	
HFA (Vienna)	2000	1	1	3	2	3	2	
		44	42	150	65	159	76	
	2001	1	3	2	2	2	2	
		48	94	80	113	88	113	
	2002	1	1	4	1	2	3	
		64	12	264	52	90	333	
	2003	2	1	3	1	2	3	
		62	38	70	32	151	93	
EMPA (St. Gallen)	1998		4	2	2	3		Feb to Dec only
			362	237	303	490		
VTT (Espoo)	1998	2	1		2			Only temp. Aug to Dec
	1999	4	1	3	2	2		Temp. only
	2000	3	2	3	3	1		Temp. only
	2001	4	2	2	2	2		Temp. only
	2002	2	2		1	3		Only temp. Jan to Aug
NTI (Birkenes)	1998	2		1				Oct to Dec only
		188		224				
	1999	3	2	3	3	1		
		545	274	353	624	38		
	2000		5	2	4	1		
			1290	462	515	155		
	2001	4	2	1	4	1		
		504	250	318	388	94		
	2002	2	4	1	3	2		
		255	584	145	342	260		
	2003	2	2	1	1	2		Jan to Aug only
		166	189	223	83	221		
LNEC (Lisbon)	2001			1	4	3	4	No precip. of Dec
					765	213	81	

Table 9. Mean of three years of months/temperatures and accumulated precipitations

Participant (weather-station)	Year	Range of mean monthly temperatures [°C] 1st line: N° of months; 2nd line: corresp. accumulated precipit. [mm]					
		< 0	> 0...5	> 5...10	> 10...15	> 15...20	> 20
BRE (Garston)	2000		1	5	3	3	
			18	453	341	95	
	2001		1	5	2	4	
			67	364	217	192	
	2002			5	3	4	
				498	200	224	
	Mean		0.7	5.0	2.7	3.7	
	Mean		28.3	438.3	252.7	170.3	
BAM (Baruth)	2000		4	1	3	4	
			184	27	107	144	
	2001	1	4	1	4	2	
		48	173	54	251	79	
	2002	1	2	4	1	4	
		30	107	228	38	336	
	Mean	0.7	3.3	2.0	2.7	3.3	
	Mean	26.0	154.7	103.0	132.0	186.3	
CTBA (Fontainebleau)	2000		1	4	2	5	
			23	301	236	327	
	2001		2	4	2	4	
			131	315	150	344	
	2002			6	3	3	
				356	158	170	
	Mean		1.0	4.7	2.3	4.0	
	Mean		51.3	324.0	181.3	280.3	
SLU (Ultuna)	2000	2	2	3	3	2	
		7	17	54	24	36	
	2001	4	1	2	3	2	
		32	13	20	22	47	
	2002	3	3	1	2	3	
		27	33	2	15	38	
	Mean	3.0	2.0	2.0	2.7	2.3	
	Mean	22.0	21.0	25.3	20.3	40.3	

Table 9. Continued

Participant (weather-station)	Year	Range of mean monthly temperatures [°C] 1st line: N° of months; 2nd line: corresp. accumulated precipit. [mm]					
		< 0	> 0...5	> 5...10	> 10...15	> 15...20	> 20
Janssen Pharmaceutica (KleineBrogel)	2000		1	4	3	4	
			55	307	248	378	
	2001		3	4	3	2	
			192	325	225	190	
	2002		2	5	3	2	
			240	415	234	160	
	Mean		2.0	4.3	3.0	2.7	
	Mean		162.3	349.0	235.7	242.7	
RUG (Semmerzake)	2000		1	5	2	4	
			32	330	197	363	
	2001		2	4	3	3	
			135	353	282	183	
	2002		1	4	4	3	
			112	374	193	319	
	Mean		1.3	4.3	3.0	3.3	
	Mean		93.0	352.3	224.0	288.3	
HFA (Vienna)	2000	1	1	3	2	3	2
		44	42	150	65	159	76
	2001	1	3	2	2	2	2
		48	94	80	113	88	113
	2002	1	1	4	1	2	3
		64	12	264	52	90	333
	Mean	1.0	1.7	3.0	1.7	2.3	2.3
	Mean	52.0	49.3	164.7	76.7	112.3	174.0
NTI (Birkenes)	2000		5	2	4	1	
			1290	462	515	155	
	2001	4	2	1	4	1	
		504	250	318	388	94	
	2002	2	4	1	3	2	
		255	584	145	342	260	
	Mean	2.0	3.7	1.3	3.7	1.3	
	Mean	253.0	708.0	308.3	415.0	169.7	

Table 10. Approximate ranking (max 1, min 16) of test fields according to the degree of decay in untreated Scots pine lap-joints.

Participant	Country	Test site	Northern latitude	Ranking
SLU	Sweden	Ultuna	60°	16
BFH	Germany	Hamburg	about 53.5°	15
BAM	Germany	Horstwalde	about 52°	14
CTBA	France	Bois le Roi	about 48.5°	13
EMPA	Switzerland	St. Gallen	about 47.5°	12
SHR	Netherlands	Wageningen	about 52°	11
NTI	Norway	Birkenes	little above 58°	10
Dr. Wolman GmbH	Germany	Limburgerhof	about 49.5°	9
VTT	Finland	Espoo	little above 60°	8
RUG	Belgium	Gent	about 51°	7
Janssen Pharmaceutica	Belgium	Beerse	about 51°	6
HFA	Austria	Vienna	little above 48°	5
IMP COL	UK	Sunningdale	about 51.5°	4
BRE	UK	Garston	about 51.5°	3
LNEC	Portugal	Lisbon	little below 39°	2
CTBA	France (overseas department)	Guadeloupe	little above 16°	1

Table 11a. Summary of lap-joint decay data (nominal mean rating, number of lap-joints decay rating 3 in parenthesis) of selected test sites.

Participant (Northern latitude)	Exposure period - months								
	45 … 54			58 … 64			66 … 68		
	Us	Ls	Jt	Us	Ls	Jt	Us	Ls	Jt
16** SLU (little below 60°)	0.2	0.2	0.8	0.3	0.3	1			
14 BAM (about 52°)	0	0.3	1.1	0.2	0.7	1.5			
13 CTBA BLR (about 48.5°)	0.1	0.2	1.1	0.1	0.3	1.4			
10 NTI (about 58°)	0	0.3	0.4	1.1	1.5	1.4			
8 VTT (little above 60°)	0.7	1.4	1.4						
5 HFA (little above 48°)	1.4 (2)	1.4 (2)	2.2 (2)	1.6 (3)	1.4 (2)	2.3 (4)			
4 IMP COL (about 51.5°)	2	1.8	1.7				2.3 (6)	2.1 (2)	1.9 (1)
3 BRE (about 51.5°)				2.1 (1)	1.9 (1)	2.3 (2)			

Table 11b. Local climate of the selected test sites of table 11a: Mean of three years (2000 to 2002)of mean monthly temperatures and the corresponding accumulated precipitations.

Weather station	Mean of 3 years	Range of mean monthly temperatures [°C] 1st line: N° of months; 2nd line: corresp. accumulated precipit. [mm]					
		< 0	> 0…5	> 5…10	> 10…15	> 15…20	> 20
Ultuna	Mean	3.0	2.0	2.0	2.7	2.3	
	Mean	22.0	21.0	25.3	20.3	40.3	
Baruth	Mean	0.7	3.3	2.0	2.7	3.3	
	Mean	26.0	154.7	103.0	132.0	186.3	
Fontainebl.	Mean		1.0	4.7	2.3	4.0	
	Mean		51.3	324.0	181.3	280.3	
Birkenes	Mean	2.0	3.7	1.3	3.7	1.3	
	Mean	253.0	708.0	308.3	415.0	169.7	
Espoo	M.***	3.7	1.7	2.7	2.3	1.7	
	Mean						
Vienna	Mean	1.0	1.7	3.0	1.7	2.3	2.3
	Mean	52.0	49.3	164.7	76.7	112.3	174.0
(Garston)	Mean		0.7	5.0	2.7	3.7	
	Mean		28.3	438.3	252.7	170.3	
Garston	Mean		0.7	5.0	2.7	3.7	
	Mean		28.3	438.3	252.7	170.3	

* Us: upper surface; Ls: lower surface; Jt: within the joint

** Numbers in front of the participant's acronyms: ranking according to the degree of decay (max 1, min 12)

*** Mean of years 1999 to 2001

7 Acknowledgements

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THE INFLUENCE OF MACRO AND MICRO CLIMATE ON DECAY

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Keywords: micro climate, moisture content, temperature, decay, service life, accelerated testing

Abstract: *Field tests were performed in EHC 3 to determine the influence of the micro climate on decay progress and on decay factors such as temperature und moisture content of the wood. It was found, that trees as well as artificial shadow increase decay activity on wood by a factor of about 2 compared to open exposure conditions. The increase of decay in the artificial shadow box is explained by the occurrence of more non-target organisms, a higher wood moisture content, lower hot-temperatures and higher low-temperatures and by a longer vegetation period. Furthermore the influence of exposure in a tropical greenhouse climate was found to accelerate decay of untreated sapwood of Pinus sylvestris by a factor of 4 compared to normal open exposure in Hamburg.*

1 Introduction

The paper focuses on the influence of the climate and particularly the macro climate on decay. This is important since prediction of performance and service life plays an important role. And service life is the result of the coaction of DURABILITY and EXPOSURE. Exposure can be regarded as climate and micro climate. So the micro climate is responsible for exciting direct decay factors (see Figure 1).

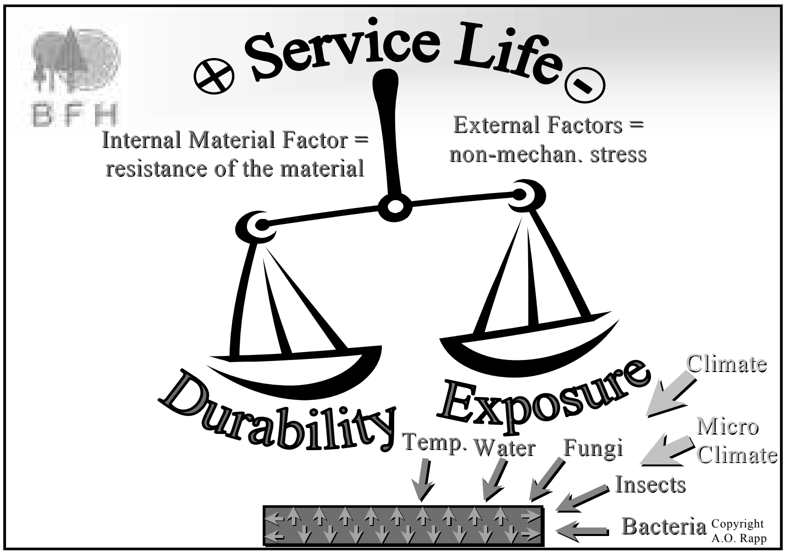


Figure 1. Service life balance

2 Materials and methods

To investigate the relation between climate and decay the experimental set up presented at the COST E22 meeting in Reinbek and Madrid was used (download from the COST E22 web-site http://www.bfafh.de/cost22.htm.

3 Results

3.1 Climate

After 3 summers of exposure there was no strong correlation between climate (rain and temperature) and decay when looking at the 32 sites. Reason for this might be that it was too early for evaluation and/or there might be additional factors to rain and temperature, for example the important role of the micro climate.

3.2 Microclimate caused by trees

First the microclimate created by trees around a wooden double layer construction was regarded (Figure 2).

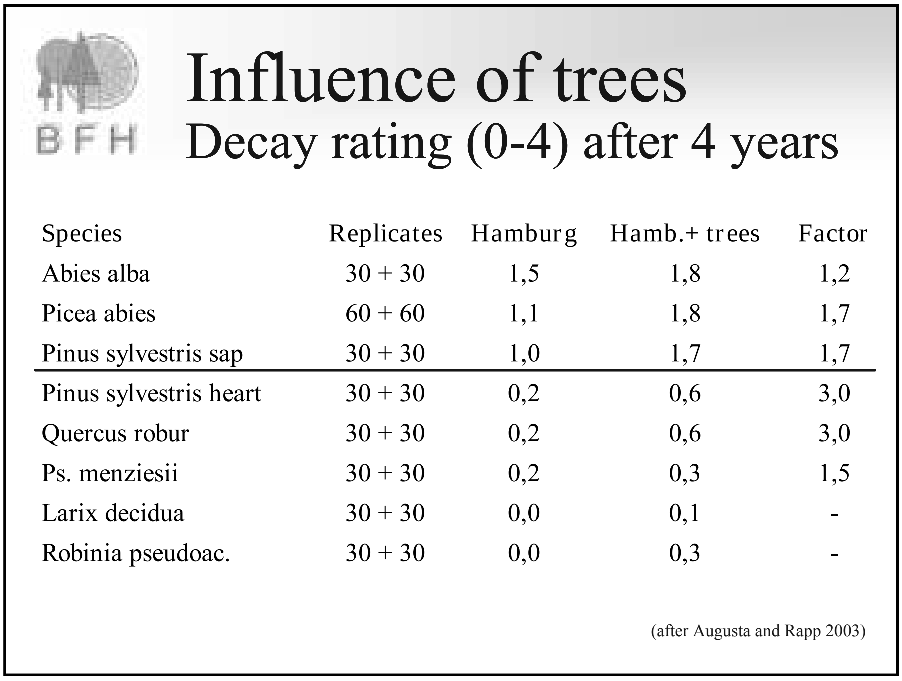
Micro climate
Influence of trees

Figure 2. Test site in Hamburg close to trees, showing a double layer deck which is opened for evaluation after 4 years

The results of 2 such double layer decks about 200 m apart from each other one close to the trees and one in the open field were compared. Each sample tested consisted of at least 30 replicates in the open and 30 in the shadow of the trees so the data can be regarded as quite reliable.

It can be easily seen from Table 1 that the samples exposed close to the trees were considerably more decayed than the samples exposed in the open field 200 m away from the trees. When taking the average then there was nearly a factor of 2 in difference of progress of decay. This means nearly half service life in the shadow of trees.

Table 1. Decay rating from 0 (sound) to 4 (failure) for specimens in the size according to EN 252 exposed for 4 years in Hamburg in the open field and in a field close to trees



BFH

Influence of trees

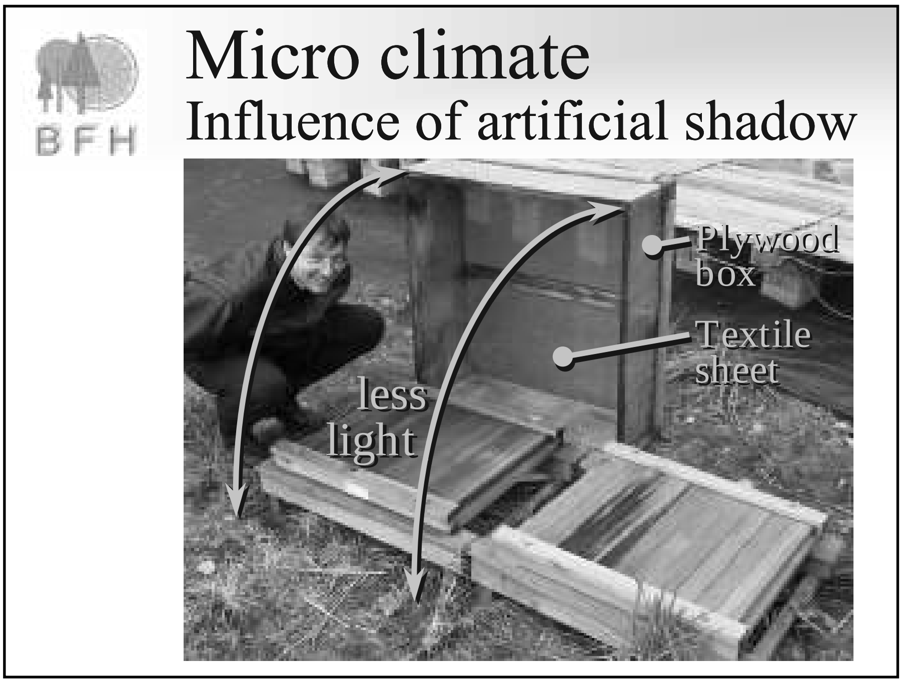
Decay rating (0-4) after 4 years

Species	Replicates	Hamburg	Hamb.+ trees	Factor
Abies alba	30 + 30	1,5	1,8	1,2
Picea abies	60 + 60	1,1	1,8	1,7
Pinus sylvestris sap	30 + 30	1,0	1,7	1,7
Pinus sylvestris heart	30 + 30	0,2	0,6	3,0
Quercus robur	30 + 30	0,2	0,6	3,0
Ps. menziesii	30 + 30	0,2	0,3	1,5
Larix decidua	30 + 30	0,0	0,1	-
Robinia pseudoac.	30 + 30	0,0	0,3	-

(after Augusta and Rapp 2003)

3.3 Microclimate caused by artificial shadow by a box covered with a textile sheet

The question was, whether it would be the same acceleration without trees, but in artificial shadow created by a water permeable textile sheet surrounded by a plywood box (Figure 3).



BFH

Micro climate

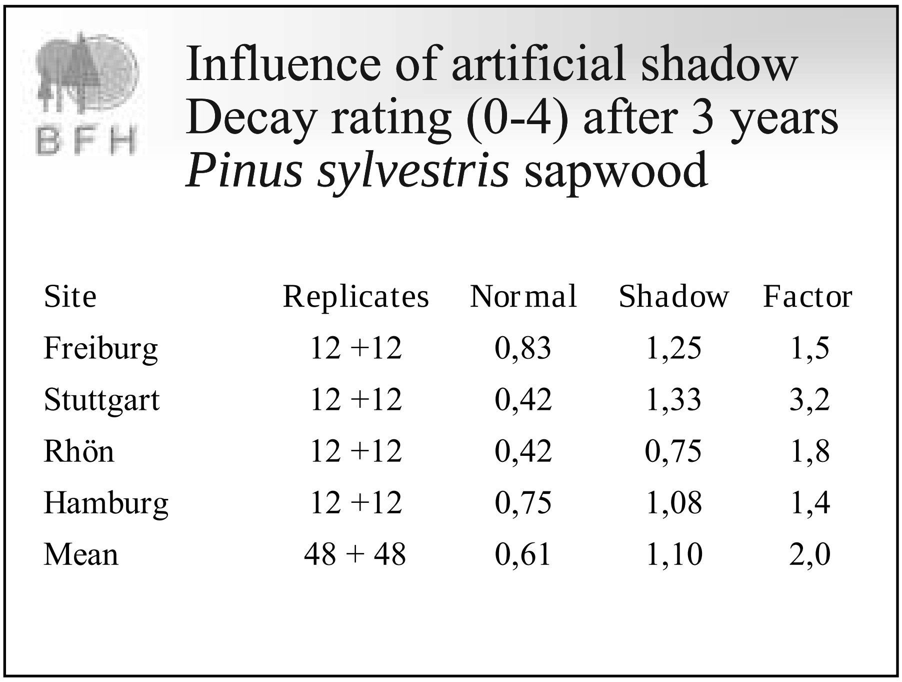
Influence of artificial shadow

Figure 3. Test set up with artificial shadow box (left) and open exposure (right)

A textile sheet was chosen, which allowed only 10% transmission of the light into the box. However, it was easily permeable for rain and moisture.

The reduced amount of sun light in the box lead to less greying of the wood (left) compared to the non-shaded wood (right) as can be seen from Figure 3. In the box a large amount of non-target organisms was found during all the years. Numerical decay ratings after 3 years are given in Table 2 for 4 different sites in Germany, each site equipped without and with artificial shadow box.

Table 2. Decay rating from 0 (sound) to 4 (failure) for specimens in the size according to EN 252 exposed for 4 years in Hamburg in the open field and in a field close to trees



Influence of artificial shadow
Decay rating (0-4) after 3 years
Pinus sylvestris sapwood

Site	Replicates	Normal	Shadow	Factor
Freiburg	12 +12	0,83	1,25	1,5
Stuttgart	12 +12	0,42	1,33	3,2
Rhön	12 +12	0,42	0,75	1,8
Hamburg	12 +12	0,75	1,08	1,4
Mean	48 + 48	0,61	1,10	2,0

It is obvious that the shadow boxes increased decay by a factor of about 2, this is about the same increase by artificial shadow as for the shadow of trees.

In both cases, artificial and natural shadow, more non-target organisms were found and the wood seemed to be more wet. To know about what happened in the boxes, the data logging devices, installed in the wood inside the shadow boxes and in the wood in the open field, were evaluated for all 4 sites. Because the principle results in all 4 different sites were similar, in the following only the data from the Hamburg site are regarded closer.

Figure 4 shows the course of electrical resistance over time. The axis on the right side gives an indication of the wood moisture content corresponding with the electrical resistance. It is obvious that especially in the summer the curves for pine sapwood in the open double layer (orange lines) indicated a lower moisture content than the curves for pine sapwood in the shadow box. In contrast to the samples in the shadow box, the mc of the specimens in the open was not always very favourable for decay.

Figure 5 shows that missing sun light in the shadow box does not automatically mean lower temperatures. In the beginning of winter the wood temperature in the open (orange line) is falling down to – 8°C (see mark 1) while the temperature of the wood in the shadow box (blue line) does not fall below 0°C. Only when it is colder, then also the temperature of the wood in the shadow box (blue line) falls below 0°C (see mark 2). If once the wood in the shadow box is frozen, then it stays (see mark 3). This can be understood by regarding the course of temperatures in spring (Figure 6).

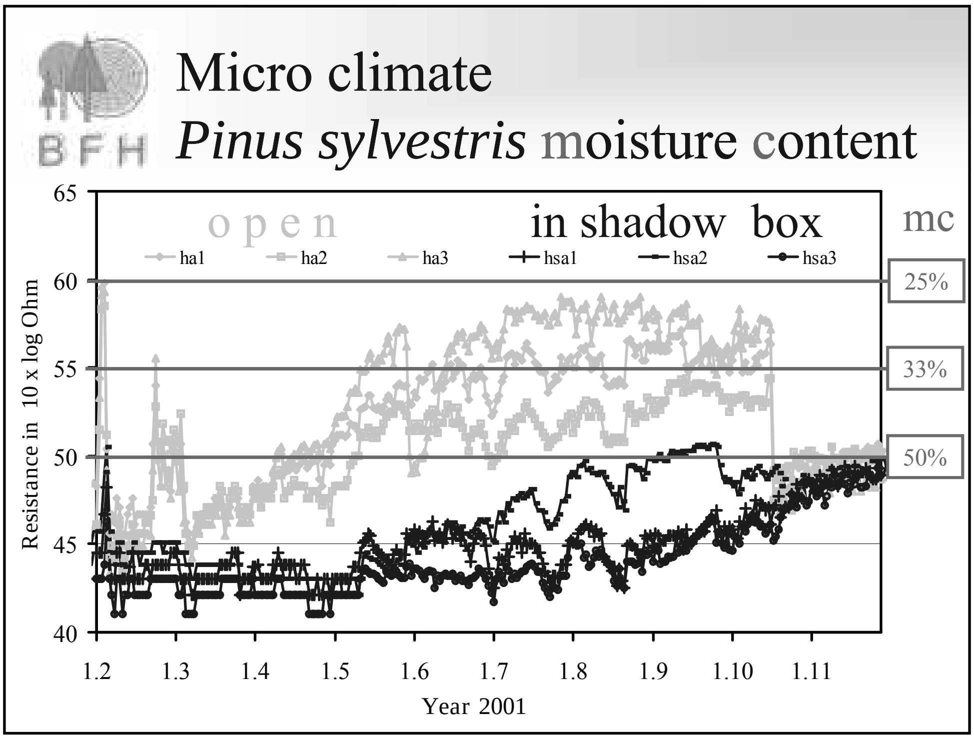


Figure 4. Course of electrical resistance (left axis) corresponding with moisture of wooden specimen (right axis) in the open field (orange lines) and in the shadow box (blue lines)

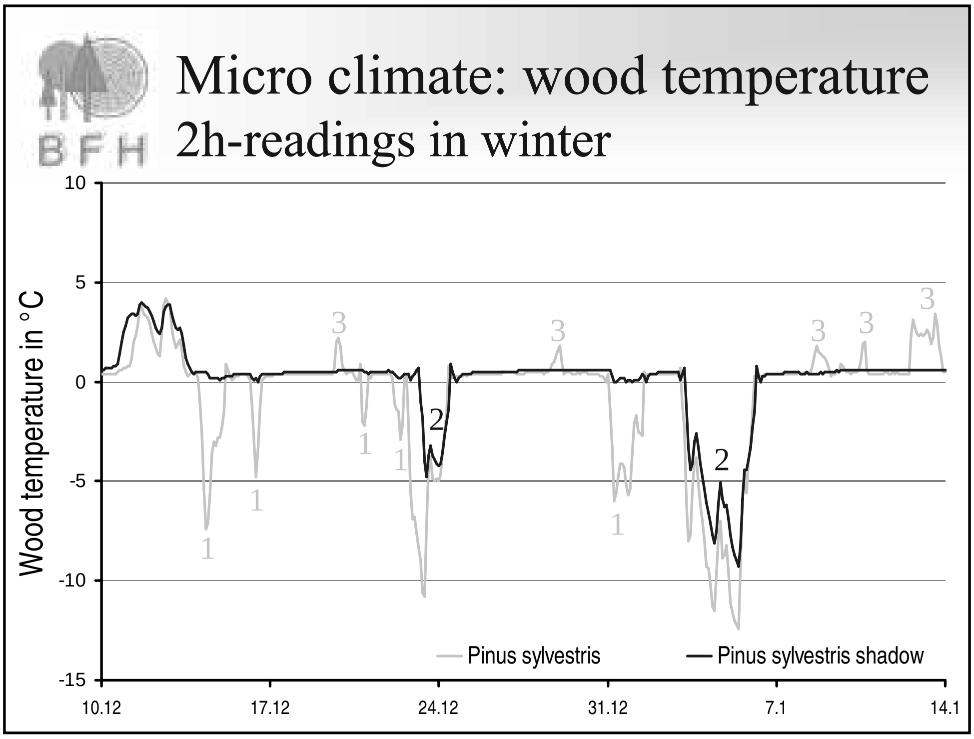


Figure 5. Course of temperature in the open (orange line) and in the shadow box (blue line) in winter

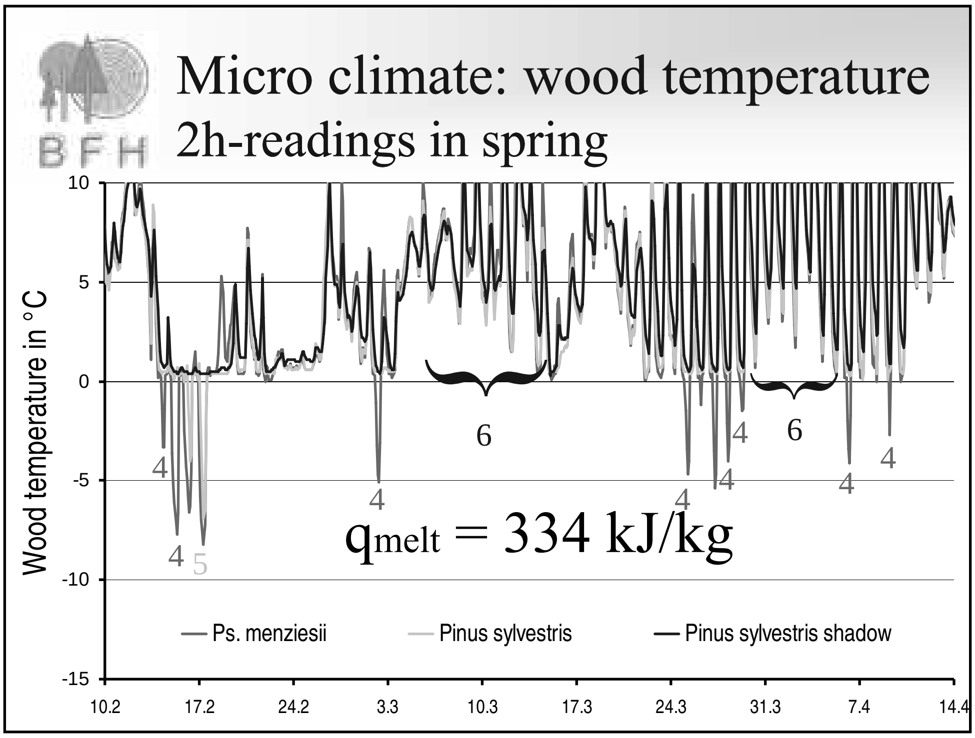


Figure 6. Course of temperature in the open (orange and pink line) and in the shadow box (blue line) in spring

The driest wood specimens (Douglas fir heartwood) went below 0°C most often (see mark 4 in Figure 6). The temperature of the wetter pine sapwood specimens went only once below 0°C (see mark 5 in Figure 6), when air temperature was very low for one week. And the wettest specimens, pine sapwood in the shadow box never went below 0°C during the whole spring of year 2001 in spite of having many days with minus temperatures as can be seen by the Douglas fir curve (mark 4 in Figure 6).

The explanation for this is simple. It is the thermal energy released while freezing, this keeps the temperature of the wood up. The more water is in the wood the more thermal energy is released. Per kg water to freeze 334 kJ are liberated which prevents the wood from going below 0°C. So the more water is in the wood, the higher is its temperature in winter.

And there is an additional effect which can be seen in cold nights, but not very cold nights, in spring and autumn. See for example the beginning of March 2001(mark 6 in Figure 6). Here, with temperatures well above the freezing point, still the wood in the shadow box has the smallest drop of temperature during cold nights. This because the textile cover sheet keeps the falling cold air away from the specimens. It is the same principle as applied in agriculture to prevent crops from night frost or cold night temperatures in spring and autumn just by covering it with a sheet. Consequently, the wood covered with the textile sheet in the shadow box always stayed at much higher temperatures at night as wood under open sky. The same effect was also seen in summer (compare mark 6 in Figure 6 and Figure 7). In summer the night temperatures in the covered box always stayed several degrees higher (sometimes up to 5°C) compared to the specimens under open sky.

About noon on hot summer days the wood temperature in the box seldom climbed higher than 35°C, whereas the uncovered specimens had more than 45 °C (see mark 7 in Figure 7). There are two causes for this effect:

1) Prevention of direct sun on the samples covered with the textile sheet acting as an awning.
2) The difference in moisture content between the wetter samples and the drier samples

The second cause becomes obvious when comparing the two samples outside the shadow box: The dry Douglas fir and the wetter pine sapwood. In spite of both samples being in the same bright sun, the wetter pine specimen stayed considerably cooler than the dry Douglas specimen (see mark 7 in Figure 7).

The explanation is simple: High mc means low temperature, because of energy consumption for evaporation of water. For one kg of water the energy of 2256 kJ is needed, this keeps the wood temperature low.

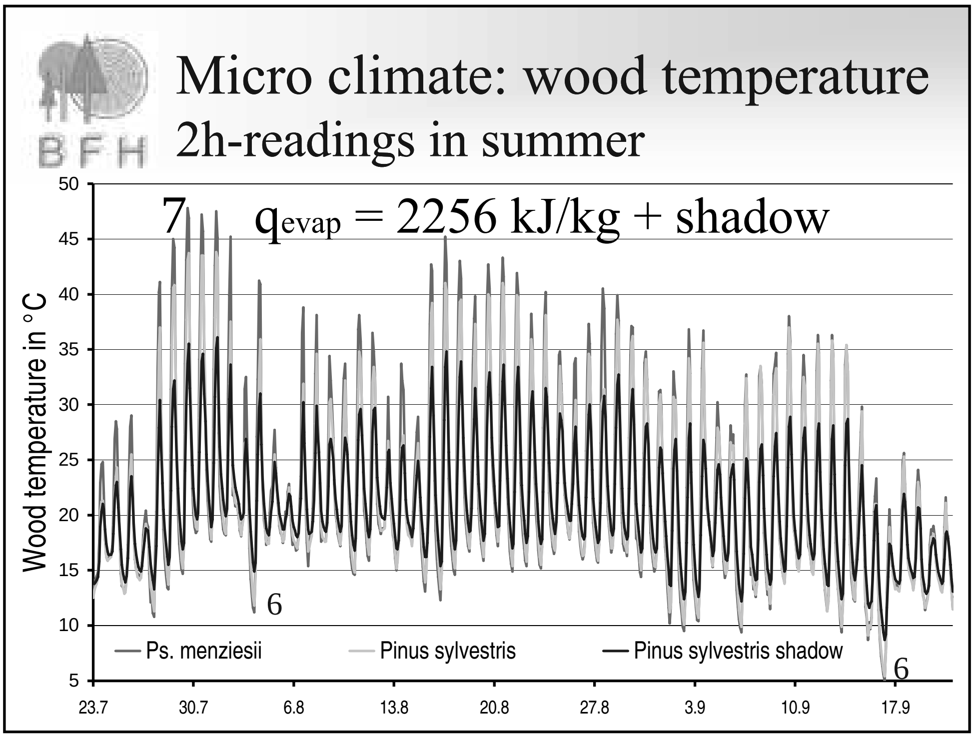


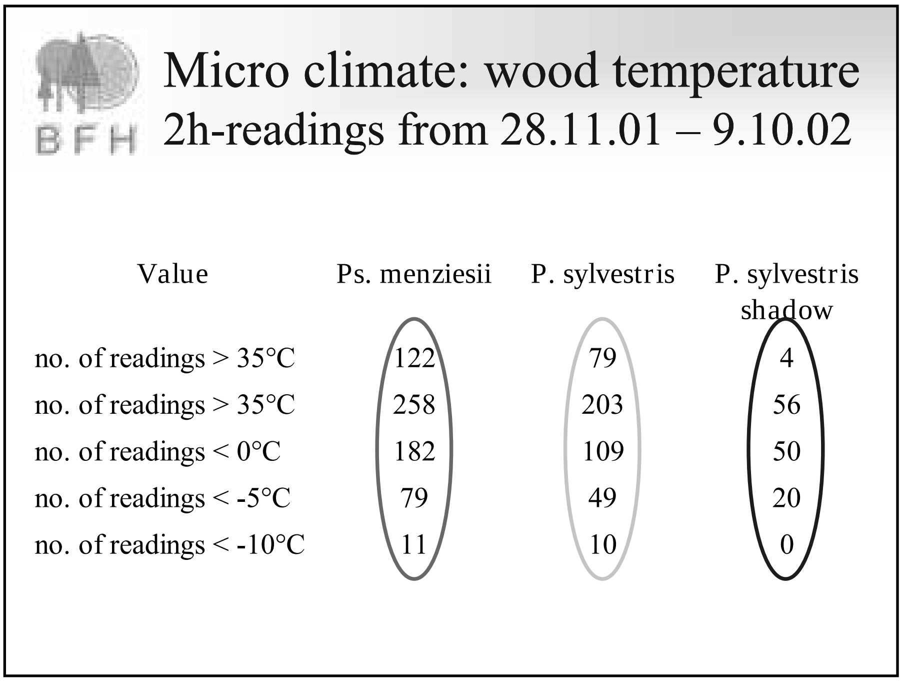
Figure 7. Course of temperature in the open (orange and pink line) and in the shadow box (blue line) in summer

Consequently when the pine sapwood (orange line) is as dry as the Douglas fir (e.g. after long sunny periods such as in the middle of September), then there is no difference between the highest temperature of Douglas and the highest temperature of pine anymore. However, the pine in the shadow (blue line) always stayed more wet than the samples outside the shadow box and therefore always had lower high-temperatures.

The numerical results over nearly one whole year of 2-hourly logged temperature readings (Table 3) can be summarized as follows:

- The amplitudes of temperature in the shadow box are much narrower than under open sky.
  This means much less extremes: less hot days and less cold nights.
- The driest wood specimen had the tallest amplitudes.
  This means the highest amplitudes = hottest days and coldest nights.

Table 3. Numerical evaluation of 2-hourly data logged temperature readings over nearly one year

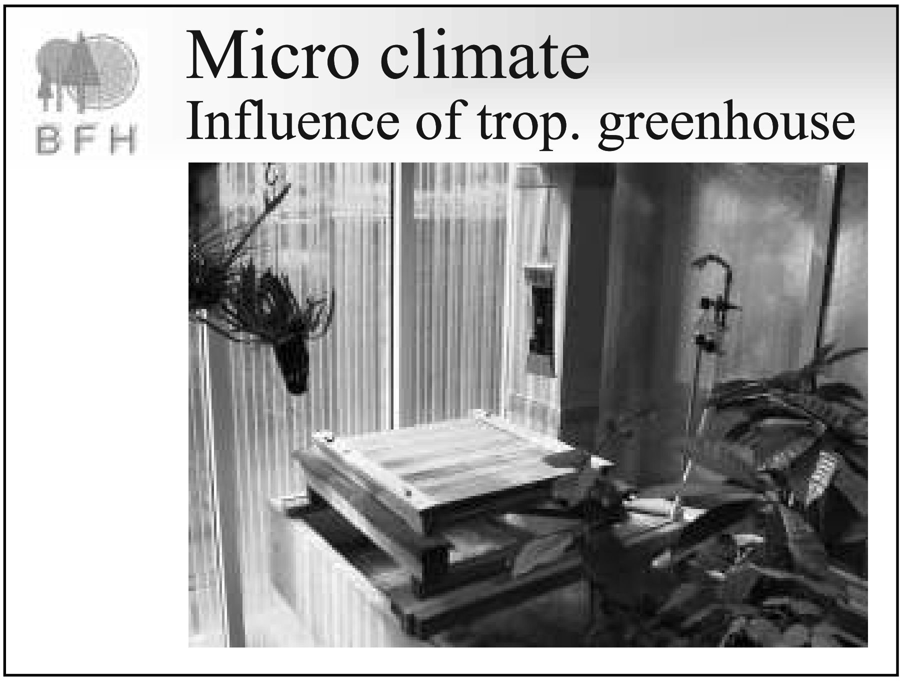


Micro climate: wood temperature
2h-readings from 28.11.01 – 9.10.02

Value	Ps. menziesii	P. sylvestris	P. sylvestris shadow
no. of readings > 35°C	122	79	4
no. of readings > 35°C	258	203	56
no. of readings < 0°C	182	109	50
no. of readings < -5°C	79	49	20
no. of readings < -10°C	11	10	0

3.4 Microclimate and decay caused by a tropical greenhouse climate

After having understood, what happens in the shadow box and what speeds up decay, it seems to be logical to think about further acceleration of decay in testing by using artificial climates, for example testing in a tropical greenhouse with an experimental set up, shown in Figure 8.



Micro climate
Influence of trop. greenhouse

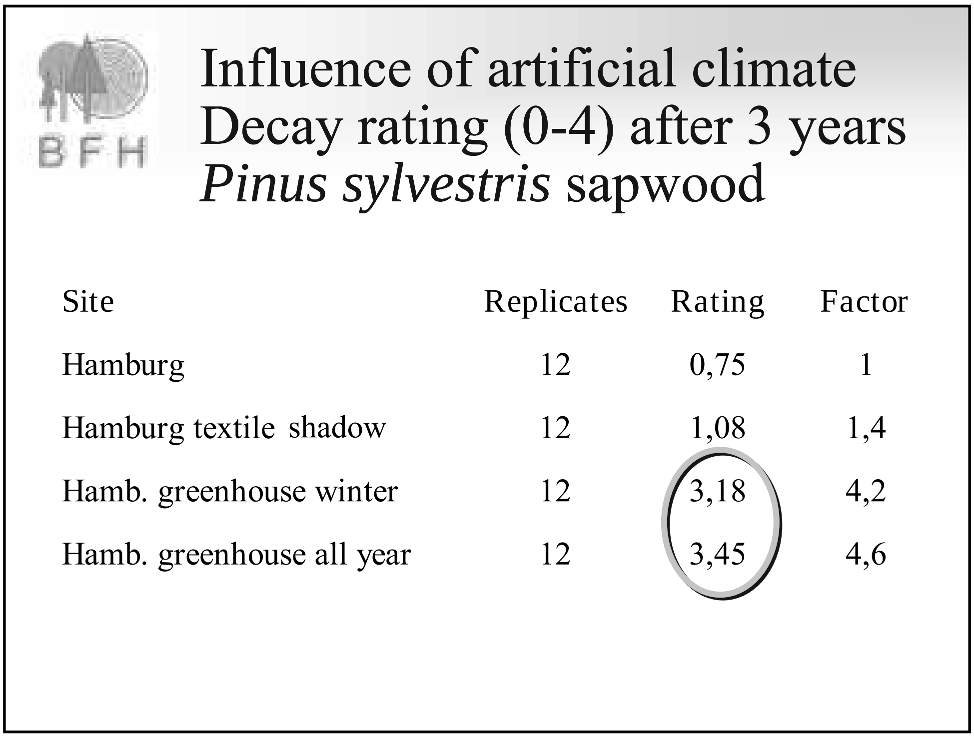
Figure 8. Experimental set up in the BFH tropical greenhouse

In this set up the rating of decay after 3 years was compared for the following 4 exposures:

- Hamburg outside (natural climate)
- Hamburg outside in the shadow box (see 3.2 and 3.3)
- Hamburg outside without frost (from 15th May to 15th October), but during the rest of the year in the BFH tropical green house
- Hamburg, all year long in the tropical greenhouse of BFH

The results are very clear by the decay rating after 3 years given in Table 4. Greenhouse extremely accelerates decay, by a factor of around 4 for *Pinus sylvestris* sapwood. Interesting is that there is so far only a slight difference between exposing the specimens all year to the tropical green house and exposing them only during the winter time to greenhouse conditions. This means that it seems to be possible to have natural infection during summer and only cut out the winter pause for the fungi by putting the set into the green house for winter.

Table 3. Decay rating from 0 (sound) to 4 (failure) for specimens in the size according to EN 252 exposed for 3 years in different natural und artificial climates in Hamburg



Influence of artificial climate
Decay rating (0-4) after 3 years
Pinus sylvestris sapwood

Site	Replicates	Rating	Factor
Hamburg	12	0,75	1
Hamburg textile shadow	12	1,08	1,4
Hamb. greenhouse winter	12	3,18	4,2
Hamb. greenhouse all year	12	3,45	4,6

4 Conclusions

Trees as well as artificial shadow increased decay activity on wood by a factor of about 2 compared to open exposure conditions. The increase of decay found in the artificial shadow box is explained by the occurrence of more non-target organisms, a higher moisture content, lower hot-temperatures and higher low-temperatures and by a longer vegetation period. The high decay rates found in a tropical greenhouse seem to be useful for accelerated testing.

5 Thanks

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TERMITES IN HAMBURG

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In 1937 termites have been detected near the harbour of Hamburg and close to tubes of the district heating system. They were identified as *Reticulitermes flavipes*, which presumably had been introduced with infested wood from the United States of America. They had spread in the surrounding of huge Court houses put up at the end of the 19th century, and there withstood all attempts of eradication. Various control measures are described: trenches were dug around the endangered houses and refilled with the soil admixed before with persistent biocides; holes were drilled into the walls of the building for pressure impregnation; additionally the ground was fumigated.

In 2000 the BAM concluded an agreement to take over control activities on the termites in Hamburg with the aim of their possible eradication. It was decided to apply baiting techniques, as these had previously shown good results in eliminating a local termite population in Berlin.

In laboratory tests with different biocides, Fipronil turned out to be the optimum substance. This could be offered in baits at concentrations that were far below the sensory threshold of the termites. The termites were unable to detect the contamination of the food offered and therefore no repellency was elicited. It was of further benefit that it was not necessary to primarily attract termites to untreated baits before replacing these with treated ones, as it is common in most of the commercially available baiting systems.
Two simple baiting methods were used: in search for the presence of termites in a certain area, stakes of pine sapwood (250 x 20 x 20 mm^3) were stuck half way into the ground. To increase attractiveness of a bait, as it is in competition to natural food sources, the wood previously had been decayed for 6 weeks by the brown rot fungus *Antrodia vaillantii*. From laboratory tests it was known that compared to untreated wood this increases the attractiveness considerably. If, after some time, many termites were found at those baits, adjacent to them a second bait type was placed into the ground: a piece of corrugated card-board was wrapped around a smaller piece of pine sapwood as described before and inserted into a plastic tube (Ø 80 mm). This was placed into a wider plastic tube (Ø 150 mm), which was put into an adapted hole in the ground. The system was covered with a lid. In this, the bait surface was increased considerably and thus gave room for a much higher number of termites.

About 500 baits of each kind were placed in the termite infested area and controlled in two week intervalls. During the first year, at maximum 80 baits were simultaneously foraged by termites, and some 50.000 termites were counted during a single inspections. However, the termite frequency at the bait stations dropped rapidly soon after. In the second year only about five percent of the original number was registered. During the following year the termite population could be kept on that low level, although additional, previously unknown infestation spots were detected. In the third year after baiting, only a few traps were still contacted by termites. This strong downward tendency, however, must not mislead to the hope of an early completion of the control measures. The biology of those termite species allows minor undetected termite groups to rapidly increase in number and restart a severe infestation. Therefore, many years of systematic inspection will be necessary until a final signal of success can be given.
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A CASE STUDY FROM THE UK OF POSSIBLE SUCCESSFUL ERADICATION OF *RETICULITERMES GRASSEI*

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Abstract: *Most efforts intended for the regulation of subterranean termite populations are directed at management rather than eradication strategies, as the latter are rarely feasible given the risk of re-infestation from surrounding areas. However, the identification of a non-indigenous population of Reticulitermes grassei in North Devon, England, in 1998, lent itself to an intensive eradication attempt as the population was highly localised. This population was situated in a semi-rural environment and there were no other known established subterranean termite populations in the UK. Other known non-indigenous outbreaks of Reticulitermes spp. related to accidental importation have been reported in Hamburg (Germany), Hallein and Salzburg (Austria), Montreal (Canada), Montevideo (Uruguay) and Santiago (Chile).*

This paper provides technical details of the initial surveys, identification, the baiting system used, genetic studies and the intensive monitoring that have been deployed since the discovery of the infestation. The paper describes how the strategy adopted, based on consumption of bespoke hexaflumuron-impregnated timber baits, led to the collapse of the populations in 1999. Absence of evidence of activity since late 2000 suggests that it is possible that eradication has been achieved successfully. Monitoring of the area known to have been infested will continue until at least 2010.

1 Introduction

Reticulitermes is the only known indigenous subterranean termite species present in Europe. Its range has been expanding rapidly in the twentieth century notably along lines of transportation such as railways and canals, particularly in France (Fougerousse & Perlade 1975). Six species of *Reticulitermes* are commonly found in Europe including *Reticulitermes grassei*, which was, until 2001, considered a subspecies of *Reticulitermes lucifugus* (Clément *et al* 2001). *Reticulitermes grassei*, is native to the Iberian peninsula and south-west France (Clément *et al* 2001), but has become established in Paris and a number of other French cities and towns following apparent introduction by human activities and transportation (Fougerousse & Perlade 1975; pers.comm., I. Paulmier, CTBA).

A possible infestation of subterranean termites, of the species R. grassei, was first recognised in 1994. The infestation was discovered as a result of extensive damage to the timber wall frames of a private house in the semi-rural and coastal town of Saunton, North Devon, in the south west of England. An attempt was made to eradicate the termites using a localised liquid treatment (based on permethrin) but in May 1998, the infestation was rediscovered. On detailed investigation termites were detected also in the surrounding soil as well as in an adjacent property. The discovery of extensive active infestations in two adjacent properties demonstrated that the infestation had become fully established in the ground and signalled the species' ability to survive and develop in the UK. The risk of further expansion of the infestation and the potentially extremely damaging consequences for UK building stock led to the initiation of an eradication programme supported by the UK government.

The isolated and discrete nature of the outbreak led to a decision by the UK Government's then Department of the Environment, Transport and the Regions (DETR) to commission a report on the possibilities for eliminating the infestation by means of an intensive. On the basis of this report, a Termite Eradication Programme was developed which provided a 12 year commitment to termite baiting and monitoring of a 1 km diameter zone, centred around the two infested properties.

During the first year of the programme, communications with local inhabitants in the Saunton area revealed verbal reports of termite sightings in one of the properties (‘Brackens’), said to go back 25 years. Assuming the correctness of these reports, it would seem likely that the termites has been imported some years, even decades, prior to the 1994 detection of termites. This would also explain the extent of the infestation found during the programme’s initial survey inMay-June 1998 and the extensive damage to timber floor, wall and roof frames in the two properties.

The UK infestation is one of several examples of infestations that appear to have resulted from accidental importation of *Reticulitermes* spp. from their indigenous areas of distribution. Other cases of accidental importation have been reported in Hamburg (Germany), Hallein and Salzburg (Austria) (*Reticulitermes flavipes*) (Fougerousse & Perlade 1975), Montreal (*R. flavipes*) (Canada) (Myles 1996), Montevideo (Uruguay) (*R. lucifugus*) (Aber & Fontes 1993) and Santiago (*Reticulitermes hesperus*) (Chile) (Cabrera & Camusseicht 1997).

This paper describes the key phases of the UK Termite Eradication Programme, now in its seventh year, in which the last evidence of detected termite activity was August 2000 (Year 3). More details on the justification and implementation of the eradication programme were outlined by Verkerk & Bravery (2001).

2 Methods

2.1 Identification

Samples of the termites from Devon were collected and identified morphologically by F. Vieau (Laboratoire d'Endocrinologie des Insectes Sociaux, Faculte des Sciences et des Techniques, Nantes, France; pers. comm., F Vieau) and via cuticular hydrocarbon analysis by AG Bagnéres (Centre National de la Recherche Scientifique [CNRS] (pers. comm., Bagnéres; Bagnères *et al* 1991). Phylogenetic analyses using mitochondrial genes and one nuclear intron region were also carried out to facilitate understanding of the relationships between the Devon termites and other species and strains of *Reticultermes* (Jenkins *et al* 2001).

2.2 Survey

At the start of the programme in May and June 1998, a detailed site survey of the affected building and environs was carried out.

As a result of the initial survey which revealed an area of active infestation of at least 75 m by 35 m, a zone was delineated within which surveys and monitoring would be conducted. This ‘eradication zone’ encompassed 29 properties within a circular area with 500 m radius, the centre being a midpoint between the two properties (‘Brackens’ and ‘The Dunes’) in which the infestation had been detected.

This ‘eradication zone’ was subdivided into three designated sub-areas: the ‘treatment zone’ (<75 m radius), the ‘intensive monitoring area’ (75-200 m radius) and the ‘buffer zone’ (200-500 m radius) (Figure 1).

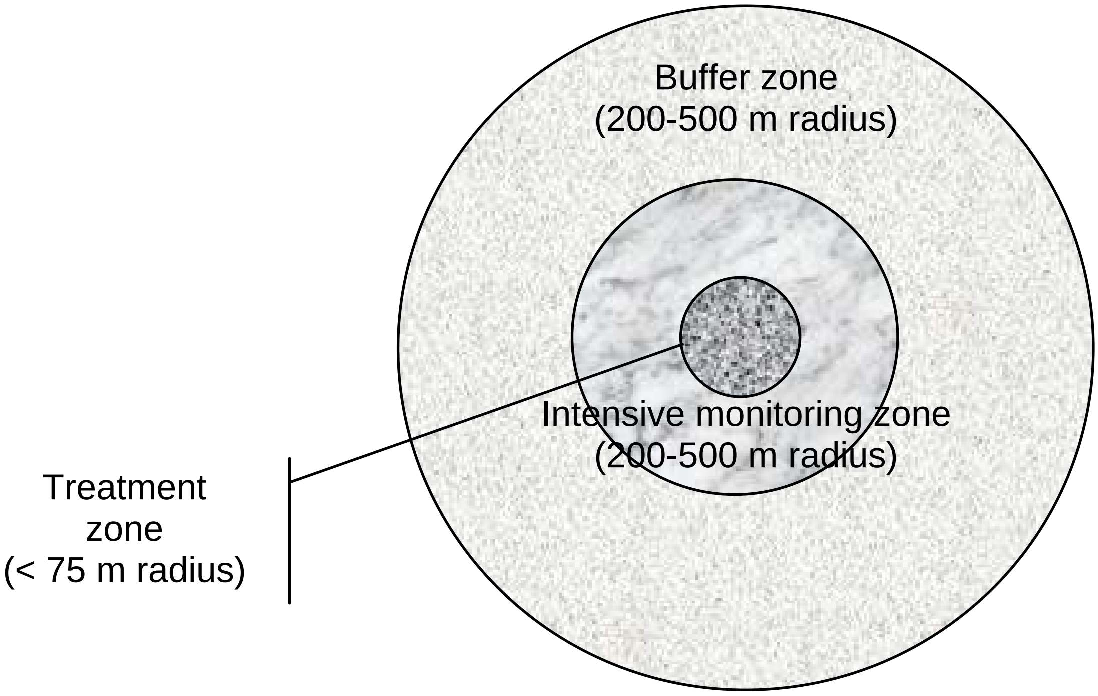

Figure 1. Zoning for UK Termite Eradication Programme.
'Treatment zone': intensive baiting and monitoring using combined baiting and monitoring devices (Sentri Tech®, Sentricon® and 'bespoke prophylactic system'), wooden slates and stakes, 'pipe and bucket system' and 'Supplementary Monitoring System';
'Intensive monitoring zone': no baiting, intensive monitoring using wooden stakes;
'Buffer zone': no baiting and less intensive monitoring using wooden stakes.

A range of different monitoring devices were employed, as summarised below:

i) Sentri Tech® and Sentricon® devices (manufactured and supplied by Dow Agrosciences). These devices contained susceptible timber baits and allow installation of hexaplumuron-impregnated cellulose matrix baits on detection of termite activity.

ii) 'Bespoke prophylactic system'; a variety of specially devised and constructed timber treatment devices comprised of 'wafers' of 1-2 mm thickness, seasoned, Scots pine sapwood, impregnated with 0.5% w/v hexaflumuron diluted in an aqueous extract of *Gloeophyllum trabeum* (brown rot fungus) (see *Laboratory development of bespoke system*, below).

iii) Wooden slats, being 150 mm long, 10 x 20 mm section, lengths of seasoned Scots pine, installed at intervals (e.g. 1 m) in 20 mm diameter holes drilled through concrete paving. Approximately 1000 in total were installed 3m apart in rows with 10 m between rows throughout the intensive monitoring zone. In both the intensive monitoring and buffer zones (Fig. 1), stakes were installed around all buildings and outbuildings.

iv) 'Pipe and bucket system': a continuous monitoring system comprised of rolled, corrugated cardboard within perforated PVC tubing (32 mm diameter), installed at an approximate depth of 100 mm beneath the soil surface around the outer perimeter of the two properties in which it was expected that any activity could be detected, linked to bucket traps (12 in total), also containing rolled corrugated cardboard. The bucket traps could be checked to determine presence or absence of termite activity. The system was installed in July 1998 and eventually removed in December 2002.

v) 'Supplementary Monitoring System' (SMS): a grid comprised of 300, one metre length, vertically-orientated, timber containing devices. Each device was composed of perforated

PVC conduit (32 mm diameter), containing two sets of paired, seasoned, Scots pine sapwood timber slats (each 500 mm in length), at 3 m centres. The upper (open) ends of each conduit were capped flush with the ground or paving levels. The system allows for installation of timber-based baits (following removal of the upper pair of slats) should activity be detected in individual monitoring devices. The system was installed in August 1999 following the collapse of termite populations in mid-1999.

The number of monitors present within the 'treatment zone' during the critical first three years of the Termite Eradication Programme is depicted in Figure 3. Note removal of untreated monitoring devices in 1999 as a means of reducing the potential for consumption of untreated baits following installation of the prophylactic baiting system.

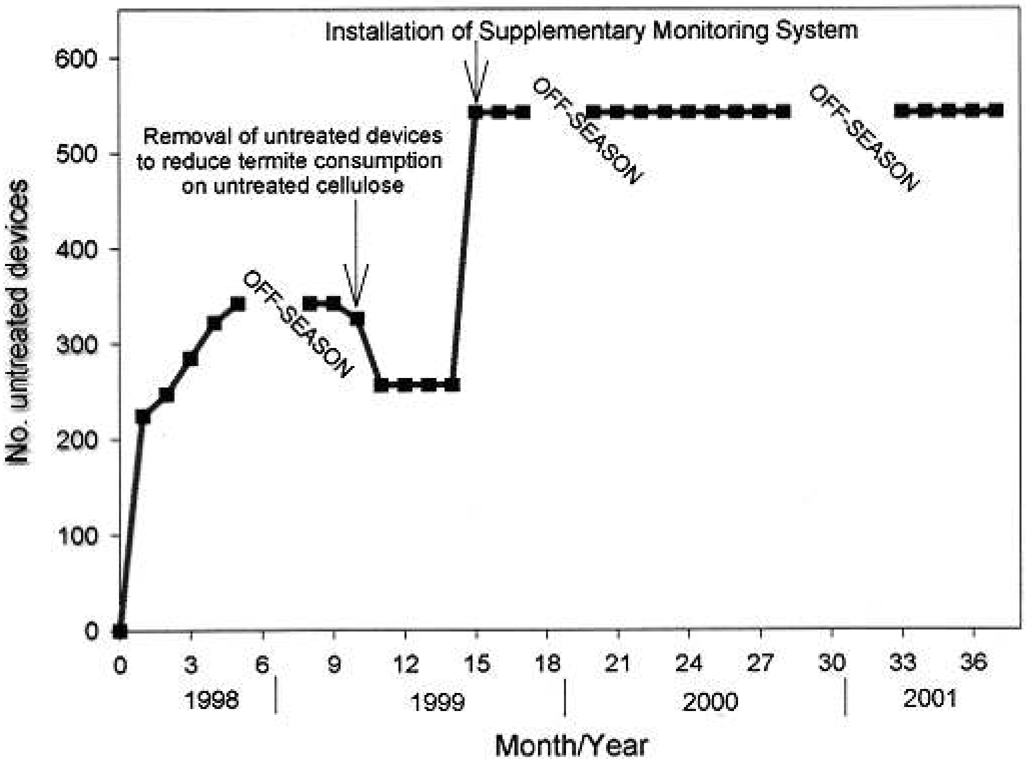

Figure 3. Number of monitors installed within 'treatment zone' during first 36 months of UK Termite Eradication Programme.

2.3 Sentri Tech® and Sentricon® baiting

The baiting system comprised of both Sentri Tech® and Sentricon® devices, was installed in July 1998. Since Sentri Tech® had been shown to be more effective in baiting and treating European termites as compared with Sentricon® (pers. comm., J-L Leca, Dow Agrosciences), Sentri Tech® devices were placed beneath, around and between the two buildings known to be infested, while the Sentricon® system was used around the perimeter of these areas. A total of 92 Sentri Tech® devices and 60 Sentricon® devices were employed.

The layout of the devices is depicted in Figure 3.

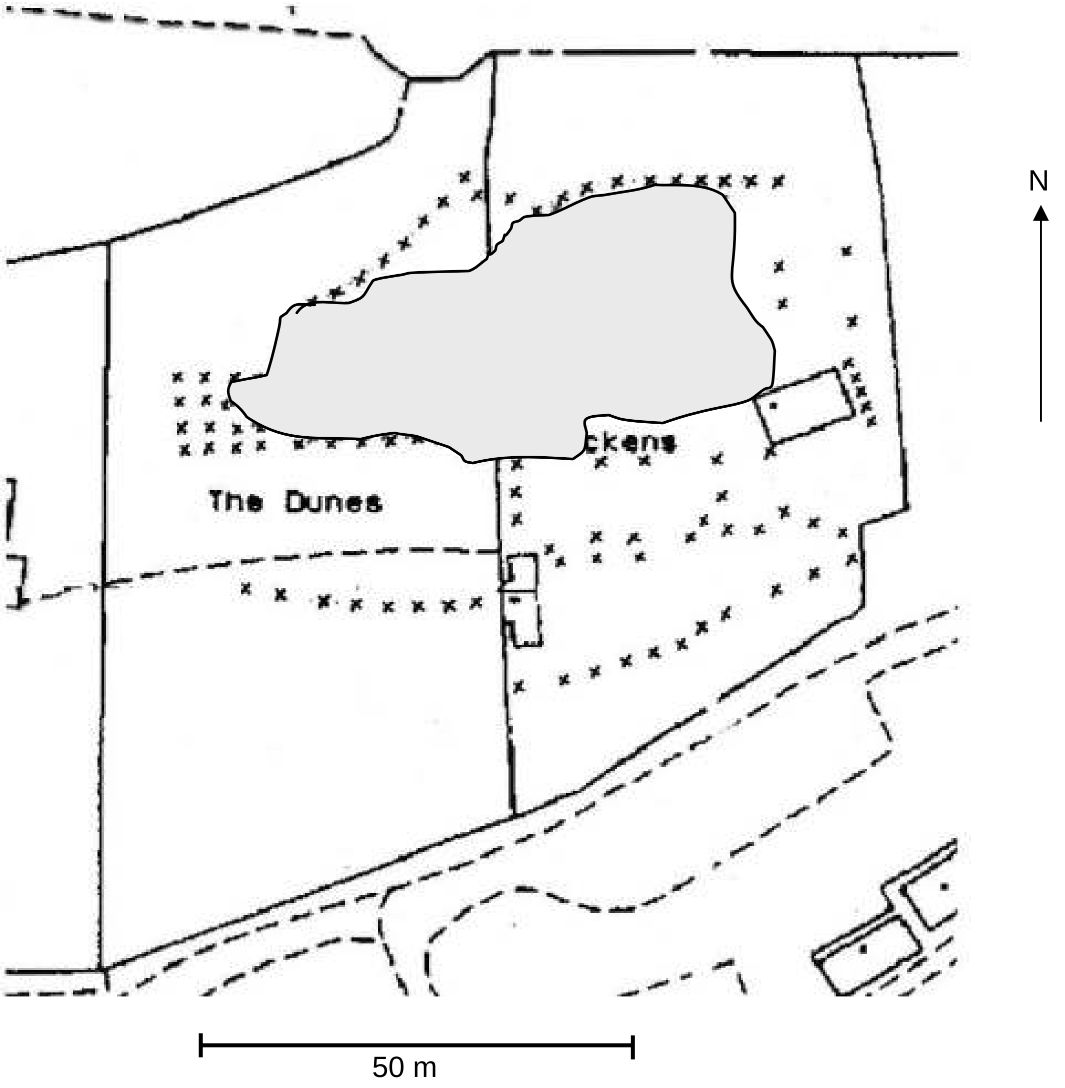

Figure 3. Layout of Sentri Tech® (marked as crosses within shaded area) and Sentricon® devices (crosses, unshaded).

2.4 Laboratory development of bespoke baiting system

Following the failure of Sentri Tech® and Sentricon® systems as a result of repellency of bait and/or matrix (September 1998; see Results and Discussion), studies were conducted with field-collected (Saunton) populations of *R. grassei* and laboratory cultures of *Reticulitermes santonensis* (BRE culture, originally collected from France). All experiments were conducted in constant environment facilities at 25±2°C.

Four major types of laboratory experiment were undertaken, viz.:

a) Choice tests. These were designed to test the relative attractiveness of different matrices in two-way choices and to allow quantitative assessment of consumption rates. The arenas for these experiments were 500ml semi-squat glass jars (*ca.* 100 mm diameter x 80 mm height) half-filled with vermiculite (medium grade). One hundred workers and 1-3 soldiers of the selected species were transferred to each jar and matrices in the form of discs (25mm diameter, 2 mm height) were placed on the surface of the vermiculite in each jar on opposing sides. Each jar was covered in aluminium foil which was pierced in a standardised manner to allow aeration and standard a volume of water was added to each jar as necessary to maintain optimum moisture. Readings were taken periodically and experimental end-points occurred after 8-10 weeks.

b) No choice tests (jar type). These involved the placement of standard blocks (50 x 50 x 50 mm) of test matrices on the surface of vermiculite within 500 ml semi-squat glass jars (see Choice tests, above) containing 100 worker termites and 1-3 soldiers.

c) Dose response and fungal attractant tests: these involved using the 500 ml semi-squat jars filled with vermiculite and 100 worker termites and 1-3 soldiers, and included paper or Scots pine discs arranged as for the two-way choice (see above) treated in one of several ways (e.g. vacuum impregnation or surface treatment, with deionised water, hexaflumuron solutions and/or *Gloeophyllum* extracts.

d) Qualitative tank tests: these involved placement of known amounts of specific matrices into culture tanks so that orientation towards and consumption of the matrices could subsequently be evaluated. These experiments approximated to a semi-field test in that they evaluated the effects of (untreated) bait matrices when placed close to relatively large colonies of reproducing termites, as compared with the quantitative experiments which evaluated responses in the absence of reproductives and a fully functioning colony.

2.5 Implementation of bespoke hexaflumuron-based prophylactic baiting system

Following failure of the commercially available, combined baiting/treatment systems (Sentri Tech® and Sentricon®) and laboratory development of a timber-based baiting system comprising 1-2 mm thickness ‘wafers’ impregnated with hexaflumuron and *G. trabeum*, a prophylactic treatment system was installed throughout areas of ‘Brackens’ and ‘The Dunes’ which were known to be infested with termites. Installation of the prophylactic system commenced in February 1999 (experimental baits) and was completed following monthly field visits in July 1999. During this time untreated baits were removed from areas known to have been infested in order to increase the probability of termite feeding on treated timber baits.

The treated ‘wafers’ were installed in one of three different ways, as follows:

a) ‘Wafer blocks’: these were composed of sixteen 150 x 50 mm wafers in turn separated by 150 x 50 mm ‘spacer strips’ positioned flush along the ‘top’ of the block. The assemblies were held together with heavy-duty rubber bands, so avoiding the need for adhesives which might cause repellency. ‘Wafer blocks’ were either placed on top of pre-bait stakes in Sentri Tech® stations or they replaced the stakes within Sentri Tech® stations on the two properties. In this latter case, the ‘bottom’ side of the block was placed on the soil surface within the station and two pairs of ‘feeder strips’ (200 x 20 mm untreated wafers) were inserted into the ground and positioned so as to contact the underside of the block. The 'feeder strips' were installed with the intention of increasing recruitment rates of termites into blocks while not comprising sufficient mass to constitute a substantial food source for the termites.

b) 'Polychain' baits: these were composed of 4-8 hexaflumuron-impregnated (as above) wafer strips (200 x 20 mm), which were cable-tied together end-to-end to form 'chains' of the required length. The 'chains' were then inserted into required lengths of perforated (4 mm diameter) PVC conduit (32 mm diameter) which had been installed horizontally into the ground from subfloor areas (only beneath 'The Dunes' south patio), vertically into the ground or through holes drilled in paving. The cable ties were linked together with nylon string which extended beyond one end of the perforated conduit so that all intact sections of treated timber could be subsequently removed and termite activity or consumption detected. In all outdoor areas, the 'polychain' baits were covered with specially constructed covers which essentially comprised ventilated plastic downpipes (150 mm diameter) to which inverted plastic pot plant saucers were glued.

c) 'Wafer crosses'. These were essentially a different configuration of the 'wafer blocks' but excluded the 'spacer strips'. They involved placing 17 alternate, treated (150 x 50 mm) wafers at 90 degrees to one another, so forming a cross shape, with each of the four arms comprising 9 wafers separated by 1-2 mm. Each assembly was again held in place using heavy-duty rubber bands around its centre. The design of 'wafer crosses' allowed easier visual detection of termites compared with 'wafer blocks' which sometimes needed to be removed from the ground or from stakes before termite activity could be verified, hence causing disturbance to feeding termites. Wafer crosses were installed in areas of major activity and each was covered with the same cover used for the 'polychain' baits.

2.6 Monitoring

Apart from the monthly field visits in 1998 (May – November inclusive), field visits were carried out during the following months:

1999: February – November inclusive
2000: February – October inclusive
2001: March, April, May, July, October
2002: March, April, May, July, October
2003: March, April, October
2004: March, April

During these visits (2-3 days each), devices were checked, maintained and treatments applied as required. Inspections were not confined only to monitoring and treatment devices, but included also timber stumps, accessible subfloor and interior timbers of buildings, as well as other cellulose containing substrates considered susceptible to termite attack.

In 1998-99, daily records were kept of maximum and minimum temperatures from a temperature probe installed in the soil at a depth of 1 m beneath the soil surface, close to the midpoint of the eradication zone.

At the start of the programme it was decided that if termite activity could be eliminated, monitoring would be continued for a total of 10 years following cessation of activity. If this can be achieved, it is highly likely that successful eradication will have been achieved.

3 Results and Discussion

Morphological identification and cuticular hydrocarbon analysis confirmed the identification of the termite species found in Devon as *R. grassei* (formerly *R. lucifigus grassei*) (pers. comm., F. Vieau, Fac Sci & Tech, Lab Endocrinol Insectes Sociaux, Nantes, France; pers. comm., Bagnéres; Bagnères *et al* 1991). Subsequent phylogenetic analysis of specimens form nine discrete samples of R. grassei from Devon revealed a monophyletic clade with some sequence differentiation among populations for COI and COII genes. This suggests that more than one maternal lineage could have been introduced to Devon, possibly either as a result of multiple introductions or one single introduction of a large 'colony' (e.g. multiple colonies in a large section of timber) (Jenkins *et al* 2001).

Temperature monitoring via the in-ground probe, one metre beneath the soil surface, during the first full year of the programme revealed a lowest temperature of 7.7°C (March 1999) and a maximum of 17.5 °C (September 1999). Accordingly, temperatures, certainly at Saunton, are unlikely to be a limiting factor in the biogeographical distribution of *R. grassei*. Other factors such as dispersal mechanisms and soil types are likely to be more important; human facilitation has clearly become a major factor in the recently expanding distribution of *Reticulitermes* worldwide (Lainé 2002).

3.1 Sentri Tech® and Sentricon® baiting

In August 1998, one month following installation of the Sentri Tech® and Sentricon® devices, 17% of the Sentri Tech® devices were found to be infested with termites, while none of the more peripheral Sentricon® devices were infested.

On installation of hexaflumuron-containing Sentry® baits within Sentri Tech® stations (which include Sentri Sol® bucket traps), termites were found to avoid feeding on the hexaflumuron-impregnated cellulose matrix rolls within the Sentri Sol® traps. Typically, the termites created a thick (ca. 10 mm) layer of mud between the top of the wooden stake in which activity arose and each Sentry® bait, effectively isolating active termites from the bait. This appeared to be the result of repellency from the matrix, the hexaflumuron or a combination of both.

Accordingly, reasons for the apparent repellency of the baits and options for a novel baiting system were investigated in the laboratory during the winter of 1998-99.

3.2 Laboratory development of bespoke baiting system

A large number of experiments and trials were conducted during the period October 1998 to February 1999. The following are some key, representative results from these laboratory tests.

a) *Choice test; comparison of untreated matrices.* The results of comparing dry weight consumption of the untreated Dow Agrosciences cellulose matrix and Scots pine discs, and a paper matrix (recycled toilet paper) and Scots pine discs, showed that Scots pine was consumed to a greater extent than the Dow matrix. However there was no difference in consumption rates between a paper matrix (recycled toilet tissue) and Scots pine discs (Table 1). The paper matrix was ruled out as being unsuitable in the field owing to its tendency to breakdown rapidly under field conditions and become infected by decay fungi.

Table 1. Choice tests comparing dry weight consumption rate by *Reticulitermes grassei* (Devon strain) on untreated matrices after 8 weeks.

Choices	Dry weight consumption (g)	Statistical significance	No. replicates
Dow matrix vs Scots pine	0.069 vs 0.186	$P > 0.05$	4
Paper matrix vs Scots pine	0.114 vs 0.104	$P < 0.05$	4

b) *No-choice test (jar type); comparison of untreated matrices.* Although there were no significant differences in dry weight consumption rates between matrices, termite aggregation on the Scots pine matrix (discs) was considerably greater than on the two paper based matrices (Dow matrix and toilet tissue) (Table 2). This suggested that the natural, unprocessed substrate was preferred as a food source and habitat over the processed substrates.

Table 2. No-Choice tests comparing dry weight consumption rate by *Reticulitermes grassei* (Devon strain) on untreated matrices after 8 weeks.

Matrix	% Survival	Mean no. termites aggregated on matrix at end-point	Dry weight consumed (g)	No. replicates
Dow matrix	65%	6.0 a	0.382 a	4
Paper matrix	74%	1.7 a	0.439 a	4
Scots pine	79%	30.0 b	0.358 a	4

Within columns, means followed by a common letter are not significantly ($P > 0.05$) different.

c) *Choice test; Hexaflumuron dose response.* The effects of impregnation of two doses of hexaflumuron and untreated controls (blank) were examined in choice tests. The results (Table 3) provided evidence of repellency of hexaflumuron to *Reticulitermes grassei*.

Table 3. Choice tests comparing dry weight consumption rate by *Reticulitermes grassei* (Devon strain) on treated and untreated matrices after 8 weeks.

Choices	Dry weight consumption (g)	Statistical significance	No. replicates
1000 ppm vs blank	0.01 vs 0.09	$P > 0.05$	4
5000 ppm vs blank	0 vs 0.09	$P > 0.05$	4
Blank vs blank	0.06 vs 0.07	$P < 0.05$	4

d) *Choice test; Consumption rate, survival and attraction of Scots pine discs treated with hexaflumuron (5000 ppm), deionised water (dH$_2$O) or* Gloeophyllum trabeum *(Gt) aqueous extract.* The following experiment with *R. santonensis* (BRE culture) demonstrated that consumption of Scots pine discs occurred in the presence of Gt, and that the treatment with Gt extract appeared to enhance aggregation irrespective whether the discs were treated with HXF or untreated (Table 4). The presence of HXF in the choice arena appeared to reduce survival rate at end-point (8 weeks). Similar results were achieved for only one or two replicates in experiments with *R. grassei*, but the lack of replication, caused by inadequate numbers of available specimens from the Devon field strain, prevented meaningful comparisons and statistical analysis.

Table 4. *Reticulitermes santonensis* consumption rate, survival and attraction by Scots pine discs treated with hexaflumuron (5000 ppm), deionised water (dH2O) or *Gloeophyllum trabeum* (Gt) aqueous extract after 8 weeks.

Treatment Choice	Mass consumed (g)	Mean no. termites present per rep on substrate at end-point	% Survival	No. replicates
dH$_2$O vs Gt	0.091 vs 0.056 a	17 vs 10	53	5
HXF+dH$_2$O vs HXF+Gt	0.014 vs 0.048 b	18 vs 50	21	5
HXF+Gt vs dH$_2$O	0.104 vs 0.036 b	66 vs 16	20	5

Studies in culture tanks (‘qualitative tank tests’; see Methods) in the Building Research Establishment’s controlled environment termite rearing facility demonstrated that colonisation rates of Scots pine baits by the Devon strain of *R. grassei* was substantially increased by presenting the timber substrate in the form of 1-2 mm thickness ‘wafers’ separated by air gaps compared with as solid blocks. These and related findings led to the development of hexaflumuron and *G. trabeum* aqueous extract treated ‘wafers’ of Scots pine as the key delivery mechanism for hexaflumuron in the novel prophylactic baiting system.

Further tank and field tests were carried out with the recycled, fungicide-free, corrugated cardboard that was used within monitoring devices (notably the ‘pipe and bucket system’) and this cellulose substrate was found to be both attractive and palatable.

3.3 Implementation of bespoke hexaflumuron-based prophylactic baiting system and on-going monitoring

Figure 4 provides a summary of the progression of the Termite Eradication Programme during its critical first three years. The Figure shows clearly the collapse of the termite populations shortly after installation of the prophylactic baiting system in 1999.

The Supplementary Monitoring System was installed as a contiguous grid system (3 m centres) across all areas known to have been previously infested. It was designed to detect activity up to a metre beneath the ground level following the unexpectedly rapid collapse of the population and to be able to be converted to a treatment system should termite activity be detected. To date, this system, along with all other forms of monitoring, have failed to detect any further activity excepting a small amount of activity that continued for about a further year in one of the properties (‘The Dunes’) beneath a patio and within a subfloor timber.

Activity of any kind was last detected in these areas in August 2000 and accordingly the monitoring frequency is being gradually reduced. Considerable effort continues to be expended to ensure that all monitoring devices remain in good condition and replacement of timber is ongoing to compensate for damage caused by weathering and fungal decay.

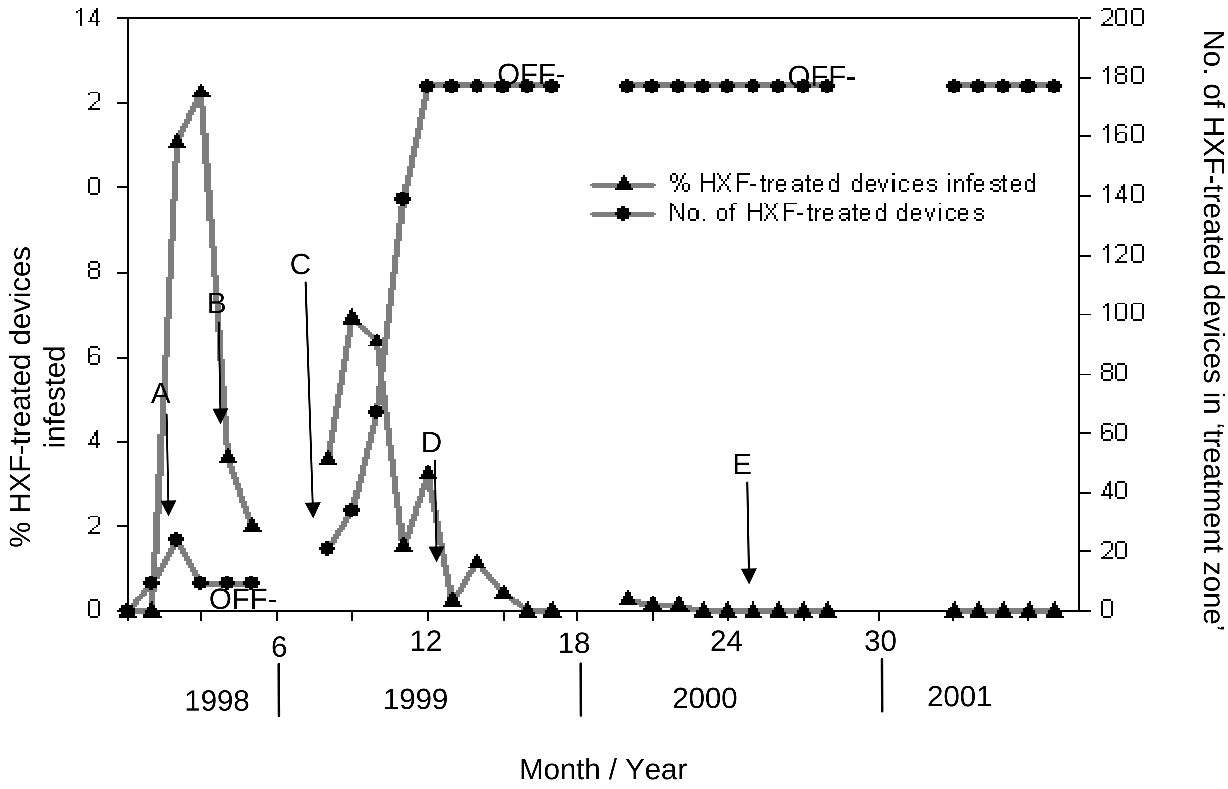

Figure 4. The UK Termite Eradication Programme 1998-2001: treated devices and % infestation within two properties found to be infested in 1998, at Saunton, Devon. Regular monitoring visits will continue until 2010.

A, *in-ground commercial treated devices removed after bait avoidance discovered;*

B, *natural end-of-season decline in foraging activity;*

C, *full-scale installation of novel prophylactic baiting system started;*

D, *mid-season apparent collapse of termite populations, prompting installation of 'Supplementary Monitoring System' (see text);*

E, *last detection of in-ground activity.*

4 Conclusions

The UK termite Eradication Programme is likely to represent one of the world's most intensive efforts aiming to eradicate subterranean termites that have been accidentally imported and subsequently have become established.

Such an intensive effort is justified as a defensive measure in UK given the immense structural damage that can result from attacks to buildings. This has been estimated at some US$11 billion annually in the USA alone (Culliney & Grace 2000; Su 2002).

The absence of any detected termite activity within the programme zone, despite regular and intensive monitoring, for approximately four years, is indicative of the success of the novel prophylactic baiting system deployed at Saunton. Additionally, the lack of detected activity may be indicative of successful eradication of the original infestation which appeared previously to have been expanding over several decades. Continued monitoring remains of paramount importance to ensure early detection and treatment of termite activity should any resurgence occur.

Although there may remain a theoretical risk that termites may have been unwittingly transported from the site at Saunton to other areas in the UK or beyond, prior to the commencement of the programme, as yet no indications of this have emerged.

5 Acknowledgments

The eradication programme is currently being overseen and funded by the UK Office of the Deputy Prime Minister. It is subject to expert evaluation by a Steering Group comprising ODPM representatives, the Building Research Establishment and Imperial College (University of London) as implementors, and representatives of Dow AgroSciences, the Forestry Commission (UK), the Health and Safety Executive (UK), the Natural Resources Institute (University of Greenwich), the Centre Téchnique du Bois et de l'Ameublement (CTBA) (France) and the British Wood Preserving and Dampproofing Association. Additional technical input from the Termite Research Group, Natural History Museum (UK), and the Department of Entomology, the University of Georgia (USA) is gratefully acknowledged. The Forestry Commission UK provided funds to support a PhD programme (1999-2002) at Imperial College London (Lainé 2002). Finally the authors would like to thank the residents of Saunton for their cooperation during the programme, especially the owners of 'Brackens' and 'The Dunes'. Further special thanks are offered to the members of the termite field team (from the Centre for Timber Technology and Construction, Building Research Establishment), notably Nancy Howard, Terry Dearling, Chris Grimes and Geraldine Lea.

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BAITING IN FRANCE

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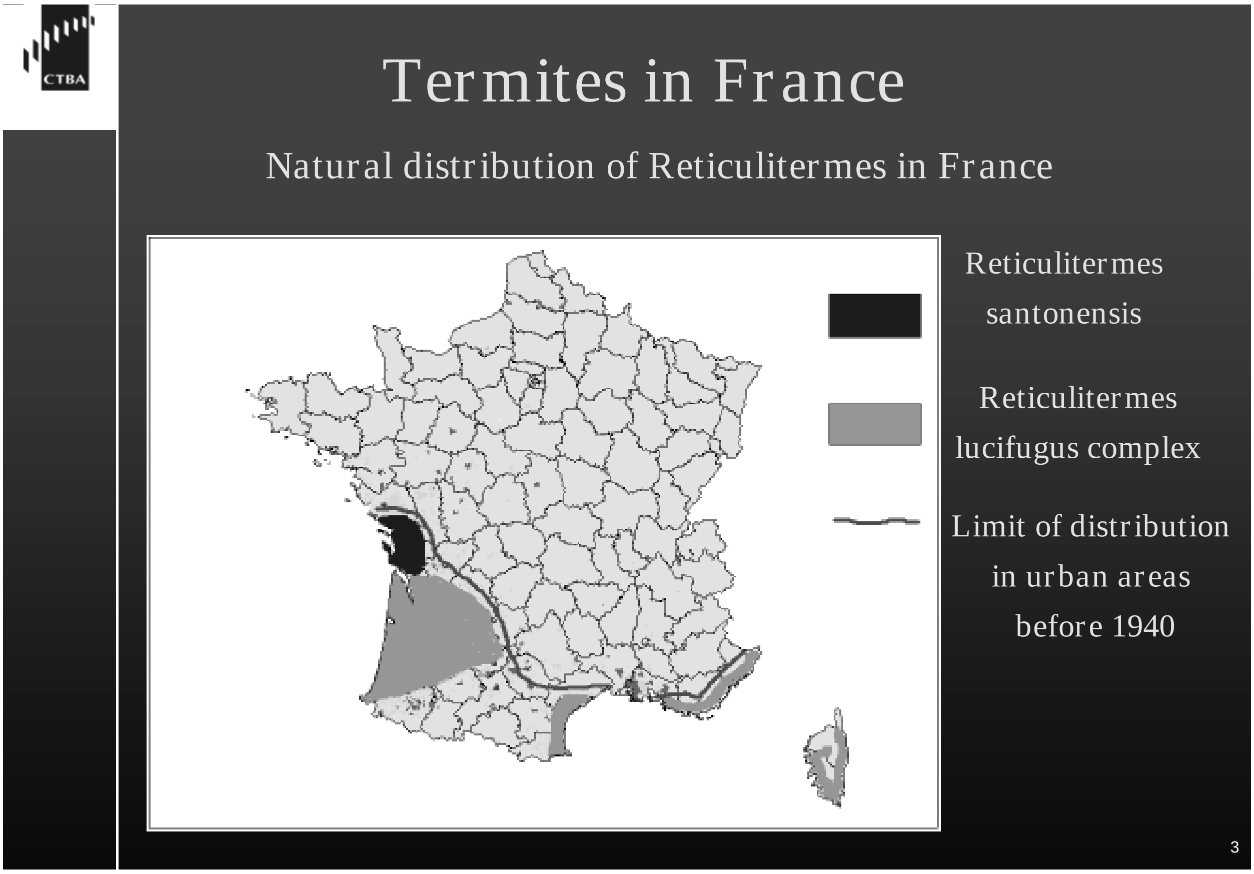
Termites in France

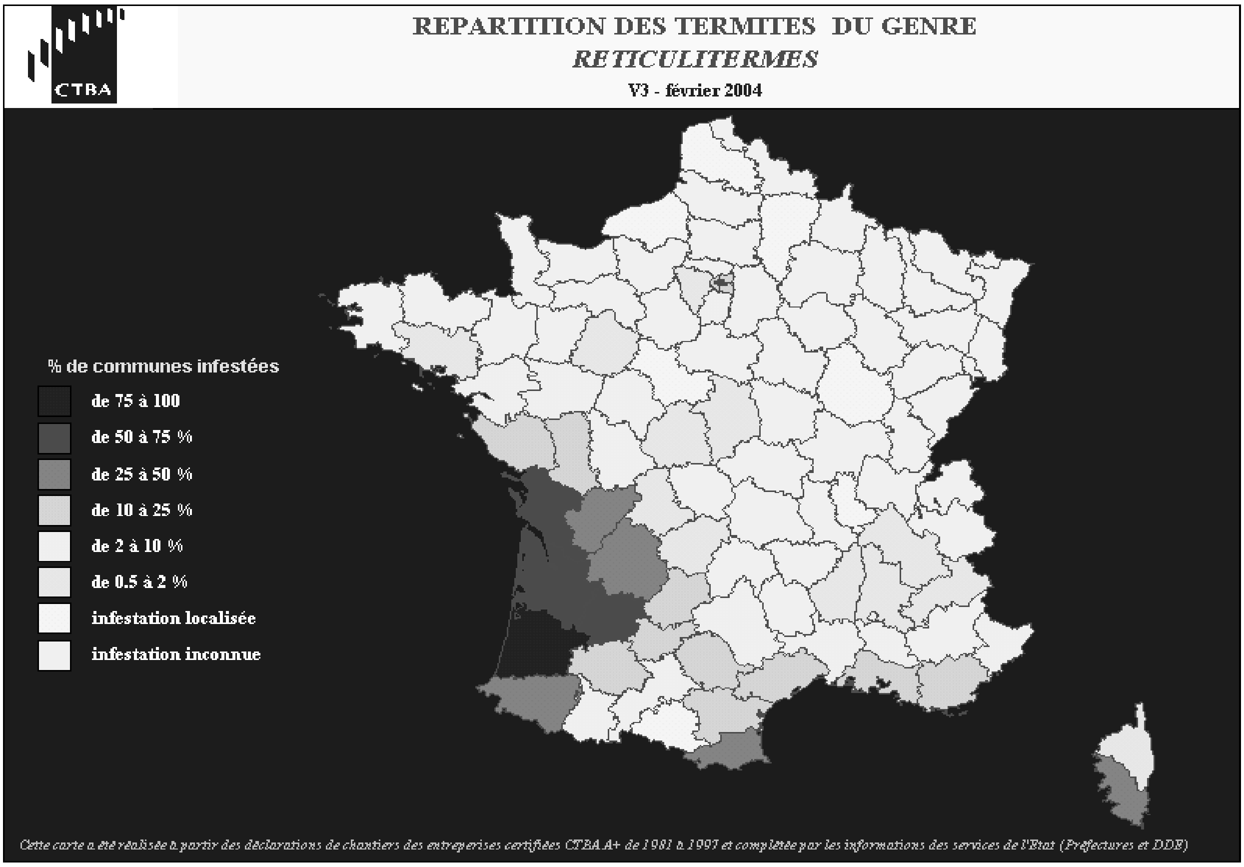
Termites in France



Termites in France

Natural distribution of Reticulitermes in France

■	Reticulitermes santonensis
▨	Reticulitermes lucifugus complex
—	Limit of distribution in urban areas before 1940

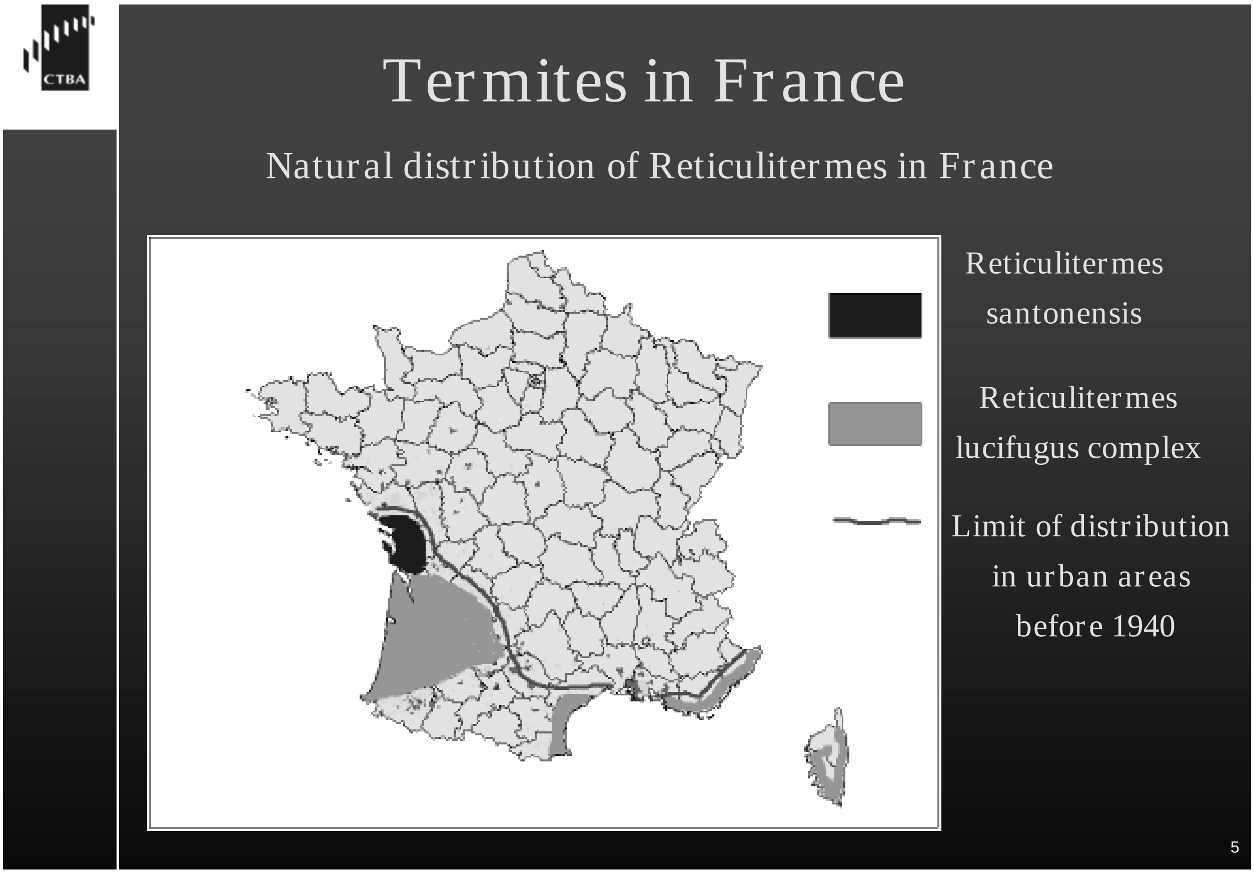


REPARTITION DES TERMITES DU GENRE
RETICULITERMES
V3 - février 2004

% de communes infestées

- ■ **de 75 à 100**
- ■ **de 50 à 75 %**
- ▨ **de 25 à 50 %**
- ▨ **de 10 à 25 %**
- □ **de 2 à 10 %**
- □ **de 0.5 à 2 %**
- □ **infestation localisée**
- □ **infestation inconnue**

Cette carte a été réalisée à partir des déclarations de chantiers des entreperises certifiées CTBA A+ de 1981 à 1997 et complétée par les informations des services de l'Etat (Préfectures et DDE)



Termites in France

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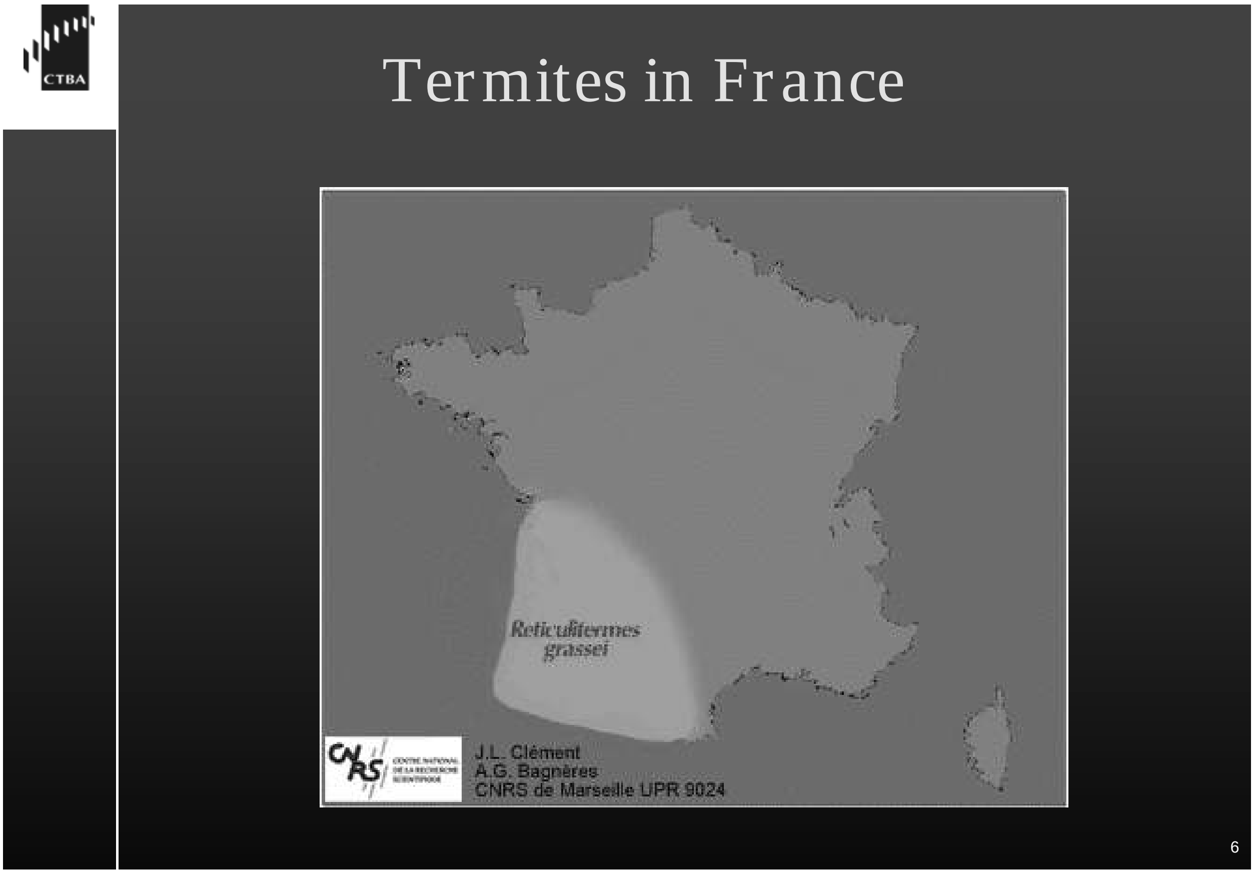

Termites in France

J.L. Clément
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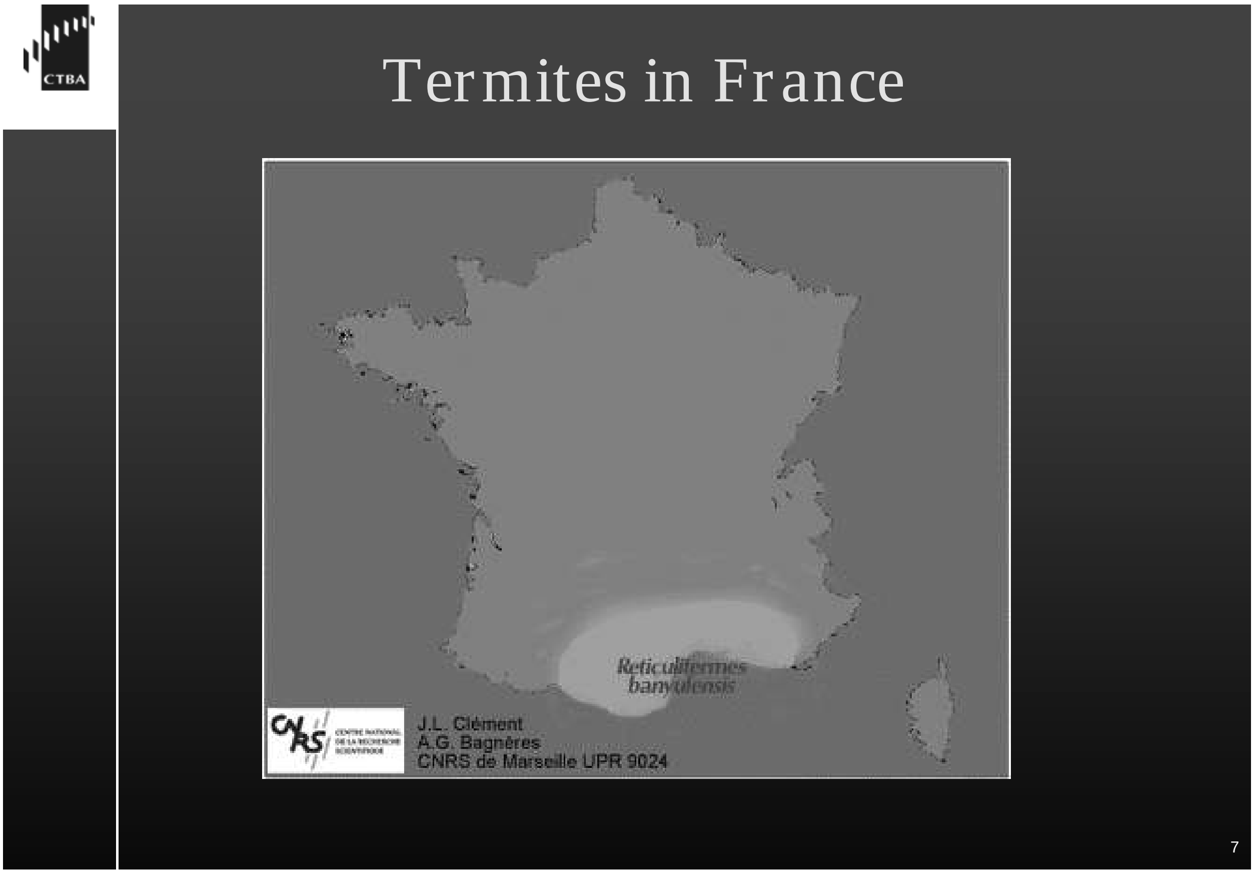

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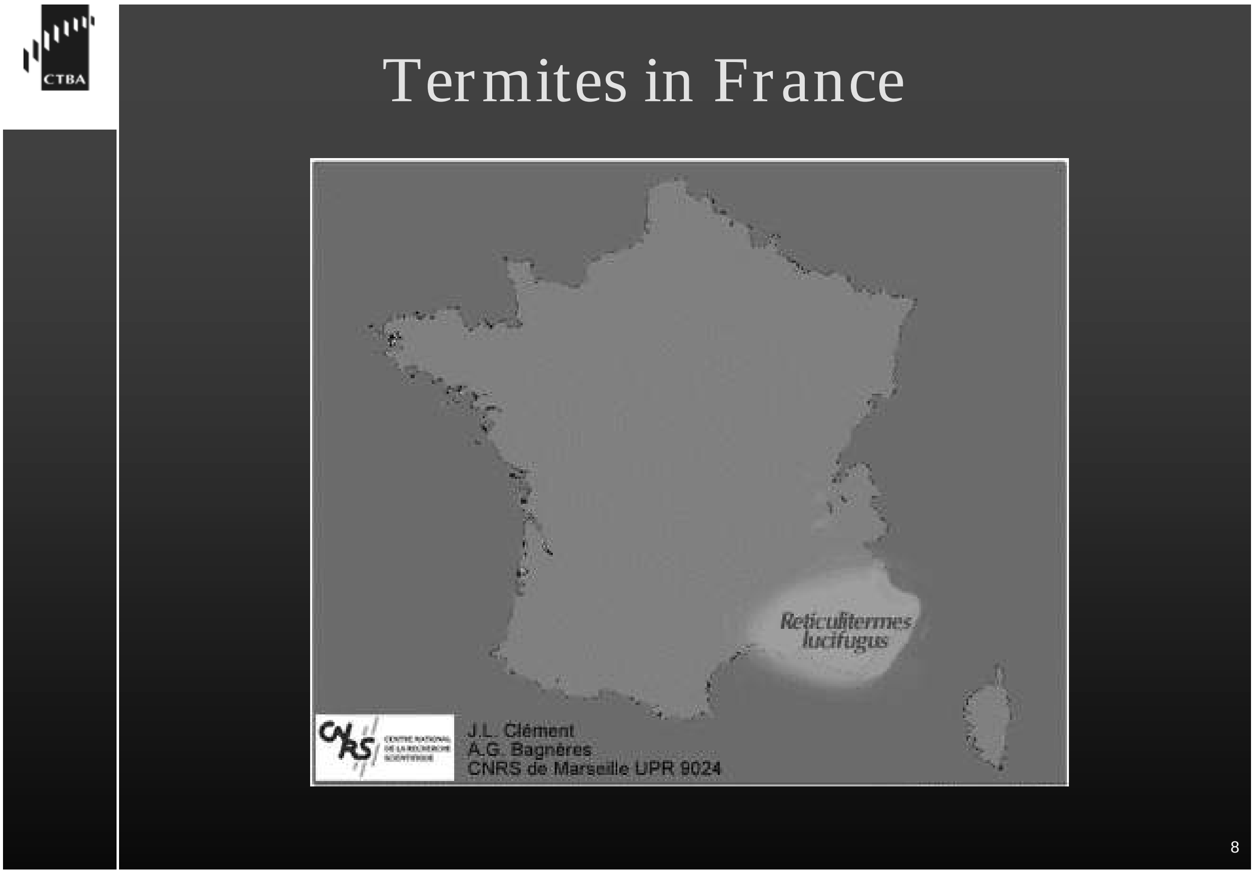

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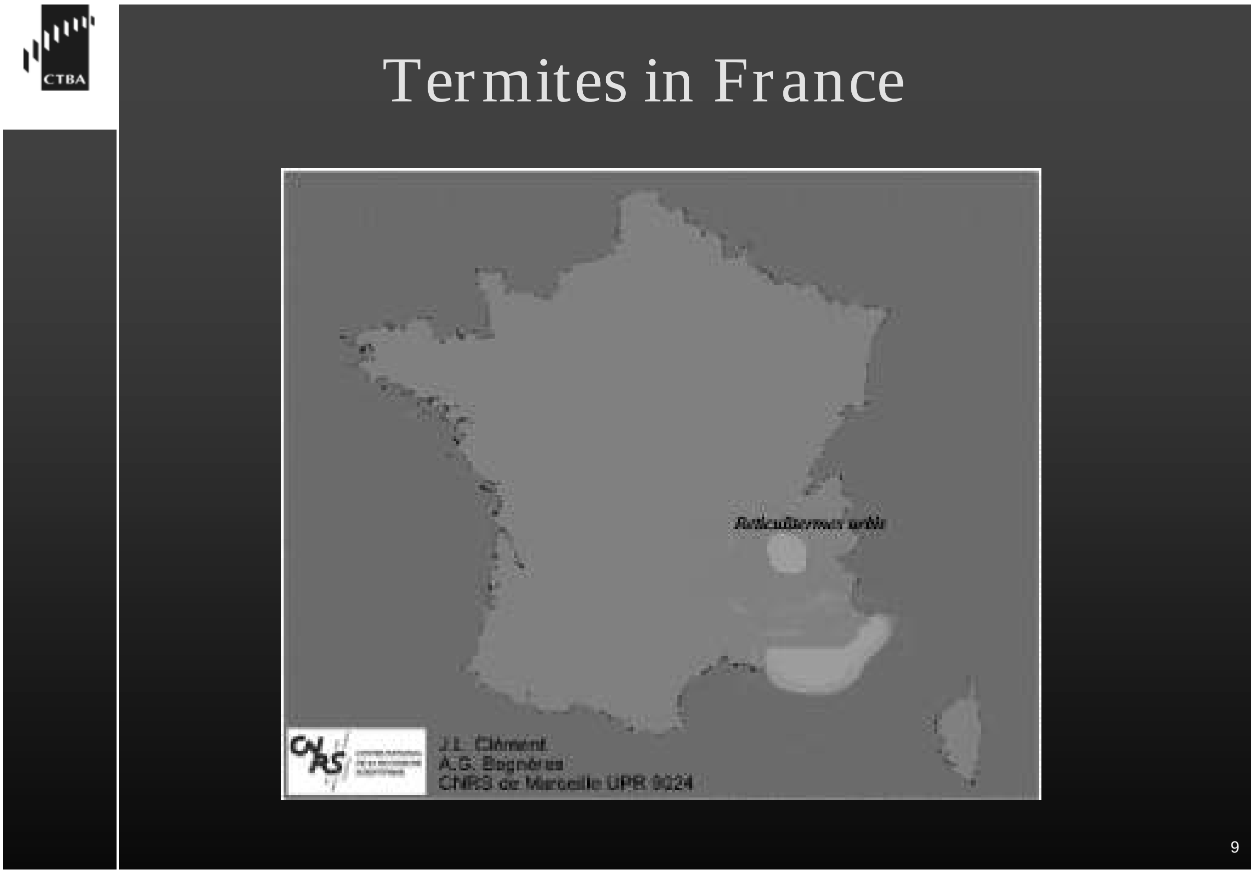

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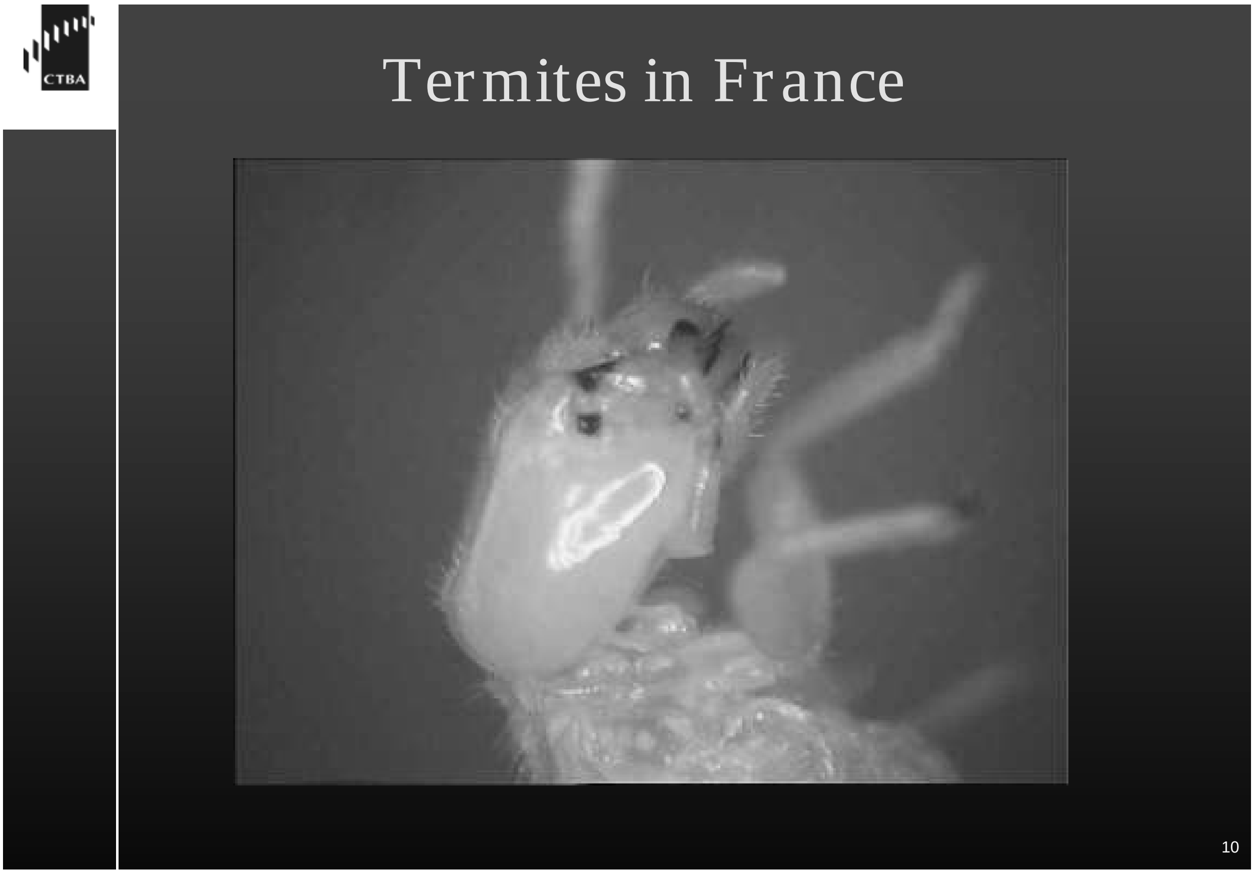

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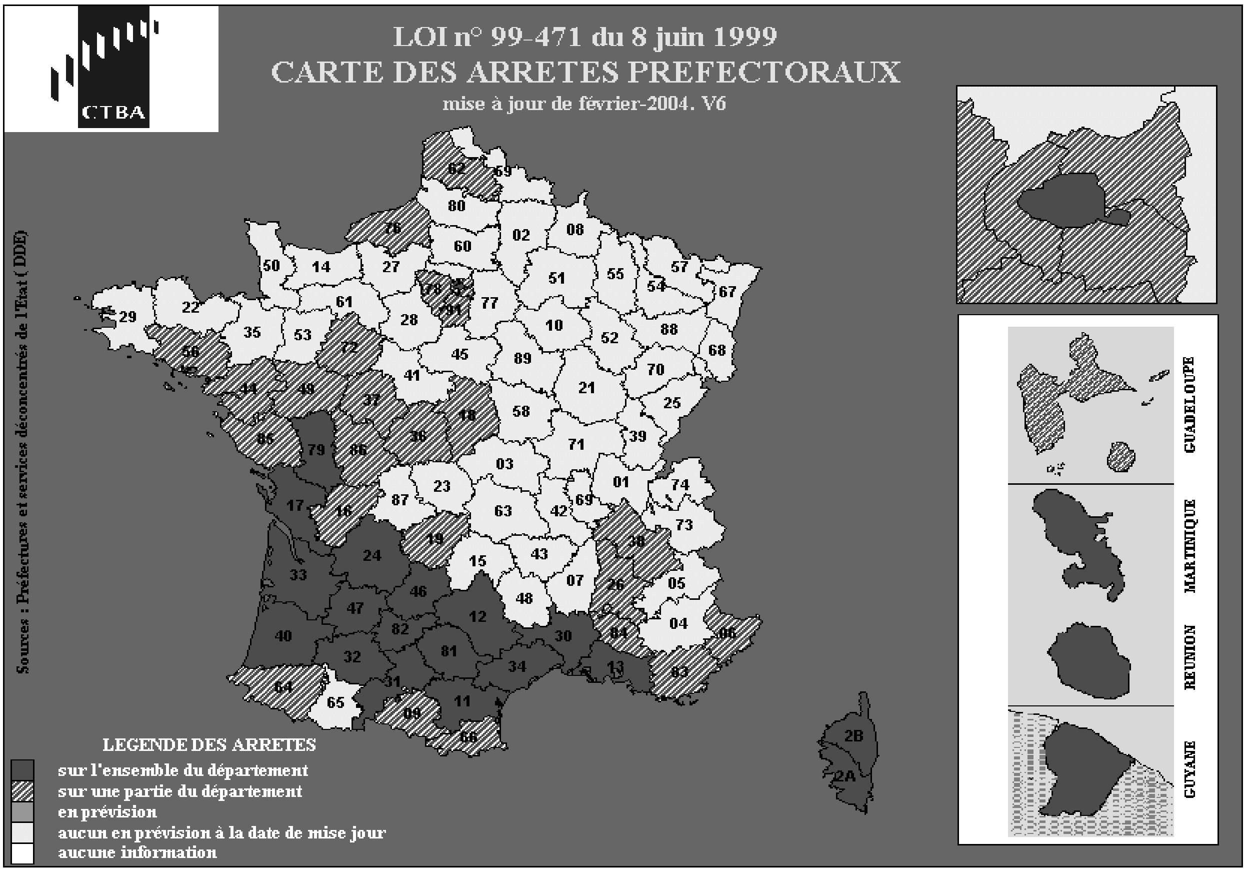


LOI n° 99-471 du 8 juin 1999
CARTE DES ARRETES PREFECTORAUX

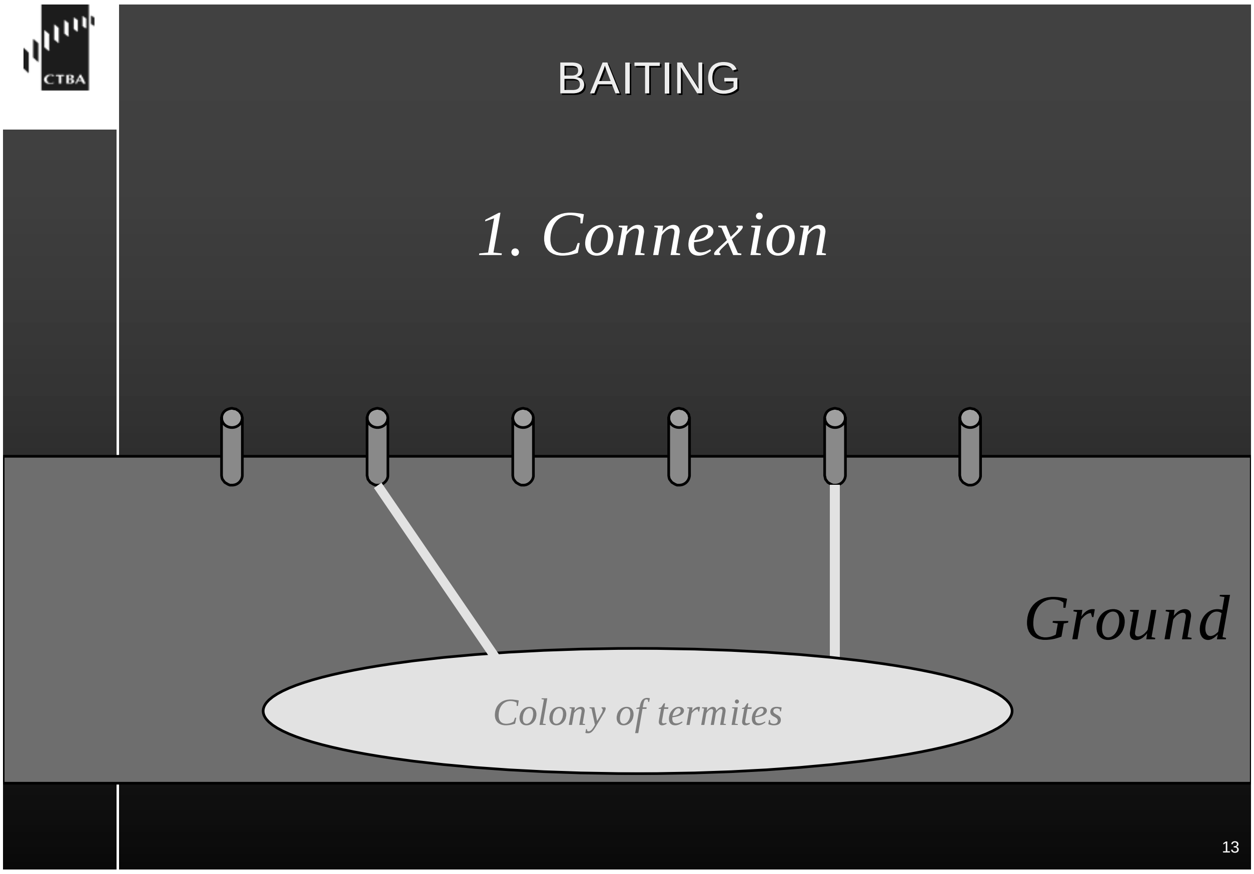
mise à jour de février-2004. V6

Sources : Préfectures et services déconcentrés de l'Etat (DDE)

LEGENDE DES ARRETES

- ■ sur l'ensemble du département
- ▨ sur une partie du département
- ▩ en prévision
- ░ aucun en prévision à la date de mise jour
- □ aucune information

Termites in France

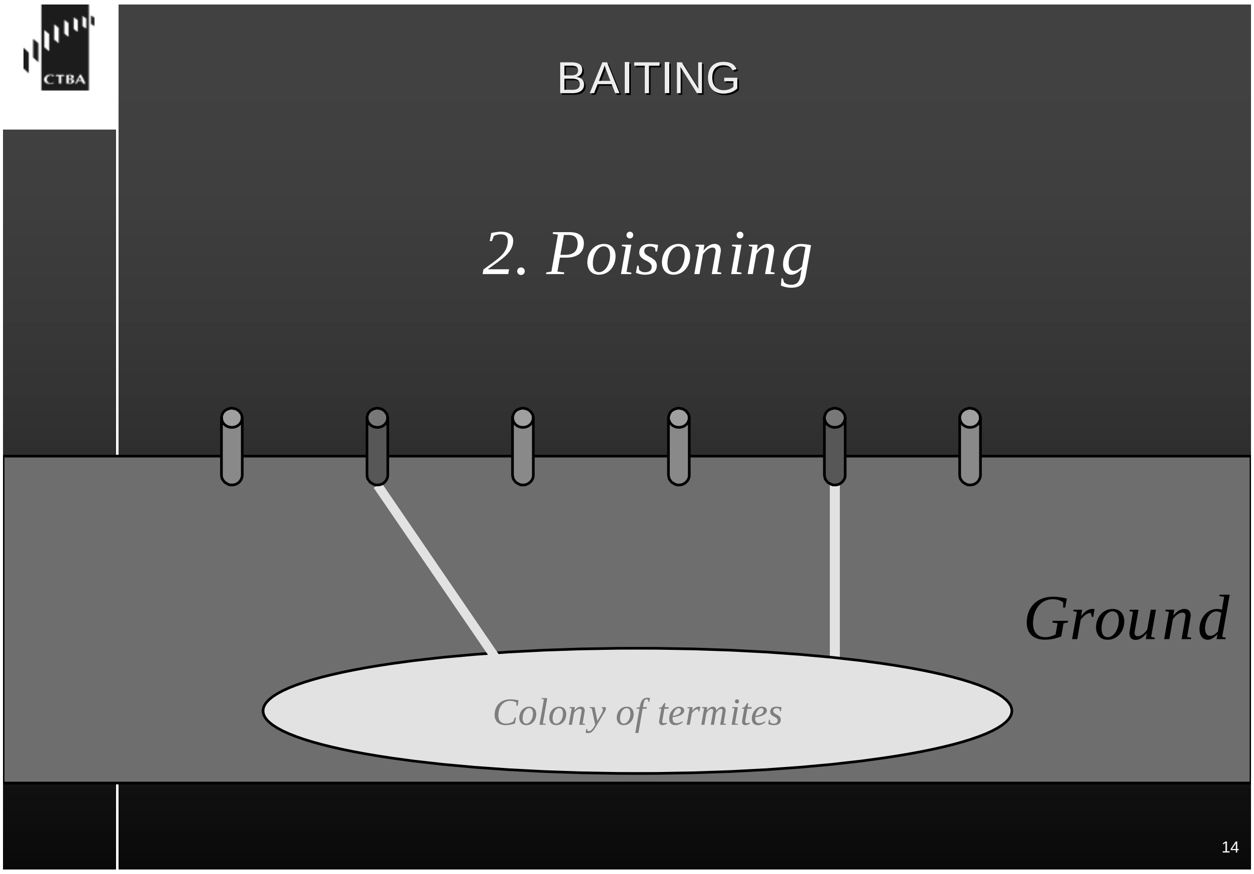


BAITING

1. Connexion

Ground

Colony of termites



CTBA

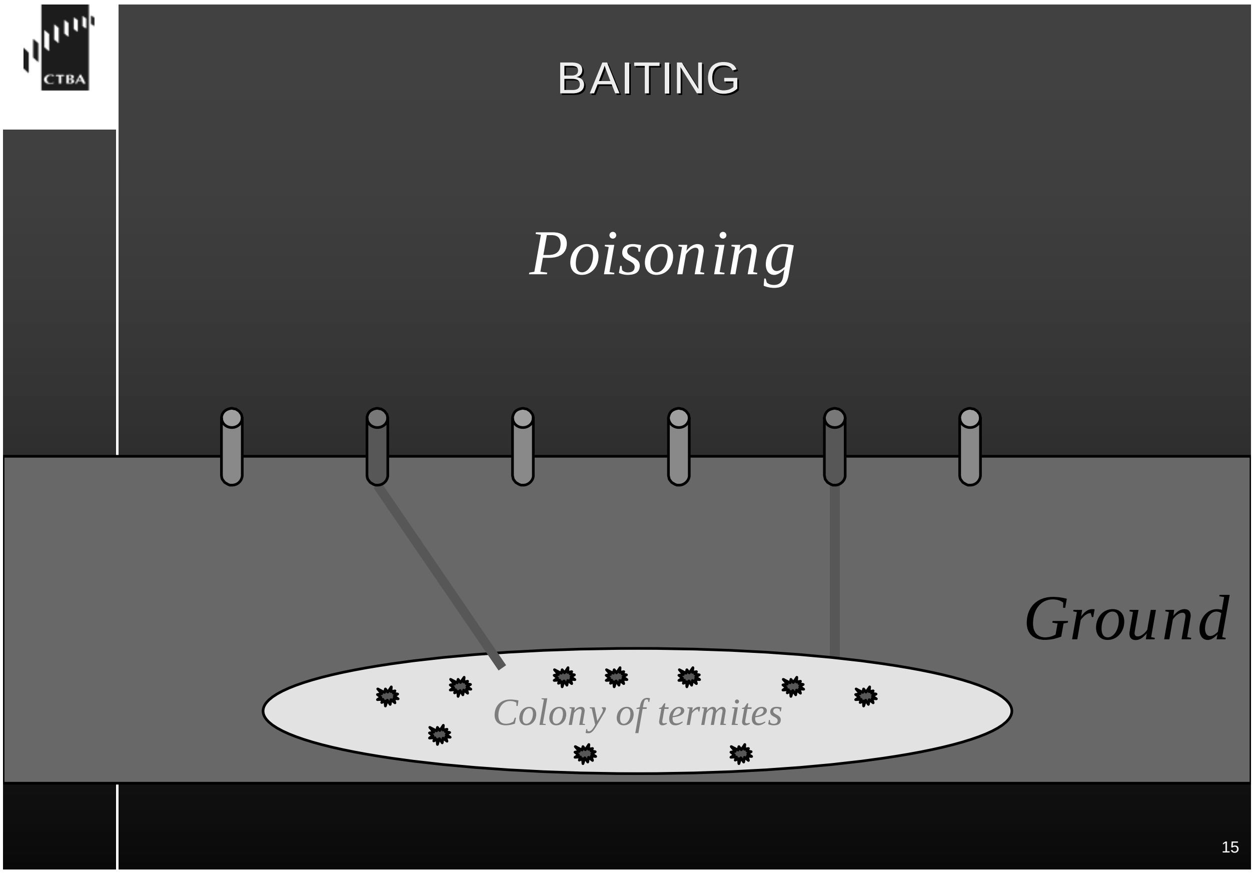
BAITING

2. Poisoning

Ground

Colony of termites

14



CTBA

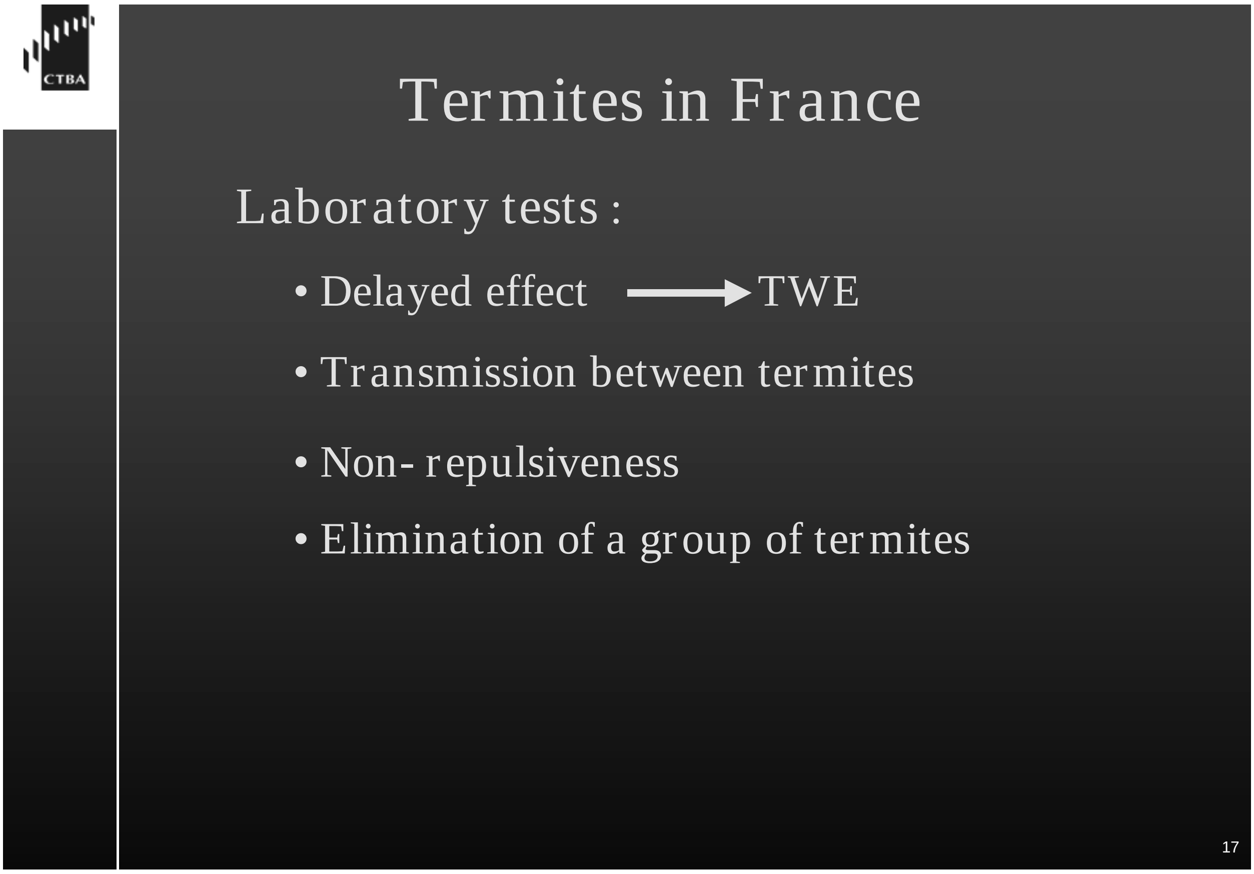
BAITING

Poisoning

Ground

Colony of termites

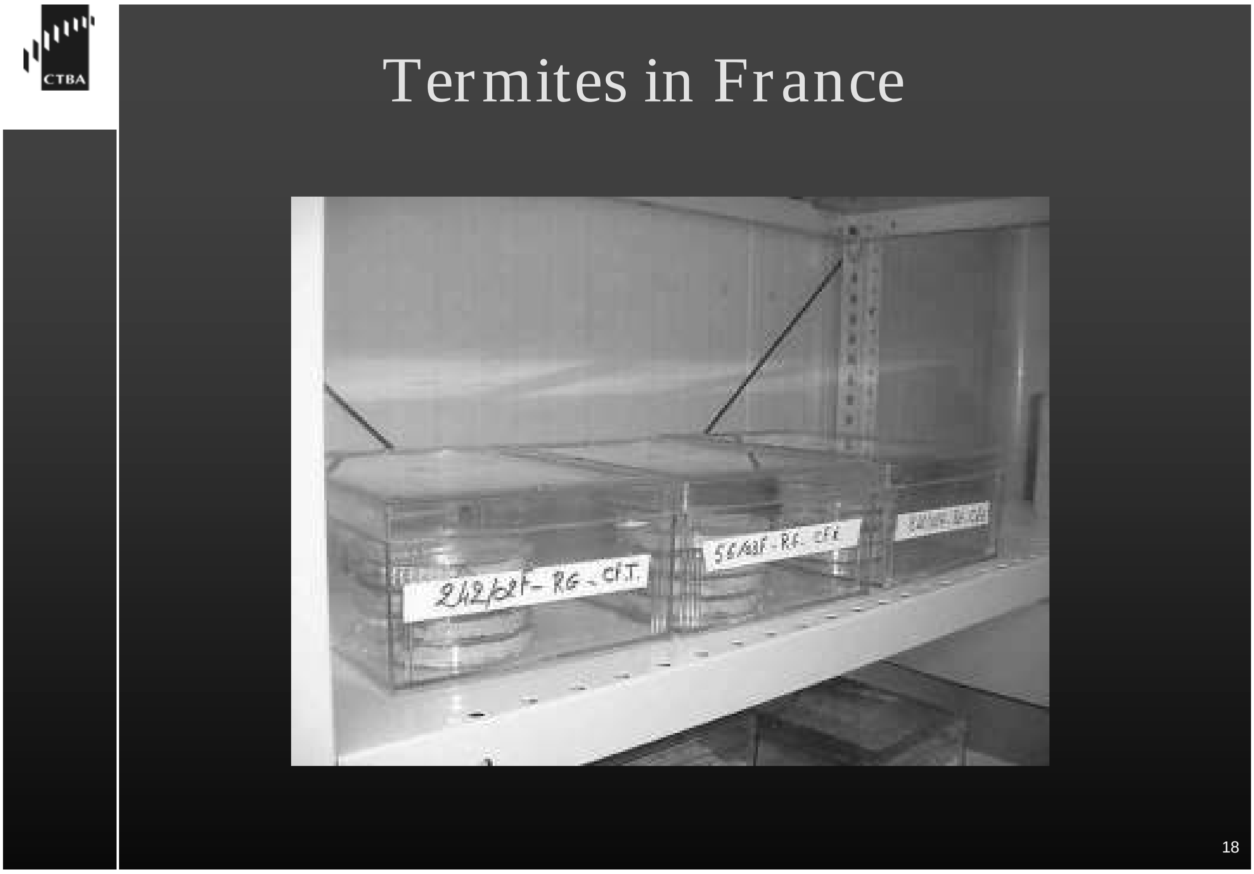
15



Termites in France

Laboratory tests :

- Delayed effect → TWE
- Transmission between termites
- Non-repulsiveness
- Elimination of a group of termites



Termites in France

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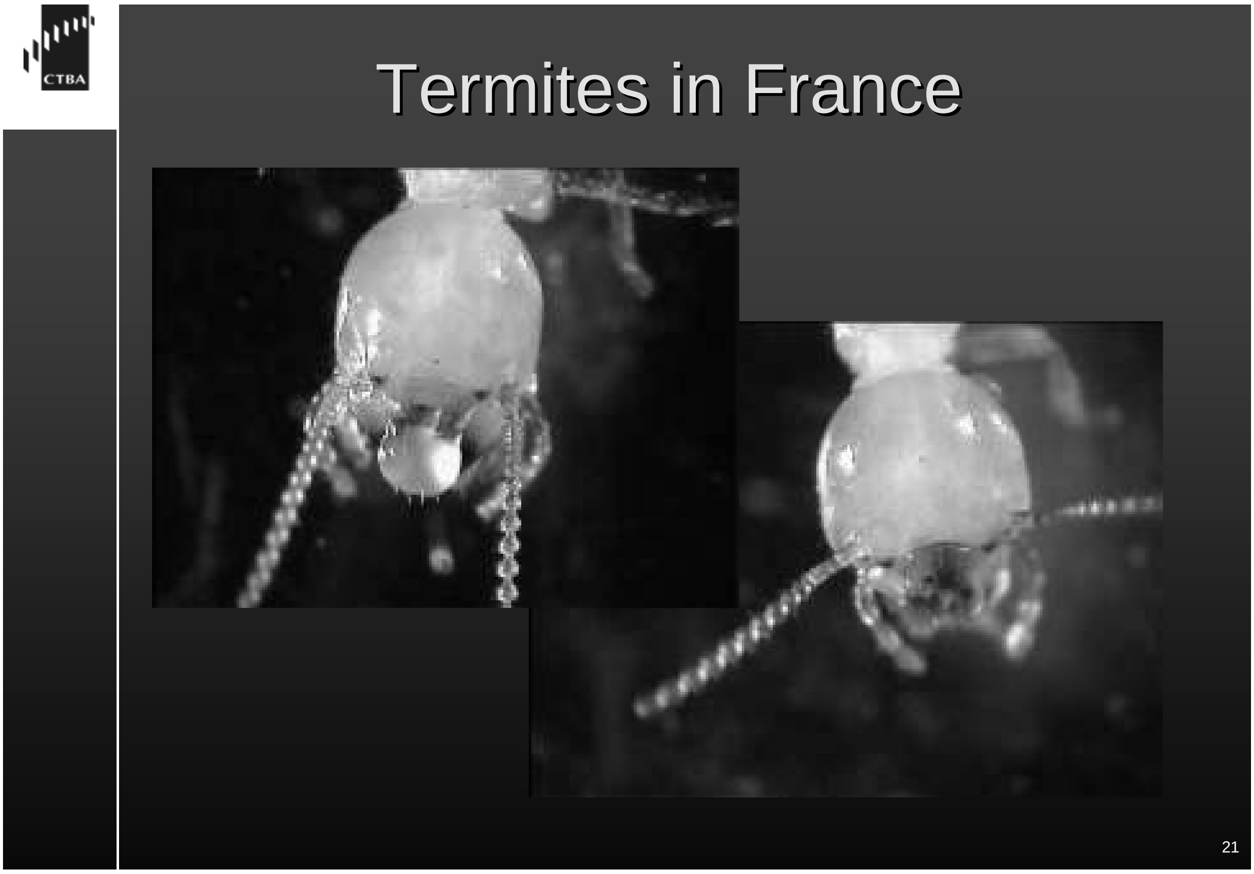

Termites in France

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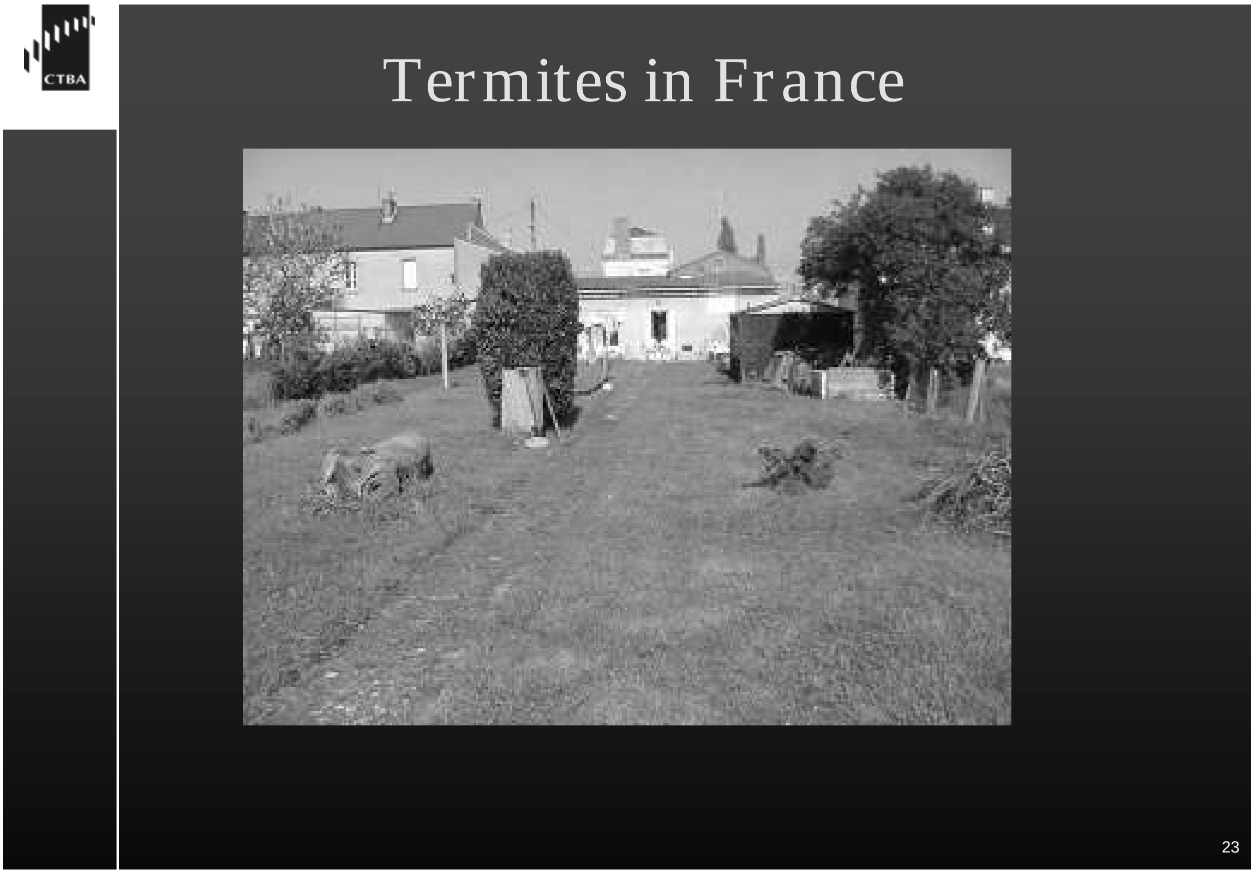

Termites in France

ONE YEAR MONITORING OF THE TRAITS OF ATTACK IN THE SOIL BY SUBTERRANEAN TERMITES

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Keywords: Reticulitermes, baits, foraging centers.

Abstract: *A baiting grid system was established in an undisturbed silvicultural plantation of Eucalyptus globulus in southern Portugal. The termite occupancy was monitored monthly during one year, and the degree of bait attack allocated to classes. The initial colonization of the baits occurred rapidly and seems to show a seasonal pattern. Reticulitermes was less active during summer months, and it started to increase after the first rainfalls and drop of temperature. The existence of two different foraging centers is suggested in light of the bait occupancy pattern, which was shown to be aggregated. Considerations are taken on the need of population-level studies for the correct implementation of baiting control methods.*

1 Introduction

The current methods of termite pest management include techniques to prevent the infestation and techniques to control it, when it occurs. Once an infestation has begun, control of termites is extremely difficult. The current control options of subterranean termites include the use of a natural durable wood species, wood treatments (chemical or not), placement of chemicals and physical barriers, and population control using baits. In recent years, this last method has gathered an increasing interest, to simultaneously monitoring termite populations and then delivering acceptable dosages of termiticide, which are confined to the bait and are not broadcast in the environment. Practical success is already claimed (Verkerk and Bravery, 2001), but evaluation continues, both from the point of view of the active ingredients and the baiting programme itself (*e.g.* Forschler, 1996; Pawson and Gold, 1996; Sheets *et al*., 2000).

However, the baiting control method is only feasible on the assumption that the toxicant will be transferred to all the colonies foraging on the infested area, which leads to the need of further knowledge on the organization and spreading of colonies, as well as on foraging behaviour of the targeted termite species. As Nutting and Jones (1990) put it, the baiting approach demands much more refined information on the biology of specific termites, particularly at the population level. But at the same time provides a higher targeted specificity of the toxicant and less environmental risk.

In Europe and in the areas where termites are present, the majority of infestations are due to the activity of subterranean termites belonging to the *Reticulitermes* genus. The subterranean termites form colonies that do not nest in the food source and are generally made of several thousands individuals that are known to be able to develop from one caste to another if needed. These termites are largely confined to an extensive subterranean gallery system and there are no visible nest structures on the surface of the ground, thus increasing the difficulty of identifying where they are. Direct observation and sampling by digging necessarily disrupts the colony (which may rapidly abandon parts of its gallery system), may generate misleading results unless undertaken on a very large scale (Bignell and Eggleton, 2000), and most of the times it is not really feasible.

The use of baits to sample subterranean termites is nowadays a common method that compared with the other available options increases sampling efficiency. It provides good estimates of frequency occurrence and can be used in this way to access cumulative bait occupation through seasons. Combined with other methods, such as mark-release-recapture techniques (MRR) for instance, can also be used to identify the foraging areas and dynamics (*e.g.* Su and Scheffrahn, 1988; Marini & Ferrari, 1998; Tsunoda *et al.* 1999; Evans, 2001). The identification of colony boundaries is a more difficult task, but assuming that subterranean termites belonging to different colonies do not forage simultaneously on the same food source, it can also be inferred by baiting methods jointed with MRR.

A one-year monitoring of the traits of bait attack by subterranean termites is here reported, as the first stages of a wider study on *Reticulitermes* population-level ecology. Understanding of the delimitation of colonies and foraging activities (including competitive interactions between colonies) are particularly relevant in the context of baiting methods as a tool of pest management.

2 Methodology

A field site was established in southern Portugal for studies on foraging behaviour and colony structure. It is a managed silvicultural plantation of *Eucalyptus globulus* Labill. located in Peroguarda (38º 11'05 N; 8º 04'33 W; alt 160m asl). During the study period, no interventions were performed there.

A baiting grid system was establish, consisting of ten rows of baits with seven baits per row with a separation of 3 m between baiting units. The baits consist of a perforated polypropylene tube, 9x30 cm, closed at the upper end with a plastic cap and sunk into the ground 30 cm. To accommodate the tube, the ground was first bored and the tube filled with a bait of several pieces of laminated, pesticide-free maritime pine (*Pinus pinaster* Ait.). The baits occupancy was evaluated monthly according to the classification system given in table 1.

Table 1:Ocupancy classes allocated in the evaluation of the baits.

Occupancy class	Description
0	No termites and no signs of attack
1	Initial colonization state with few termites exploring the surface of wood; No termites present but showing signs of attack
2	Intermediate colonization state, with termites already installed but in small numbers
3	Colony actively installed in the bait, but wood still with superficial attack
4	Colony actively installed in the bait and wood fully attacked; no termites present but wood fully attacked.

A test for complete spatial randomness (Dixon, 2001; Krebs, 1989) was applied, considering traps occupied as an event occurring. It aimed to verify if the pattern of that event was statistically at random. The test proposed by Clark & Evans (1954) (*in:* Krebs 1989, Dixon, 2001) for complete spatial randomness, based on nearest neighbour distances, was applied. Due to the absence of a boundary strip and to avoid bias and edge effects, the Donnelly's modification (1978) (*in*: Krebs, 1989; Dixon, 2001) to the above test was taken into account. The significance of the deviation from random was tested at $\propto$ = 0,05.

3 Results

The initial colonization of the baits occurred rapidly, within the first month of exposure, 16 out 70 of baits were occupied (approx. 23%). As we can see in Figure 1 the increase of the number and degree of attack on the baits colonised was at a lower rate.

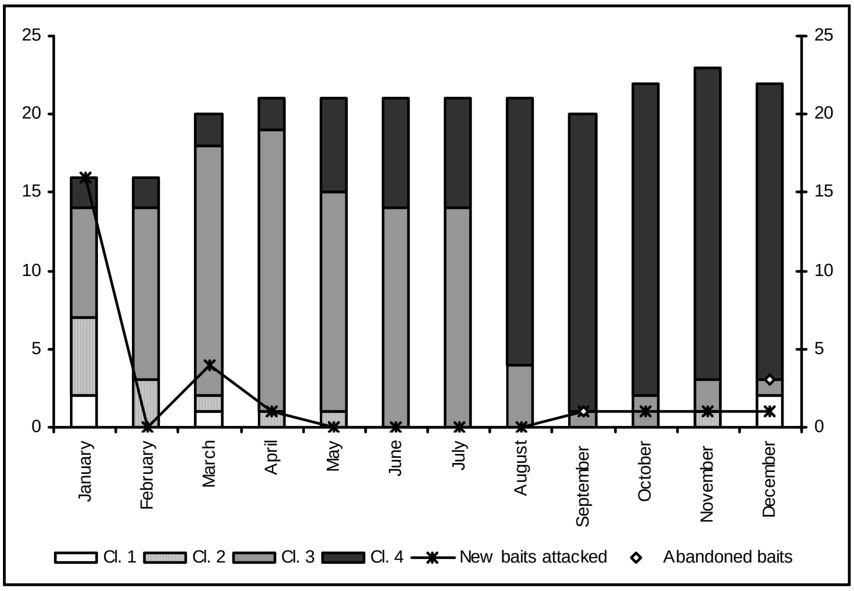

Figure 1: Changes in the termite occupancy classes in the baits

In June, July and August, when the average maximum air temperature was above 33ºC (Figure 2) and the rainfall was almost absent, the number of new baits attacked was null but an increase of qualitative class attack occurred, particularly in August. In September the number of new occupied baits starts to change, coinciding with a decline in temperature and an increase in rainfall (Figure 2).

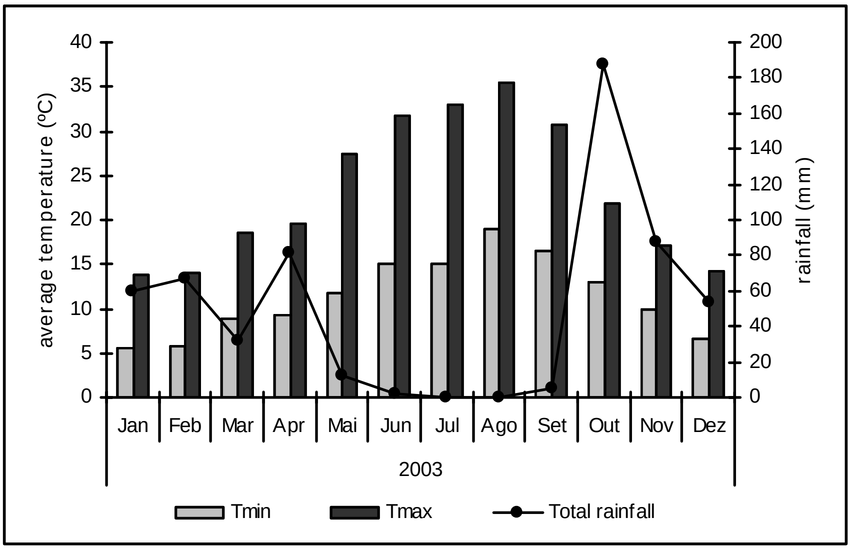

Figure 2: Monthly average minimum temperature (Tmin.), maximum temperature (Tmax.) and total rainfall for nearest meteorological station to the field site (Beja).

The cumulative data from monitoring suggests the existence of two spatially separated foraging centers, within the sampling area. Taking as an example the tenth month of exposure (Figure 3), the pattern of baits occupancy proved to be aggregated (aggregation index of 0,470), and significantly ($\propto$=0,05) deviated from randomness.

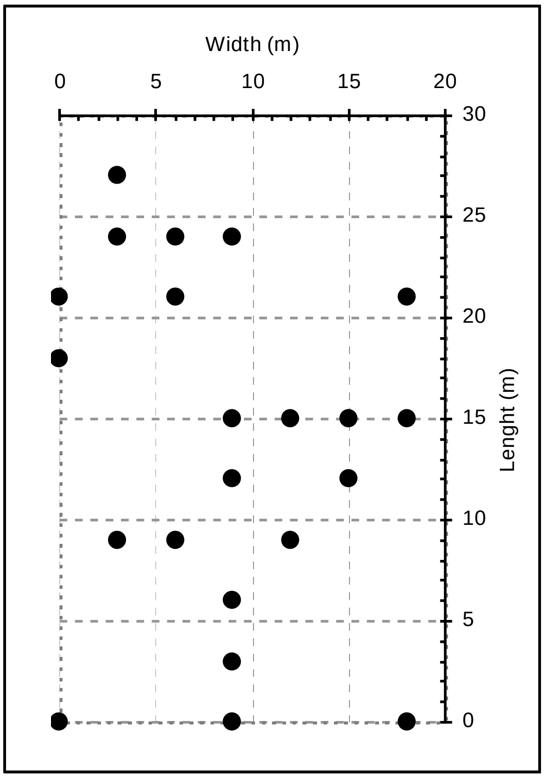

Figure 3: Spatial pattern of occupied baits in the study area

4 Discussion

The colonization of the baits seems to show a seasonal pattern, although this can only be confirmed with further monitoring. However, the decrease of activity during summer times was clear, as there were no new baits neither colonized nor abandoned. The termites appear to remain faithful to the same food source suggesting that during this period, where the average temperatures are rather high and with no rainfall, they decrease their search for new wood. Data from the same field site but using the line intersection method has shown an absence of termites foraging on above ground wood during summertime (Nobre *et al.*, 2003). This contrasts with the data recorded by Haverty *et al.* (1999), where *Reticulitermes* spp. in California were reported to be more active in summer months, underlying the notion of population behaviour diversity according to region conditions.

Nash and co-workers (1998) suggested that subterranean termites foraging in baits during late summer is likely to vary as a function of the quantity of natural wood available and the availability of moist soil for the construction of foraging galleries. The quantity of wood available on the surface was not lower than the observed in spring or autumn (Nobre *et al.* 2003) but, having in mind the climatic situation, the soil moisture was probably quite low which may led to a decrease in the construction of foraging galleries and thus the reduction observed on the occupancy of new baits.

The degree of attack of the baits also suffered an increase after summertime; either indicating a higher consumption of the baits (probably own to less mobility) if assuming the foraging population stationary, or an increase in the foraging population if level of consumption is kept constant. Further studies are need that would clarify the population dynamics of the colonies involved as well as on the relation between consumption and environmental conditions.

The data here shown suggest the existence of two foraging centres within the baiting grid established, as the occupancy of the baits was done in an aggregated pattern. Nevertheless, the conclusion that those foraging centres are independent, and their assignment to colonies, can only be made if in conjunction with other methods.

5 Acknowledgments

To the Portuguese Foundation of Science and Technology (FCT) for funding T. Nobre PhD research (ref. SFRH/BD/8761/2002), from which this study is part. To João Cappas e Sousa for his help and for making the field site available to us.

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Working Group 2 : *Impacts*

WORKING GROUP 2 : *IMPACTS*

WG 2 has been concerned with the analysis and reduction of environmental impacts of both existing and innovative wood protection. The main objectives were defined in the MOU as:

- risk assessment methods and rationale, including impact studies,
- implementing remediation technologies,
- recycling and disposal,
- scientific basis for quantifying biodegradability,

During the Action, the working group focussed on “New Principles for impact studies on risk assessment” all along the action, wood preservatives contaminated soils remediation (which leaded to the publication of the proceedings of a special seminar on this topic), Improved safety and effectiveness in the use of creosote and creosote based products which was discussed in a Joint WG1/WG2 workshop «creosote in the 2nd Millenium » in july 2002. The treated wood waste management topic was also discussed along the action and has been the main WG2 topic of the Final Conference, focusing on the situation in different countries and in Europe in general (regulations, characteristics of resources, management), the emerging techniques for handling wood wastes and treated wood after service and the research on this topic.

Dr Gérard Deroubaix
Chairman Working Group 2 ‘*Impacts*’

LEGISLATION AND RESEARCH CONCERNING WASTE WOOD IN DENMARK

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Keywords: waste wood, CCA, Danish legislation, research

Summery: *The present paper is the Danish part of a series of national papers about waste wood that were presented at the final workshop of COST E22, Environmental Optimisation of Wood Protection held in Lisbon March 2004. In Denmark wood waste containing creosote or As is classified as hazardous waste. Creosote was never used to a larger extent in Denmark and As has not been used in wood preservatives since 1993 where the use was forbidden. Still it has been estimated that about 2.7 mio. ton impregnated wood will end up as waste in Denmark of which most of it is within the hazardous class. Impregnated wood must be sorted out from other waste since most waste is incinerated in Denmark and incineration of CCA treated wood leads to As emission if special flue gas purification systems are not installed. The in practice impregnated wood from private households is sorted on basis of visual inspection and knowledge on use at public recycling centres. From demolition companies the wood must be sorted as well. Until methods ensuring utilization of the energy as well as metals in the wood are developed, impregnated waste wood must be deposited in landfills. The methods that have been investigated are the two thermal methods gassification and pyrolysis, and an electrochemical method for extraction of As before combustion are under development. Neither of the methods is implemented yet.*

1 Introduction

A heavy increase in the amount of chromated copper arsenate-treated (CCA) wood waste is expected in the next ten years in many countries. CCA has been widely used for wood preservation in the Northern European countries since the early 1950s. The fixation of CCA in the wood is quite good and eventhough some leaching occur during use, the concentrations of Cu, Cr and As is still high in the wood, when it ends up as waste, and due to this special handling of the wood waste is necessary.

The two most commonly used practices for handling of waste is disposal or combustion. Meanwhile both of these cause problems when dealing with CCA impregnated wood. Disposal of CCA impregnated wood in landfills can cause problems with elevated As concentrations in the percolate (Jambeck et al. 2004). If the landfill is unlined As is spread into the environment and if the landfill is lined the percolate needs special treatment for many years. Combustion of CCA treated is problematic, too, due to As emission to the air (Helsen and Van den Bulck, 2004). To avoid emission of As in this case it is necessary to install special flue gas cleaning systems and these are very expensive. Furthermore the result will be a very large amount of ash residues enriched with toxic As in a highly mobile form. There is simply a lack of methods that can ensure no emission of especially As to the environment.

The present paper is a part of a series of national contributions within the subject of impregnated waste wood presented at the final workshop of COST E22, Environmental Optimisation of Wood Protection in March 2004. This paper describes the Danish legislation in the field of wood impregnation and waste wood handling. The handling of waste wood in practice is outlined and so are different research projects that have been carried out in Denmark within the field.

2 Amount of impregnated waste wood in Denmark

Impregnated wood is in Denmark defined as wood treated with a wood preservative containing biologically active ingredients to protect the wood against degradation. This includes pressure- and vacuum impregnated wood as well as surface treated wood with wood preserving chemicals. Wood treated with ordinary paint or oil is not included.

CCA was a commonly used wood preservative in Denmark from the end of 1950s until 1993. In 1993 the use of As was forbidden. Later in 1996 a voluntary agreement between the environmental authorities and the wood preservers concerning Cr was made and after this year Cr has only been used in the preservatives for poles. It may be possible to buy imported wood containing Cr, but import of wood with As is not legal.

It has been estimated by Miljøstyrelsen (the Danish EPA) that a total of 2.7 mio. ton of impregnated wood will end up as waste wood in Denmark. Furthermore it is estimated that in 2010 about 100,000 ton impregnated waste wood must handled and this is a heavy increase compared to 17,000 ton in 1992 (Hansen et al. 1997). A newer estimate of the amount of impregnated waste wood in Denmark, based on production, import, export data, amounts the impregnated wood to 4 mio. ton, but this investigation does not take degradation and uncontrolled combustion of impregnated wood into account (Andersson and Quistgaard 2002).

The use of creosote treated wood has been limited in Denmark and the use has mainly been sleepers, poles and in relation to harbour constructions. A survey has been made to investigate the amount of creosote treated waste wood to be treated. The energy trading companies, Banedanmark (a railway enterprise, operating and developing the Danish state railway network) and Copenhagen Harbour was contacted (Andersson and Quistgaard, 2002). In the period from 2000 to 2010 a total of approximately 112,000 tonnes creosote impregnated wood are expected disposed of from these companies.

3 Legislation

The general departmental order about waste in Denmark includes criteria for classification of hazardous waste (based on type and concentration of compounds). Wood impregnated with As, creosote or PCP is classified as hazardous waste in Denmark. This decision will not be influenced by guidelines from EU (Andersson and Quistgaard, 2002).

From April 1st 2001 all local councils had to assign disposal possibilities for impregnated wood, as well as systems for separate collection (Miljø- og Energiministeriet, 2000). Until methods for handling of the CCA impregnated waste wood are developed the wood must be deposited in landfills and combustion is not allowed. Creosote impregnated wood can be combusted in special plants.

When new methods are developed that ensures utilization of the energy resource and the metals in the impregnated wood, all impregnated waste wood must be collected and treated. Until then the impregnated wood must be deposited in special landfills (Affald 21, 1999). Hazardous waste must not be deposited in landfills with non-hazardous waste in accordance to Departmental order of disposal, but the impregnated wood can be deposited together with other waste that is not suitable for combustion

4 Sorting

Sorting of impregnated waste wood from other waste was decided by law and came into force in April 2001. In practice the sorting procedure from private households is carried out at local, public recycling centres. At some recycling centres the impregnated wood is collected in containers together with other materials that will not be incinerated e.g. PVC and asphalt. At a few recycling centres a special container for impregnated wood is present, but this is not required in Departmental Order of Waste.

As aid for sorting out impregnated wood Miljøstyrelsen has produced a handbook that provides an overview of various types of impregnated and non-impregnated wood. The book, which is intended for the staff at recycling centres, describes in words and pictures the visual characteristics and typical use of impregnated and non-impregnated wood. The book also explains that impregnated wood should be collected and managed separately at the recycling centres.

Danish industries producing hazardous waste during production must ensure that the hazardous waste is not mixed with other sorts of hazardous waste or non-hazardous waste according to the Departmental Order of Waste. Hazardous impregnated waste wood is mainly produced from demolition companies, since newly impregnated wood do not contain wood preservatives that classifies the wood as hazardous. In Denmark demolition companies do not have to map building materials prior to demolition as the case is in Norway and Sweden, and the sorting seems to be carried out insufficiently compared to the required (Andersson and Quistgaard, 2002).

At present sorting is done on basis of visual inspection and knowledge on use of the specific wood. Meanwhile, a sensor for detection of heavy metals in wood has been developed by DK-teknik and Force in Denmark. The sensor can distinguish between different inorganic wood preservatives and can thus be used for separation of salt impregnated wood into a hazardous and a non-hazardous fraction. The sensor is stationary and developed for use at recycling centres. Whether the sensor will be used for sorting of impregnated waste wood is uncertain at present since the methods that are under development for handling the wood waste can handle a mix of impregnated and un-impregnated wood. The sensor can be used for sorting PVC out from other plastic as well, and here the use is considered to be more promising than for sorting wood by the inventors of the sensor.

5 Landfilling

From the recycling centres the impregnated wood is transported to landfills e.g. AV Miljø in the Copenhagen area. Here impregnated wood is deposit and there is no separation of hazardous and non-hazardous impregnated waste wood. The creosote-treated wood that could be combusted is not sorted out either. If the wood arrives mixed with other non-combustable waste types the waste is deposited together, but in the case of well sorted impregnated wood this fraction is deposited so the wood can be taken out from the deposit and treated when methods are developed for this (Andersson and Quistgaard).
Miljøstyrelsen recommend that impregnated wood is disposed off in landfill without any cutting or chipping into smaller pieces since this may cause a risk to the workers and furthermore leaching is expected to increase due to a larger surface area.

6 Direct reuse

With very few exceptions it is not allowed to reuse CCA impregnated wood as such. The CCA impregnated wood must be handled as waste when first removed from service at the original place. The exceptions are poles that can be reused as poles in other places and in marine environments but in both situations a dispensation must be applied for (Miljøstyrelsen homepage)

7 Treatment methods under development

Miljøstyrelsen prescribes that methods, that ensures the utilization of heavy metals and energy from impregnated wood must be developed. The absolute possible maximum of impregnated waste wood is 4 mio. ton. This amount of wood represents an energy resource of about 4.4×10^7 GJ corresponding to the heat used in about 571.500 Danish households (Andersson and Quistgaard, 2002). Meanwhile the amount of waste wood that can be collected in practice is substantially less, but still the wood waste is an energy resource beneficial to use.

Different thermal method has been tested in Denmark and a method for extraction of the inorganic wood preservatives from the wood before combustion is under development.

7.1 Gassification and pyrolysis

At Kommunekemi (an independent limited company, owned by the Danish municipalities) two different thermal methods has been tested for treatment of waste wood: gassification and pyrolysis. Both of these methods are made to turn organic waste into a fuel to be burnt and are known from treatment of other waste products than wood. In both cases the organic waste is heated in a large rotating drum. For pyrolysis no air is allowed in and this produces a solid fuel, which can be turned into a liquid. For gassification air is allowed in and some of the waste is burnt, some is turned into a gas, which can be used. At Kommunekemi both methods has been tested in pilot scale, but most of the results are confidential.

7.2 Teatment of residues from thermal treatment

A general issue of concern in relation to the thermal treatment is As emission to the air. Meanwhile there is still a matter to cope with when methods to avoid As emission are implemented; the residues with increased concentrations of Cu, Cr and As. In (Ottosen et al., 2004) two different residues after thermal treatment are characterized: a mixed bottom and fly ash from combustion of CCA impregnated wood and a charcoal from pyrolysis of treated waste wood.

By SEM/EDX it was seen that the charcoal still showed wood structure with both tracheids and rays and that Cu, Cr and As were to be found inside this wood structure. Cu was found alone while Cr and As were often found together. By chemical analysis it was found, too, that the charcoal contained high concentration of Zn, probably from paint. Chemical extraction experiments were conducted and showed that Cu, Cr and As were all less extractable than in waste wood before pyrolysis.

A SEM/EDX investigation of the mixed ash from combustion showed presence of small particles with wood structure with elevated Cu and Cr concentrations, too, but most particles were irregular shaped matrix particles rich in Si, Al and K. Here Cr was abundant in many different particles. As was often found associated with Ca in char-like (porous) particles, indicating that Ca-arsenates had been formed during combustion. Cu was often associated with Cr in the unburned wood pieces, whereas it was less abundant inside the silica-based matrix particles. Cu was also found in almost pure form in a small layer on the surface of some matrix particles indicating condensation of volatile Cu species. Chemical extraction with inorganic acids showed about 90-100% As could be extracted at pH 2.

The two residues from the two different thermal processes were very different and a method for handling residues must be developed after the thermal treatment method.

7.3 Electrodialytic treatment of wood chips

In collaboration between the Technical University of Denmark and New University of Lisbon a method for extraction of CCA from wood has been developed. The method is based on an electrochemical principle and is described in e.g. (Ribeiro et al 2002) and (Kristensen et al. 2001) where results obtained in laboratory scale are given, too. The results obtained in laboratory scale were very promising with high removal efficiency for As (85-100%). A pilot plant for electrodialytic treatment of about 2 m^3 CCA-treated waste wood was designed and tested. The project was supported by EU´s LIFE Programme. The best result obtained in pilot scale so far was removal of 87% Cu, 81% Cr and >95% As (Christensen et al. 2004). This result is very encouraging since As emission is the main concern during thermal treatment. If As is removed prior to utilization of As from the wood this treated wood can be combusted in common waste incinerators without implementation of specialized flue gas systems.

8 Initiatives to minimize the amount of problematic waste wood in the future

The main step in hindering a continuous production of hazardous waste wood was taken in 1993 where it was decided by law that wood impregnated with As could not be used in Denmark.

It is further sought to minimize the use of impregnated wood (with the As free preservatives) to places where it is necessarily to use impregnated wood (mainly ground contact) and to use alternative wood sorts (hard wood) for other outdoor uses. Miljøstyrelsen and Træbranchens Oplysningsråd have produced a set of flyers that can help consumers in finding durable and environmentally good solutions to their work with wood. These flyers are distributed from e.g. timber merchants. At the home page of Miljøstyrelsen good advises are published, too.

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IMPREGNATED WOOD WASTE MANAGEMENT - PROCESSES UNDER DEVELOPMENT IN FRANCE

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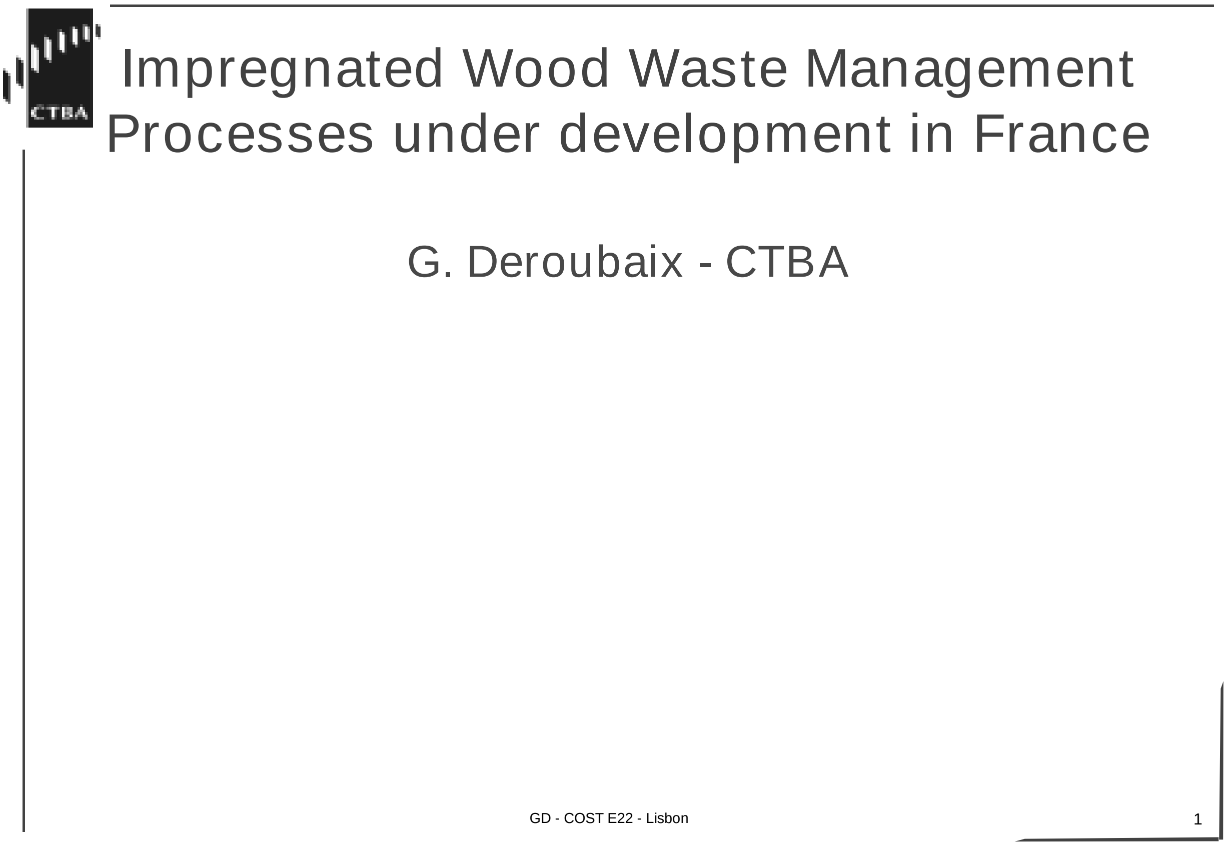

Impregnated Wood Waste Management
Processes under development in France

G. Deroubaix - CTBA

GD - COST E22 - Lisbon

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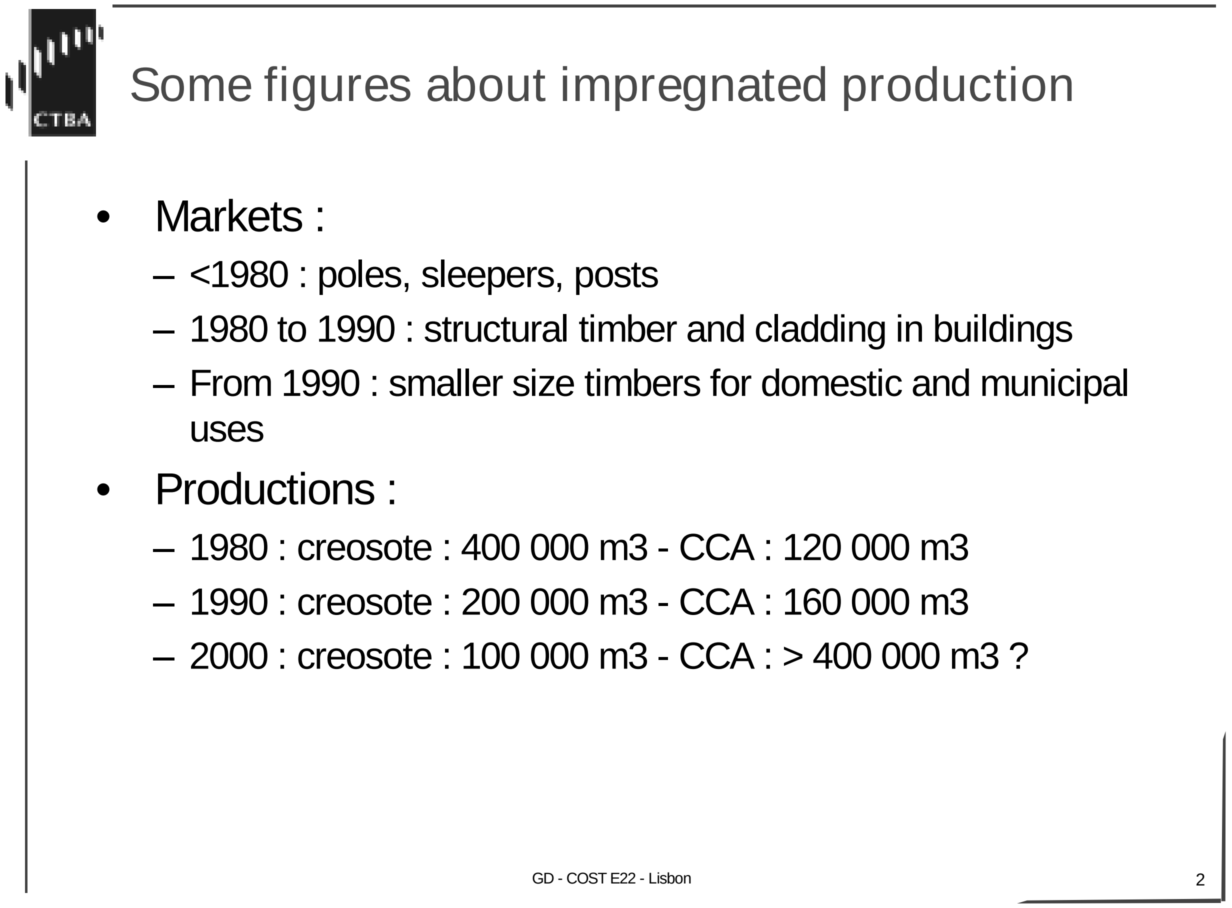
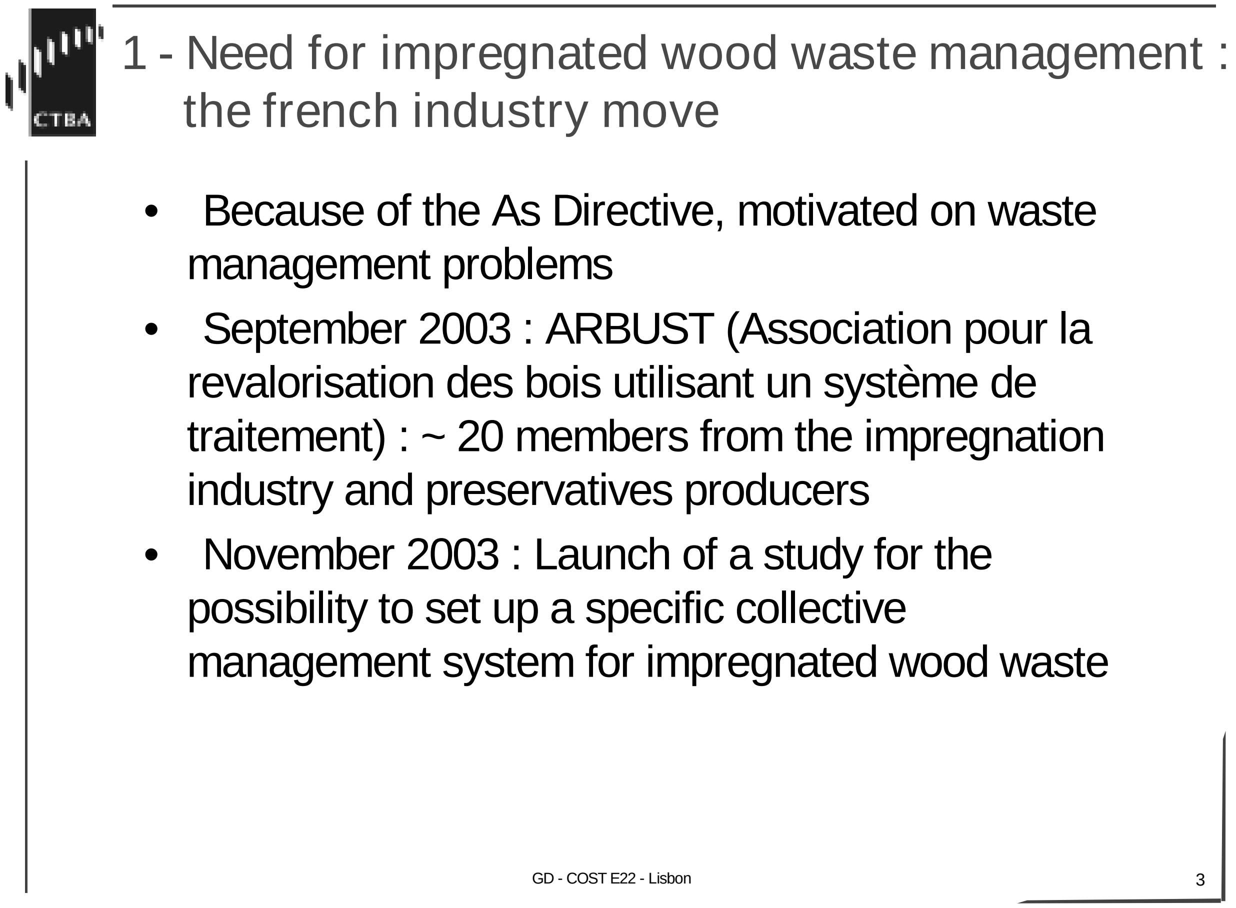


Some figures about impregnated production

- Markets :
  - <1980 : poles, sleepers, posts
  - 1980 to 1990 : structural timber and cladding in buildings
  - From 1990 : smaller size timbers for domestic and municipal uses
- Productions :
  - 1980 : creosote : 400 000 m3 - CCA : 120 000 m3
  - 1990 : creosote : 200 000 m3 - CCA : 160 000 m3
  - 2000 : creosote : 100 000 m3 - CCA : > 400 000 m3 ?



1 - Need for impregnated wood waste management : the french industry move

- Because of the As Directive, motivated on waste management problems
- September 2003 : ARBUST (Association pour la revalorisation des bois utilisant un système de traitement) : ~ 20 members from the impregnation industry and preservatives producers
- November 2003 : Launch of a study for the possibility to set up a specific collective management system for impregnated wood waste

2 - Recycling : concrete-wood composites

- Studies have been carried out for the preparation of a filling material for road works
- According to ANCOR SA leaching experiments have been carried out and results found acceptable for this use

3 - Thermal treatments :

3.1 SIDENERGIE : Charcoal production from creosote treated wood

- 500 °C thermolysis with combustion of gazes
- Charcoal evaluated by toxicologists and approved for “barbecue uses”
- Investing now for a 22000 t/y treatment capacity and to produce active carbon

3.2 THERMYA : Chartherm process

- Low temperature pyrolisis under inert gas
- Energy self sufficient system
- Mechanical separation of metals from carbon
- 1t of wood -> 280 kg of carbon; 50 kg metals
- Industrial markets for graphitic carbon
- Pilot plant developed (10 000t/y)
- Project to develop and operate a full scale plant in 2005 (30 000 t/y)

3.3 ANCOR : PGI process

- High temperature pyrolysis (1200 ℃)
- Energy production from gases combustion : cogeneration
- Pilot equipment under test (300 kg/h), with other carbon containing products (tires)
- Mineral fraction (metals, glass) can be recycled

WASTE MANAGEMENT IN NORWAY

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Keywords: Waste management, CCA, creosote, Norway

Abstract: *Since 1 January 2003, CCA and creosote wood waste have been classified as hazardous waste in Norway. Since this classifiacation came unprepared on the companies handling waste, we have had a problematic situation in Norway during 2003 and still have. The waste companies charge different feesfor the treated wood waste since they have to export it to Sweden for incineration. During 2003 and the first part of 2004 the Norwegian environmetal authorities have had a dialog with the waste companies, the preservation plants associations, the constructers associations, electricity boards etc. to sign a voluntary waste management agreement for treated wood waste. They wanted to establish a company to deal with this waste and to finance it with a fee on new treated wood.*

1 Introduction

Until 1 January 2003 all treated wood waste (mostly CCA and creosote) could be reused or delivered to the waste disposal sites as mixed waste for companies, and free of charge in most communities from private persons.

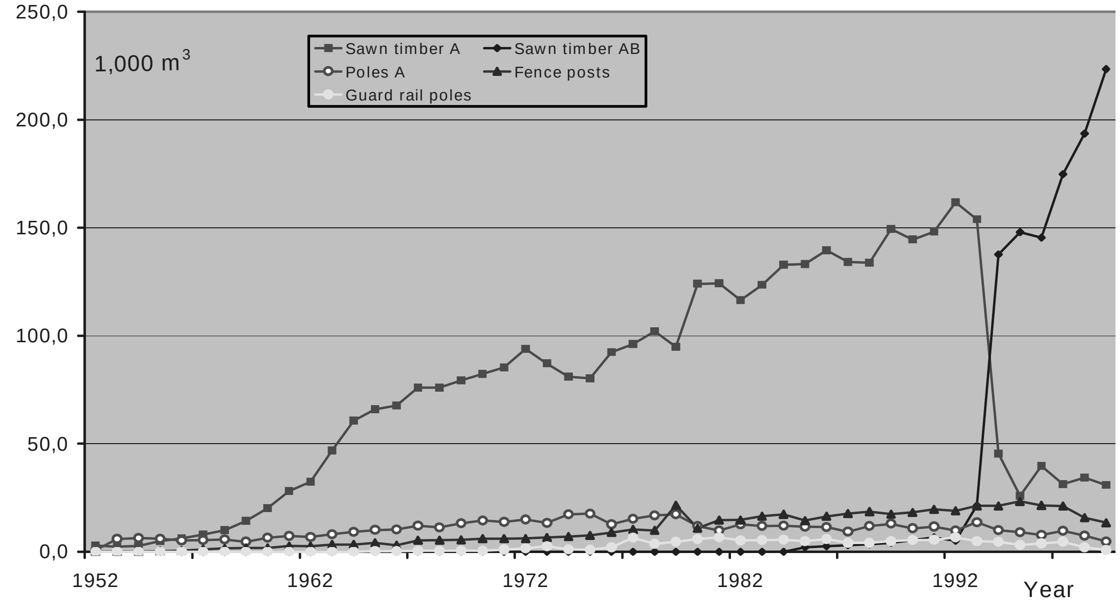

Figure 1: The production of treated wood with water-borne preservatives in Norway 1952 - 2000

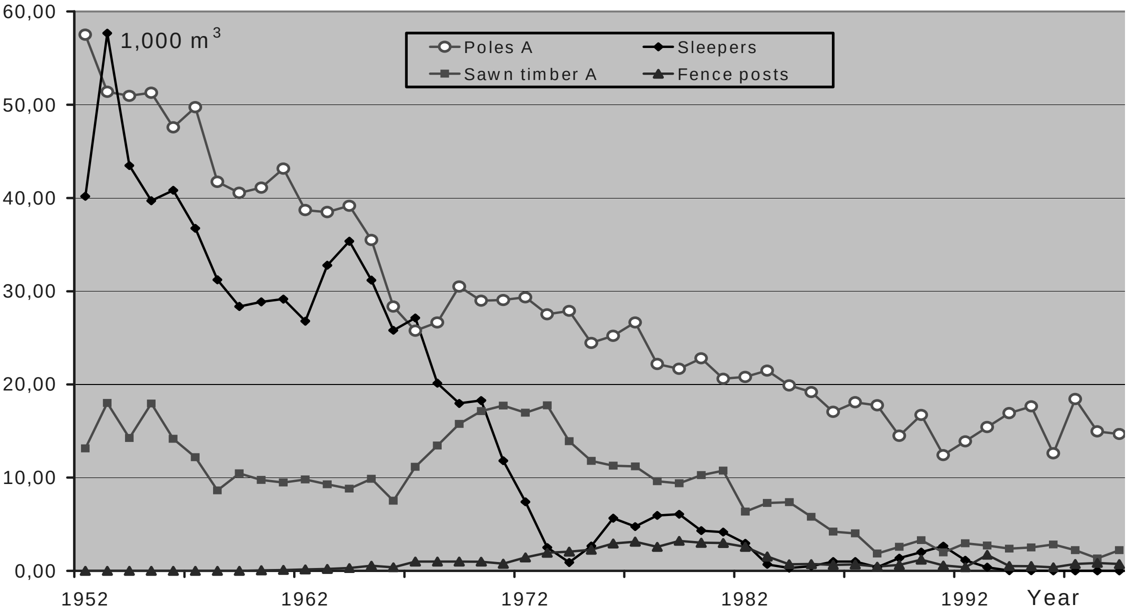

Figure 2: The production of treated wood with creosote in Norway 1952 - 2000

Based on the production of treated wood with water-borne and creosote, we can estimate the amount of wood waste based on the expected service life. These figures are, however, very uncertain, since some treated timber, e.g. decking can be changed because of appearance and not decay. All the timber treated before 1993 is produced in the Nordic class A (for ground contact) but most of the sawn timber are used above ground. This will lead to long service lives. In 2002 CCA was not allowed to reuse and in 2003 reuse of creosote treated wood was banned. This means that from 2003 all treated wood with CCA or creosote is waste when taken out of service. However, we have tried to estimate the waste of treated wood with CCA and creosote in the figures 3 and 4 in the years 2001 – 2050.

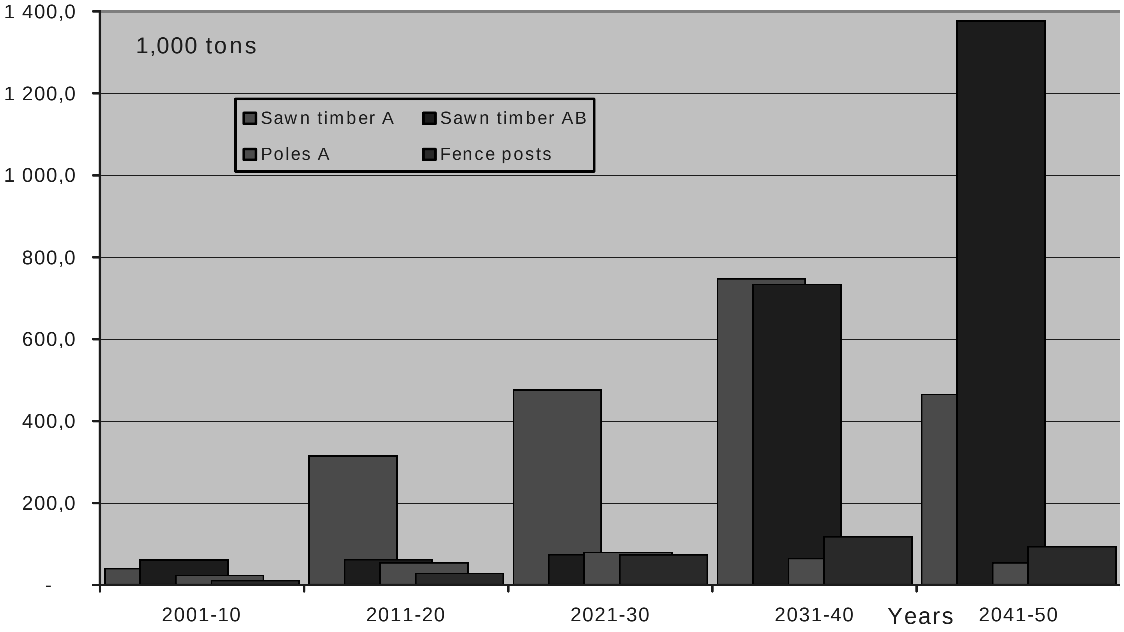

Figure 3: Estimated waste of wood treated with water-borne preservative based on expected service life

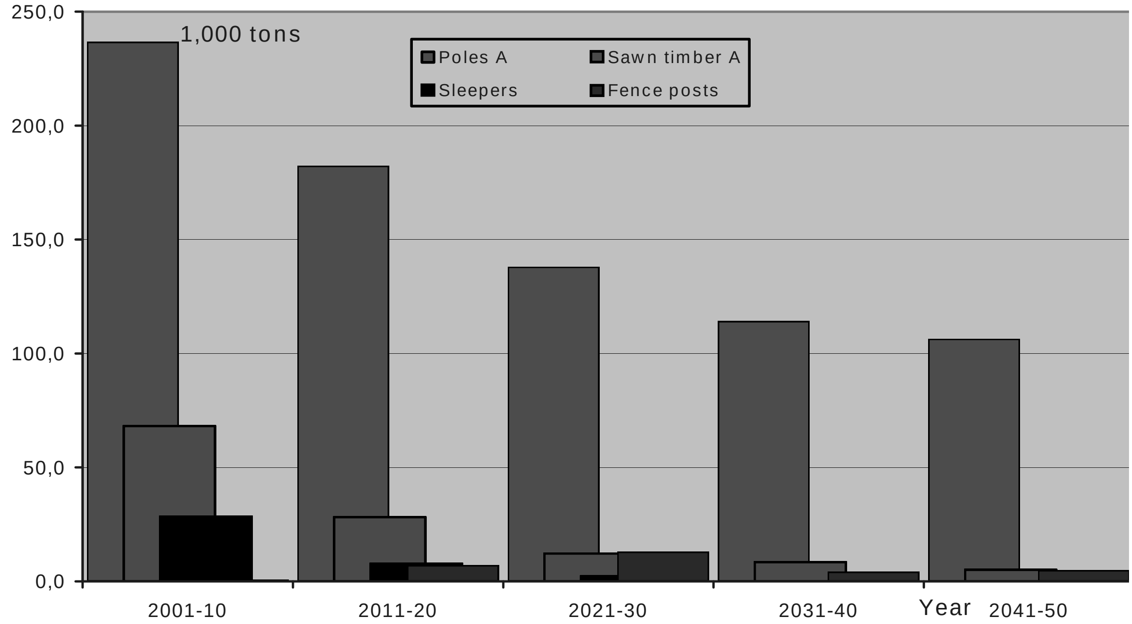

Figure 4: Estimated waste of creosoted wood based on expected service life

2 New regulation for treated wood

From 1 January, the Norwegian authorities classified CCA and creosote wood waste as hazardous waste. The waste disposal sites were unprepared for this and most of them got a dispensation to handle this kind of hazardous wood waste. The dispensation for hazardous wood has limitation in weight, so they had to send the hazardous waste to approved sites after a short while.

To cover their expenses the different sites decide their own fees and some charged as much as NOK 2,000 (app. € 250) a ton for the CCA-treated wood waste. Even the timber treated with the new chromium and arsenic free preservatives (which is no not classified as hazardous waste and produced in Norway from early 2002), i.e. all “green” wood waste has to pay the fee, since the site could not tell the different preservative apart except using expensive chemical analysis.

The sites stated the reason for the high fees was that they had to export all the CCA and creosote treated wood waste to Sweden, since we have no approved combustion plants for burning this waste in Norway.

After the fees have been discussed in the local newspapers, some sites reduced or removed their fee. However, the recommendation from the waste sites association is to charge a fee for treated wood waste from private persons even if all other type waste they deliver is free of charge.

3 Establish a new company for treated wood?

After the authorities had passed the new restriction 1 January 2003, they started to discuss the waste problems with the producers and professional users of treated wood. The authorities wanted to establish a new company that could handle the treated wood waste the same way they had solved the paper, plastic and other waste problems in Norway. Since the collection and sorting, as well as shipping and burning of this wood waste was expensive, since it had to be send to Sweden, they wanted to put a fee on the new produced treated wood, even if it has no restrictions (so far) as wood waste. The authorities hoped to have a written agreement with the producers and professional users before the end of April.

However, during 2003 it had been promising tests with incineration of 10 % mixture of treated CCA-wood in a common waste combustion site. The authorities have therefore let the discussion about a new company for treated wood waste rest until the incineration test results have been evaluated. All the involved parties hope that, we will be able to burn it together with domestic waste instead of sort out the CCA and creosote treated waste and treat it separately. This can also solve the difficulty to sort out the CCA from the new copper containing preservatives, since all the waste wood then can be burned in the same way.

4 The current situation

So far, the uncertain situation continues. The waste sites still charges different fees for treated wood waste and cannot differ between CCA and other copper treated wood. They have only dispensation to handle a small amount of hazardous waste and must forward it to special plants for hazardous waste or to Sweden for combustion.

WOOD WASTE MANAGEMENT – UK UPDATE

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Keywords: Waste, recycling, reuse, research, wood

Summary: *This paper presents an overview of wood waste management in the United Kingdom and provides an analysis of the volumes of waste wood generated and reused. Issues influencing the reuse of waste wood in the UK are discussed and a summary of research activity in the UK is presented. The paper concludes that waste minimisation should be the initial focus and that considerable opportunities exist for the 5 million tonnes of wood waste annually sent for landfill or incineration. Particular opportunities exist for increased production of panel products incorporating recyclate and through the development of an energy recovery strategy.*

1 Introduction

COST Action E22 aims to increase the cost-effective use of European wood by means of optimizing the environmental factors. Specifically the practices of recycling and wood waste management need to be evaluated. In the United Kingdom approximately 7.4 million tonnes of post consumer wood waste are generated per year. Added to this figure are timber-based wastes from furniture manufacture, fencing, infrastructure (such as telegraph poles and railway sleepers), agriculture and horticulture, and the do-it-yourself (DIY) sectors. Whilst maximizing materials recovery is important there are concerns regarding contaminated timber, increasing environmental impacts through recovery routes as well as resolving the question of how best to manage specific waste wood sources.

Presently only a small fraction of this resource is recycled in the UK. European and national legislation along with fiscal instruments, such as the landfill tax, influence wood waste management. The trend towards timber recycling has been boosted by landfill taxes and is continuing to increase with the main constraint being transportation to markets (typically chipboard manufacture). Wood-based panel product manufacturers also have serious concerns with respect to quality and consistency of supply, which diminishes their ability to make full use of recycled wood fibre.

There are currently several hundred wood recycling facilities in the UK. The Wood Recycling Association has been established to provide a lobbying force for the wood recyclers and to provide focus for determining new markets and quality standards for recyclate but they need support from the research and technology communities.

This paper presents an overview of wood waste management and research activities in the UK. The limitations of this paper allow only a brief overview and it is recommended that data be cited from their original sources.

2 Wood waste in the United Kingdom

The UK does not lead Europe in the recycling and reuse of materials. An example is the percentage of waste paper that is reused in some member states (Figure 1). However, with one of the largest construction industries and wood markets in Europe the potential impacts for recycling or reuse are considerable.

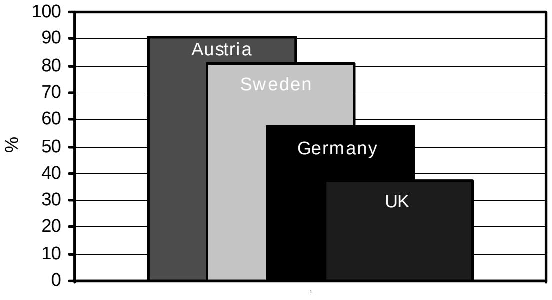

Figure 1. Paper recycling rates in four member states for 1999

The United Kingdom consumes 48 million tonnes of wood each year and generates 7.4m tonnes of post consumer wood waste. Of this 0.7 million tonnes of wood waste are reclaimed each year (usually from demolition sites e.g. floorboards and beams have an inherent value and can be sold on for reuse) and 0.9 million tonnes of wood waste are recycled into panel products (70% of this comes from the pallets and packaging industry). The majority of wood waste goes into landfill or incineration at the bottom of the wood waste hierarchy. The wood waste hierarchy (Figure 2) highlights the desirability of reducing the volume of wood in waste streams.

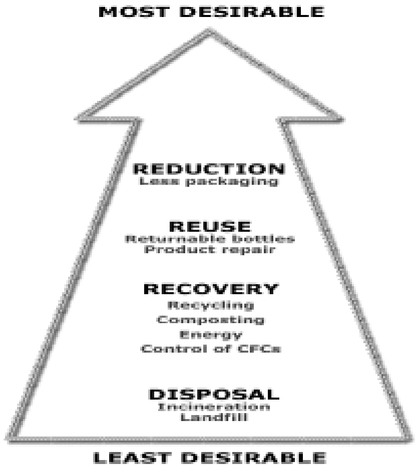

Figure 2. The wood waste hierarchy

Primary processing of logs at UK sawmills is not considered within this evaluation of wood waste. For the 365 UK sawmills the average yield of a log is 56% although this figure varies considerably. The remaining 44% is not classified as waste but as co-products. Approximately 1.7 million green tonnes per year of co-products are generated of which 83% go to the wood processing industries (panel products), 11% bark (mulches and surface dressings) and 2% is burnt for local heating.

The sawn timber trade also generates wood waste though figures are difficult to collate. An exemplar of national operations is shown by the UK's largest do-it-yourself (DIY) retail chain. The company, B&Q, have over 300 stores nationwide and operates a 'backhaul' scheme for waste wood generated from damaged timber goods and from their processing and sawing activities. The vehicles delivering new products to the stores collect the waste wood and deliver it back to a central processing station. In 1999 over 2500 tonnes of woodchips from this source were sold to panel product manufacturers. This practice is expected to increase amongst other sawn timber trade outlets and retailers of wood products.

In any review of data from various sources and approaches there has to be a degree of caution applied to the figures as there have been few detailed studies on which to base decisions. The reliability of the data needs to be continuously questioned. The BRE SMARTWaste™ data (see later in this paper) are perhaps the most detailed series of studies of wood waste in the construction and demolition industry. The data are gathered from various sources but the overall breakdown of construction wood waste is estimated to be in the region of:

- Construction 1.2 million tonnes
- Demolition 2.1 million tonnes
- Packaging from construction 1.3 million tonnes
- Commercial 750,000 tonnes (mostly packaging)
- Industrial 840,000 tonnes (mostly packaging and furniture)
- Civic amenity sites 672,000 tonnes (mostly furniture)
- Municipal Solid Waste 140,000 tonnes (mostly furniture)
- Furniture manufacturing 335,000 tonnes
- Fencing 60,000 tonnes

The total construction wood waste believed to be available in UK for reuse, recycling or recovery of energy is approximately 7.4 million tonnes per year. Demolition activities produce 2.1 million tonnes per year of waste wood, of which 50% is reclaimed and 50% is disposed. It is noted that recycling levels are very low and that reclamation is highly labour intensive (floorboards, beams). Pallets and wood packaging provide 1.3 million tonnes per year of wood waste (equivalent to 13% of the total UK packaging waste stream) and a considerable proportion of this is recycled into panel product manufacture. The Timber Packaging and Pallet Confederation estimate that half needs to be recovered each year to meet the EU Packaging Directive regulations. For furniture there are reuse networks, however much of this and of general household wood waste ends up in civic amenity sites or landfill.

Figure 3 shows that packaging (25.9%) represents the greatest amount of waste from construction sites followed by timber (13.8%), plaster and cement (11.5%) and miscellaneous (9.6%). When timber packaging is included with the timber figure, this increases to 26%. Therefore we can presume that more than a quarter of the waste from construction sites is timber (2.5 million tonnes using current estimates). However, this information is based on a limited number of case studies of different size and type but the data are believed to be the most accurate and reliable available for construction sites.

It is also important to note the variability of the composition of construction waste between sites and work packages. The percentage variability of wood waste between 11 construction sites has shown that although the average figure is 13.8%, this can range between 3% and 23%. Information is even more scarce for the demolition industry. Recent figures produced by BRE (Figure 4) show that timber is estimated to be only 3.4% of demolition waste, although this would be increased if timber furniture was included. A conservative figure of 7% wood waste from demolition is perhaps more realistic.

The secondary processing industries which manufacture in wood contribute a considerable volume to wood waste streams. For example, in the manufacture of furniture, 40% of hardwood, 30% of softwoods and 15% of panel products ends up as waste. Similarly for the manufacture of joinery products, 35-60% of hardwood and 25% of softwood are wasted. Construction sites and building projects are focussing on waste minimisation and separation of materials.

Similarly providing estimates or figures for the volumes of wood waste containing treated timber is difficult and must be viewed and interpreted with caution. It is estimated that 80,000 tonnes of preservative treated wood waste (of which as much as 50% may be CCA treated) are committed to landfill every year in the UK.

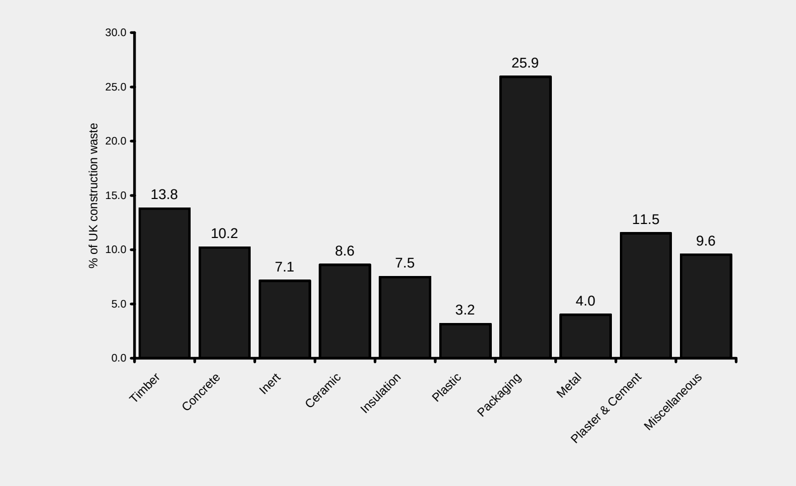

Figure 3. Average Composition (by volume) of UK Construction Waste by Group

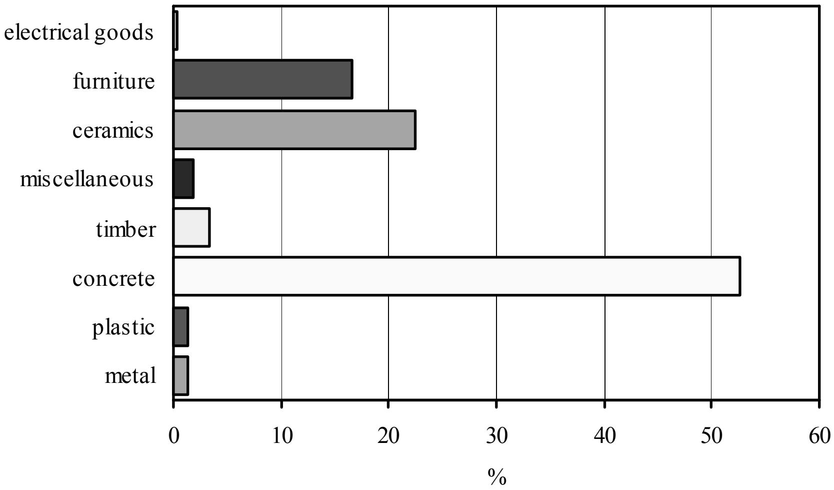

Figure 4. Average composition (by volume) of UK demolition waste

3 Reuse and recycling

Previous work carried out on minimisation and recycling of construction and demolition waste has shown that:

- The greatest recycling opportunities derive from demolition,
- Recycling of inert material from construction and demolition waste is widely practised in the UK,
- Active components, such as wood and plastic, are often regarded as contaminants in the recycling of aggregates,
- The main exceptions to this are large quantities of untreated timber from demolition and reusable timber suitable for the reclamation industry,
- Wood waste most likely to be disposed of without any form of recovery includes treated and painted timber, wood-based panel products and small quantities of any waste including untreated and reusable timber,
- Low value waste will be disposed of at the nearest appropriate facility.

The options for waste timber from demolition and construction activities are reclamation for building reuse, provision of wood chip for panel products, provision of a feedstock for composting, animal bedding, surface treatments and mulches. Energy recovery is going to become increasingly more important. Some 50,000 tonnes of charcoal are produced annually in the UK and more of this might be produced from waste hardwoods from the furniture and joinery industries. A helpful tool for minimising and identifying the best option for waste is the Best Practicable Environmental Option (BPEO) defined as:

"…the outcome of a systematic and consultative decision-making procedure which emphasizes the protection and conservation of the environment across land, air and water. The BPEO procedure establishes, for a given set of objectives, the option that provides the most benefits or the least damage to the environment as a whole, at acceptable cost, in the long term as well as the short term." (RCEP, 1988)
The England and Wales Waste Strategy (DETR, 2000) suggests that it is unlikely that one approach will represent the BPEO for all elements of the waste stream (including the wood waste stream). Wood waste can be a complex mixture of different timber types and applications, in differing proportions originating in various locations and varying levels of contamination. The theory of BPEO means that local environmental, social and economic criteria will be important in any decision. For some wood waste streams options nearer the bottom of the waste hierarchy may be the most practical BPEO (e.g. CHP plants, wood burners, energy recovery). Obviously, locality plays an important role in deciding the BPEO – what may be the BPEO for one area generating waste may be entirely different for another area. This is dependent on the proximity of waste management facilities and transportation options. The criteria that are an essential part in the BPEO assessment include:

- Technical: new technologies, current and future management routes, innovation, specifications
- Environmental: transportation, impacts on water, air, lands, global and local
- Social: health, community, employment, transport
- Legislative: landfill tax, landfill directive, packaging regulations, European waste catalogue, etc
- Economic: markets, supply, financial cost, environmental cost, viability, proximity

A key issue is how these criteria interact with each other and if necessary how the criteria will be weighted.

The barriers to recycling and reuse are:

- low cost of landfill
- irregular feedstock supplies
- contamination issues
- limited (or distant) collection and processing networks
- lack of reprocessing technologies
- low demand for recycled wood products
- lack of target for wood in EU Packaging Directive
- practicalities of recycling

The provision of a mechanism and an incentive can create the right conditions to stimulate the provision of a feedstock. Legislation is often a key driver in either providing the incentive (or penalty for generating waste) or encouraging the development of a mechanism to identify the best options.

The drivers for recycling and reuse in the UK are:

- The Landfill Tax (1996): £15 (€23) per tonne for biodegradable waste
- European Landfill Directive (1999): reduce biodegradable waste to 35% of 1995 levels by 2016
- European Packaging Directive (1997): 50% of UK packaging must be recycled or recovered by 2001. However, no specific target for wood.
- Climate change agreements (Kyoto 1997, UNFCCC 1994): limit methane production from landfill
- Sustainable development initiatives
- Increasing demand for wood based panel products
- R&D funding
- Bio-fuel power stations
- Taxes and tools to facilitate recycling

There has been considerable effort at Local Government level in the UK to promote a recycling philosophy. Under UK government targets, 45% of household rubbish must be recycled by 2015. In order to achieve this, new approaches for legislation are being considered which will cascade down to Local

Government. These include tax increases on 'disposable' products, value added tax (VAT) reductions on products made from recycled materials (VAT is currently 17.5%), a charge for households per bag of rubbish generated and local tax refunds for households that recycle.

4 Facilitation of wood waste minimisation and recycling

The separation of wood waste needs to deliver a financial benefit to those generating it, be that a construction company or manufacturer. Some skip and waste management companies offer reduced rates for the removal of segregated wastes, but there is still a need to provide a cost benefit in order to pay for the increased manpower required to establish and maintain these activities. There is a gap here in present UK operations. Pressures on rural manufacturers are exacerbated by their geographical position which may be distant from waste management centres. Extra costs of transportation or collection and the inherent inefficiencies in this process, impact upon waste management strategy selection. It seems sensible therefore to concentrate initially on reducing volumes of wood in the waste streams, encouraging a more environmentally aware waste philosophy and promoting the practical options for wood waste recycling.

SMARTWaste^{TM} (www.smartwaste.co.uk) is a waste auditing tool that has been in operation in the UK for several years on construction, demolition, refurbishment and manufacturing sites. The waste is separated out into 12 groups, one of which is timber. These material groups help identify the reuse and recycling potential of individual products within those groups. SMARTWaste™ is presented in several components. SMARTStart™ records the waste arisings, composition, levels of segregation and recycling. It also provides information on the selection of environmental performance indicators (EPI) and key performance indicators (KPI). SMARTAudit™ benchmarks the sources of waste, type, amount and costs for specific projects (construction, demolition, refurbishment) and identifies actions for reducing waste and maximising recovery. BREMAP™ helps define the BPEO using a GIS (Geographic Information System) to locate facilities for the project (e.g. composting yards, reclamation sites, landfills, transfer stations, incinerators). Finally a package has been developed for Local Government called SMARTStartLG™ to help Local Authorities with their construction projects. Local Government procures 60% of UK construction so this is an important step forward.

There are numerous tools available to the consumer of wood products that are designed to promote recycling and to provide routes for appropriate end of use disposal. Many of these options are web-based and are briefly summarized here.

www.recyclewood.org is an interactive mapping tool designed for use by members of the public and businesses in order to help them find their local wood waste management facilities. The www.wasteaware.org.uk site provides a similar resource for a wider range of materials.

recycle-more.co.uk was launched by Valpak to help its 3000 members of the packaging industry with the implementation of the European Union Packaging Directive. It is also of value to other businesses, schools, households and local authorities who want advice on the recycling options that are available and the location of waste management facilities.

www.rethinkrubbish.com provides ideas for reducing waste for the home owner and business. It is sponsored by a collaboration of retailers and DIY stores. It also provides links to Local Authorities which provide helpful advice and information on a regional scale.

salvomie.com is a materials exchange site that provides a marketplace for construction and landscaping professionals to offer their waste materials for reuse and recycling.

www.recycle-it.org is a site maintained by the Timber Recycling Information Centre. It helps users and specifiers of construction timber products to implement sustainable waste management policies. Its primary objective is to help reduce the volumes of timber in the UK waste stream.

5 Research in the United Kingdom

The Waste & Resources Action Programme (WRAP www.wrap.org.uk) was established to manage a research fund to aid the implementation of the UK Government's Waste Strategy (2000). It has a £40 million (approximately €60 million) three year programme which aims to promote sustainable waste management and to create stable and efficient markets for recycled materials and products. Wood is one of the seven priorities of the programme, which aims to deliver a 1 million tonne increase in the amount of wood recycled in UK by the end of the programme.

WRAP identified some key areas in wood waste management that need to be addressed. They are:

- recycling of wood poles and sleepers
- wood reclamation and recycling from construction and demolition wastes
- wood collection logistics - access to distant and uneconomic sources
- new wood recycling applications
- increase wood packaging recovery
- achieve 40% recycling and re-use of construction and demolition waste

The programme has highlighted the impact of standards on recycling of wood as two fold. Firstly, there are no specific standards and secondly some existing standards and specifications are barriers to UK wood flows and markets. As a result they have developed feedstock specifications for recycling applications such as chipboard manufacture, animal bedding and surfacing products. WRAP commissioned the British Standards Institute to develop a Publicly Available Specification (PAS) for recycled wood chips as a feedstock for panel products. This should be available in mid-2004 and will assist in promoting this recycling option. They have also secured European Union State Aid grants of £0.5 million to two wood waste companies which will provide an additional 100,000 tonnes of recycled wood between 2002 and 2005.

Other important sources of research support for wood waste issues are the Landfill tax credits scheme (managed by the contactors e.g. Biffa, Onyx, Cleanaway), the UK Government's Department of Trade and Industry (DTI) and the Foundation for the Built Environment (FBE). Table 1 presents some of the wood waste research projects. More information can be found on websites listed in the acknowledgements.

Table 1. Some research activities on wood wastes in the UK

Organisation	Research project	Notes
ABFM*	Promoting recycling in the furniture industry	assessing volume & nature of wastes, options for recycling. Develop best practice guidance
Lothian Tree Services	Novel product from sawdust waste	digestion of sawdust to produce a plant health product. Growing trials are under way
Buckinghamshire Chilterns University College	Enabling manufacturers of panel products to produce boards with high recyclate content, whilst ensuring consumer safety	developing the protocols for measuring contaminants in recovered wood by reviewing methods for detecting contaminants
Buckinghamshire Chilterns University College	Bioremediation of treated wood waste	The specific wood decay fungi have been characterised and tolerances to wood preservatives determined. Optimised for CCA/CCB and Tanalith E and is now ready to be optimised for a 25 litre 'bioreactor'
BioComposites Centre	On-line monitoring	Feasibility of using IR analysis as an online monitoring tool to quantify varnish content of waste wood prior to particle board manufacture
BioComposites Centre	Recycling of Wood Panels in the Furniture Industry	see TRADA project on MDF and particleboard
BioComposites Centre	Recycled waste paper residues in construction products	hybrid waste wood/waste paper panel production
TRADA	Footprint for wood waste for the M62 corridor in England	type, volume and source of waste wood and the potential for recycling, barriers and end use markets. (report on WRAP website)
TRADA	Sorting of preservative treated timber	identify and evaluate techniques for sorting preservative treated wood to enable recycling
TRADA	Recycling of MDF and particleboard	ends March 2004 see. Next stages are scale up reactor and panel production
TRADA	Embodied Wood	UK wood industry mass balance and efficiency of use
TRADA	Waste Management: a pragmatic approach to SMEs through IT	changing attitudes through the use of five exemplar case studies of wood waste management in the joinery and construction sectors. Links to a web based tool kit to help SMEs assess waste levels and management options
Imperial College London	Predicting volumes of CCA treated timber in waste streams over the next 40 years	additional end of life research and the economics of energy recovery
BRE	Recycling opportunities for glass, timber and plastic from replacement windows sector	divert waste from landfill and to identify recycling opportunities
BRE	Cost effectiveness of collection of construction site packaging wastes	identify volumes and quantities available for reprocessing and potential collection mechanisms.
BRE	Deconstruction and reuse of construction materials.	provide a UK market share report, consider effective management and integration of products
BRE	Maximising the effective use of construction wood waste	developing a UK wood waste classification system, presenting a range of waste management options for each category, using case studies with industry and BPEO to promote change
BRE	Reduction in landfill disposal through the application of bioremediation and composting	applying techniques for remediation of brownfield site pollution to waste wood streams for the production of landscaping composts. Reducing any impact of contaminants
BRE	Technology for minimising the environmental impact from treated timber products	emissions to indoor air from panel products made from recycled timber

* Association of British Furniture Manufacturers

6 Conclusions and Opportunities

This paper has shown that in the UK there are considerable volumes of wood waste being generated by the construction industry and associated sectors. Wood waste can often be stockpiled for some time and there is little economic incentive to sort through these stockpiles for wood which can be reused in its current form or for higher value timber (e.g. certain types of hardwoods). Wood recyclers are keen to explore more markets for wood recyclate. Increasingly more wood, especially contaminated wood waste, is being sent for energy recovery. Combined Heat and Power (CHP) plants and other forms of boilers are becoming more common in the manufacturing industries. Wood off-cuts and residues will often go directly to these facilities on site. The combustion of contaminated wood should consider appropriate furnace conditions, flue gas scrubbers and the disposal route for the ashes.

It is important to classify the waste generating activities and wood waste types into a common format to make it easier to access the BPEO and thus potential opportunities to reuse, recycle, recover energy, compost or landfill. In the UK the reclamation industry, the demolition industry and the panel product manufacturers are already capitalising on some of the opportunities. The challenge is to extend this to other sectors. Future work will take further steps to bring together the producers and users of wood waste.

Opportunities for increasing sustainable wood waste management:

- Mechanisms to facilitate trading of resources
- High-value niche markets
- Affordable systems for reprocessing
- Markets for contaminated timber
- Geographical spread of wood processors
- Government encouragement of re-use and recycling
- Energy from waste wood

Through a combination of UK Government sponsorship and industry ventures, a substantial and integrated range of initiatives have been launched in the UK to promote better awareness of and more effective actions towards the management of timber wastes. The 5 million tonnes of wood waste that is currently being sent to landfill or being incinerated (without energy recovery) per year in the UK offers great opportunities for reuse, recycling and energy recovery. There are an abundance of tools and potential processes into which this resource could be directed. It is necessary to complete feasibility studies and understand the BPEO for each specific wood waste sources. The combination of the available tools, a growing research database, specifications for recyclate feedstocks and the incentives to buy recycled products will ensure a future of continued innovation and development. There is still a need to evaluate and develop sustainable, stable and above all efficient markets for the recycled wood products.

A key area for future research is the use of wood waste as fuel and the development of an energy recovery strategy by UK Government, the construction industry and research organisations. The segregation of wood waste is a complex and highly expensive option, that relies on understanding the flows of wood waste and the provision of consistent feedstocks to manufacturers of recycled products. This paper has not dealt in detail with aspects of energy recovery although this practice will be an increasingly important component of future wood waste management. A considerable wealth of research and experience exists and is being developed in France, Germany, Sweden and Finland. The UK can learn from progress in these member states. The suitability and application of these experiences into the UK would need to meet the physical, political and economic conditions of the country or particular region. The challenge will be to first of all acknowledge what is being achieved elsewhere (mechanisms like COST Actions E22 and E31 help this process), secondly to appreciate the mechanisms and principles of its success and then finally to apply the system to UK where it is deemed to be practicable and economically viable. This will take time but the opportunities for making the most of our wood waste are diverse and valuable both to industry and the environment.

7 Acknowledgements

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8 References

BRE Information Papers and Digests, available from Construction Research Communications Ltd, 151 Rosebery Avenue, London EC1R 4QX, Telephone +44 (44)20 7505 6622, Fax: (44)20 7505 6606, E-mail: crc@construct.emap.co.uk or from www.brebookshop.com

IP3/97	Demonstration of re-use and recycling of materials
IP1/96	Management of construction and demolition waste
IP 7/00	Reclamation and recycling of building materials
Digest 447	Waste minimization on a construction site
BR418	Deconstruction and reuse of construction materials
IP9/03	Best practice of wood waste management.

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The Symonds Report is a European source of C&D waste information. http://europa.eu.int/comm/environment/waste/report.htm

Timber Best Practice Website: http://www.timberbestpractcie.org.uk

WASTE WOOD MANAGEMENT IN SWEDEN – AN UPDATE

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Keywords: Waste wood, Sweden, CCA, preservative-treated wood

Abstract: *This paper summarizes briefly some recent Swedish findings related to waste wood:*

- *Approximately 14 000 tons As and 9 000 tonnes of copper and chromium respectively have been used for treating wood for the domestic market since 1970.*
- *The use of CCA has dropped dramatically during the 1990s.*
- *The waste wood stream is expected to contain approximately 5 % preservative-treated wood during the next 25-30 years.*
- *Waste wood fuels are always contaminated by active ingredients from wood preservatives.*
- *Contaminants, such as Pb, Zn and Cl, cause problems with slagging, fouling and corrosion (Cl) in the boilers. These contaminants appear mainly in the finer fractions of the waste wood.*
- *Incineration with energy recovery (electricity generation, district heating) of unsorted waste wood, i.e. no need to separate painted wood and preservative-treated wood in particular, in plants that comply with environmental regulations seems to be the most promising alternative for waste wood management.*

1 Introduction

Waste wood has been increasingly used as a fuel in Sweden during the last ten years.
Incineration with energy recovery is presently the main waste wood management option and the use of waste wood fuels corresponds to approximately 1 500 GWh per year (Andersson, Tullin, 1999). The waste wood is of domestic origin as well as imported, mainly from Germany and the Netherlands.

Decommissioned creosoted railway sleepers and poles are normally separated in different waste streams, whereas wood treated with other preservatives, with few exceptions, is mixed into a general stream of waste wood.

The recent research and studies related to waste wood have been focussed mainly on operational problems, such as slagging, fouling of heat exchanger surfaces and accelerated corrosion.

2 Preservative-treated wood

The amounts of wood preservative active ingredients used in Sweden before and since 1970 for the production of treated wood for the *domestic* market are shown in Table 1. The reason for including the amounts before and after 1970 is that in the early 1970s the use of treated sawn timber increased greatly. Thus, that timber treated from 1970 is of primary interest from a waste management point of view.

The CCA-type wood preservatives have dominated the market in Sweden since the early 1950s. For about 40 years the well-known Boliden K33 formulation (a CCA type B according to AWPA Standard P5) dominated. In the early 1990s there was a shift to CCA type C formulations which contain less arsenic. With the aim of reducing the use and distribution of potentially environmentally dangerous substances, the Swedish Chemicals Inspectorate (KemI) already in 1992 introduced restrictions for the use of wood treated with arsenic and chromium containing wood preservatives.

The KemI restrictions had a dramatic impact on the use of CCA-treated sawn timber for the domestic preservative-treated wood market. It decreased from approximately 85 % before 1992 to below 40 % in 1994, whereas the arsenic and chromium free preservatives (C/Övr. in Fig. 1) increased their market share from about 10 % to nearly 60 % during the same period, see Figure 1.

Table 1. Amounts of wood preservative active ingredients used in Sweden before 1970 and from 1970 to 1997 for the production of treated wood for the domestic market (Jermer, Nilsson, 1999).

	Amounts used (tonnes of elements and compounds respectively)		
Active ingredient	Before 1970	Between 1970 and 1997	Total
Elements			
Arsenic	6 600	7 700	14 300
Boron	-	150	150
Phosphorous	-	270	270
Copper	2 500	6 100	8 600
Chromium	3 000	5 900	8 900
Zinc	2 900	-	2 900
Tin (in organotins)	-	40	40
Compounds			
Creosote	499 000	145 000	644 000
Pentachlorophenol	190	450	640
Quats	-	500	500
Triazoles	-	12	12
IPBC	-	5	5

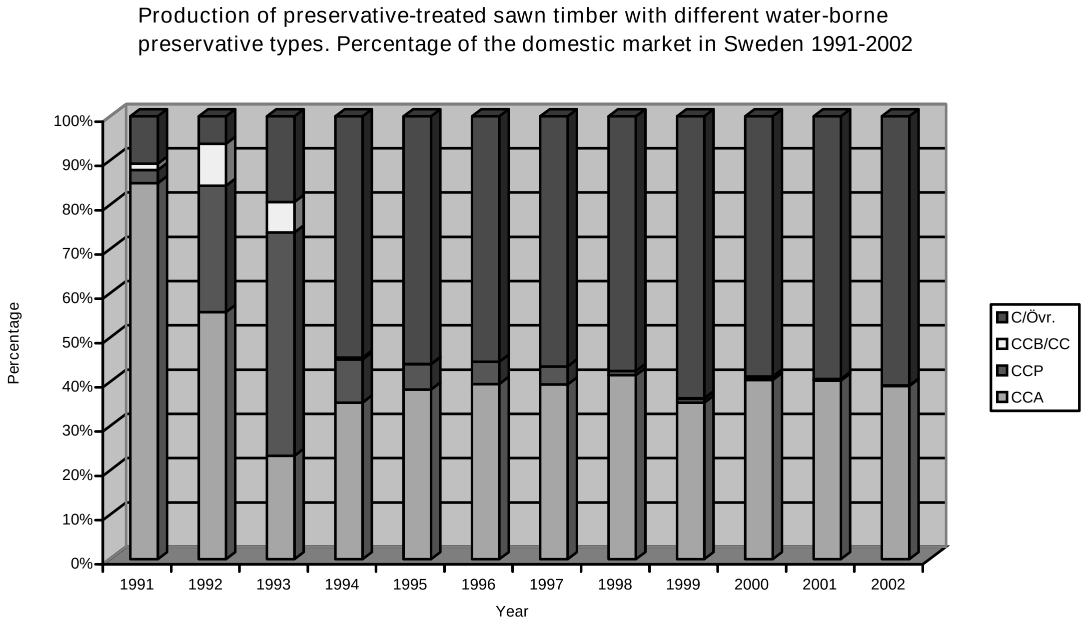

Figure 1. Production of preservative-treated sawn timber with different water-borne preservative types. Percentage of the domestic market in Sweden 1991-2002.

Since the 1970s the use of sawn and profiled treated wood has constituted between 5 and 8 % of the total market of sawn and profiled timber in Sweden, Figure 2. Assuming a service life of the treated wood of 25-30 years, it is reasonable to estimate that the wood waste flow will contain approximately 5 % preservative-treated wood, apart from poles and railway sleepers, during the upcoming 25-year period (Jermer, Ekvall, Tullin, 2001).

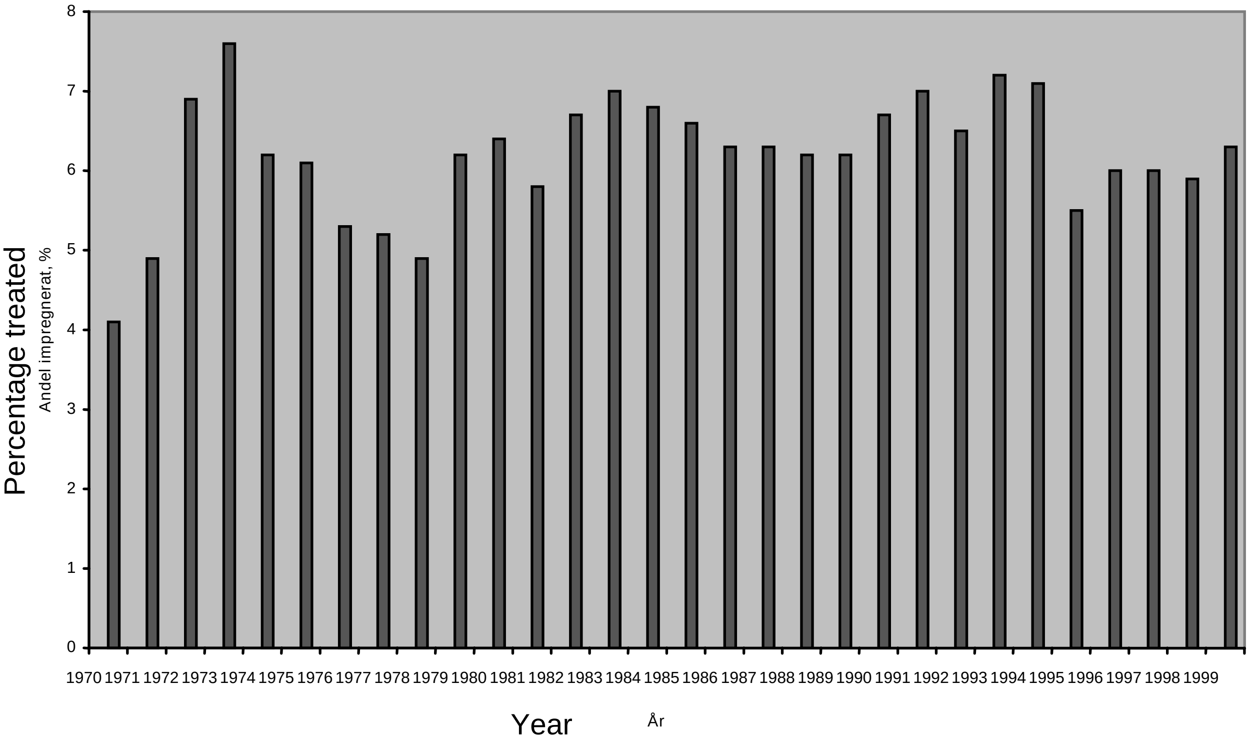

Figure 2. Percentage of preservative-treated sawn timber used in relation to total consumption of sawn timber in Sweden during the period 1970-1999.

3 Contaminants in waste wood

Recent studies, which were part of an extensive research programme on combustion of sorted waste wood (Jermer, Ekvall, Tullin, 2001; von Bahr, Ekvall, 2003), have shown that the major chemical contaminants of waste wood are surface treatments (paints etc) and wood preservatives. The surface treatments contribute in particular to contaminants of zinc and lead. Zinc can cause severe deposit formation in the furnaces. Surface treatments also contribute to increased levels of sodium, chlorine, sulphur and nitrogen.

A large number of analyses of fuel chips from waste wood and forest residues respectively were carried out. Some results of these analyses are presented in Table 2.

For each element the average content with standard deviation has been calculated. The ash content was on average 5,7±6,2 % for waste wood chips and 3,1±1,7 % for forest residues.

Table 2. Composition of waste wood and forest residues with respect to As, Cu, Cr, Na, Cl, Pb and Zn (Jermer, Ekvall, Tullin, 2001).

Elements (mg/kg dry wood)	As	Cu	Cr	Na	Cl	Pb	Zn
Waste wood Average	21	32	41	665	641	72	350
Waste wood Standard deviation	20	32	32	300	550	81	260
No. of analyses	47	42	45	32	47	50	51
Forest residues Average	1,40	7,50	10	275	211	7,3	89
Forest residues Standard deviation	1,6	6,50	8,4	190	66	8,3	76
No. of analyses	17	10	12	11	13	14	19

As can be seen from the table, the waste wood contents of CCA residues are considerably higher than in forest residues but still small compared to freshly treated wood, where, for example the arsenic contents is between 1 600 and 6 000 mg/kg sapwood. Extrapolation of the values for As and Cu to proportions of CCA in treated wood suggest that the material analysed may contain something between about 1 % and 2 % of CCA treated wood by volume, assuming a retention of approximately 10 kg/m^3 sapwood.

When analysing different fractions of the waste wood, it was found that Zn, Pb, Na, K and Cl, i.e. the contaminants causing deposits, plugging of air opening and corrosion (Cl) all were more or less concentrated in the finer fractions. Results from sieving of waste wood fuel chips indicate that approximately 60 % (~40 % for the Cl) of the elements related to deposits and corrosion (Zn, Pb, Na, K) is found in approximately 30 % of the fuel-chips, corresponding to the fine fraction (3-6,5 mm) and dust fraction (< 3 mm), see Figures 3 and 4.

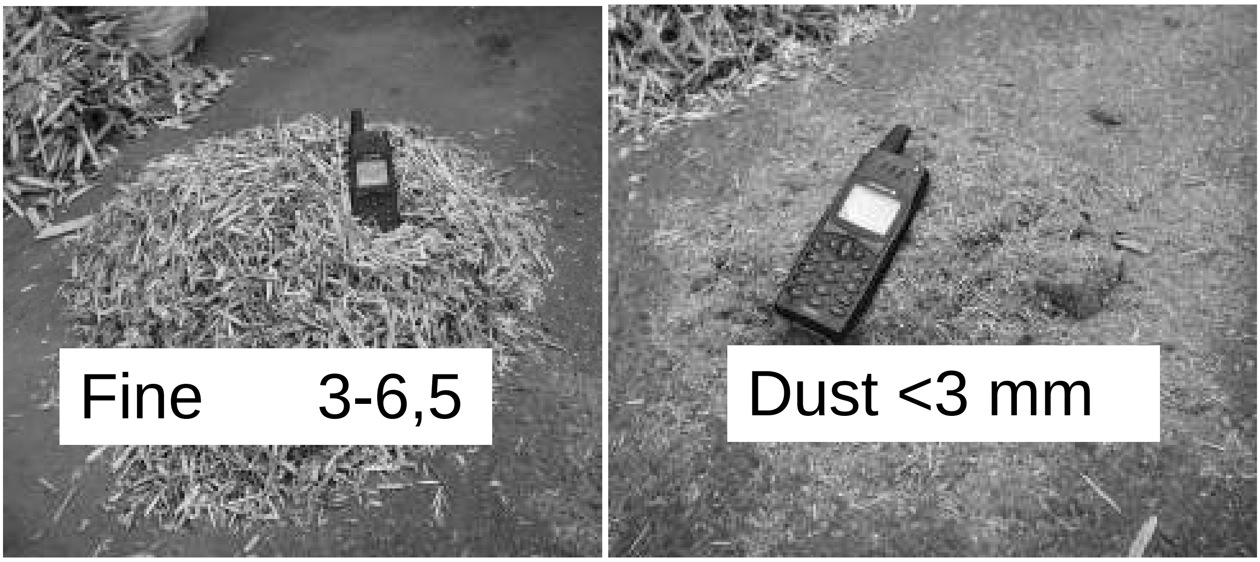

Figure 3. Illustration of fine and dust fraction respectively.

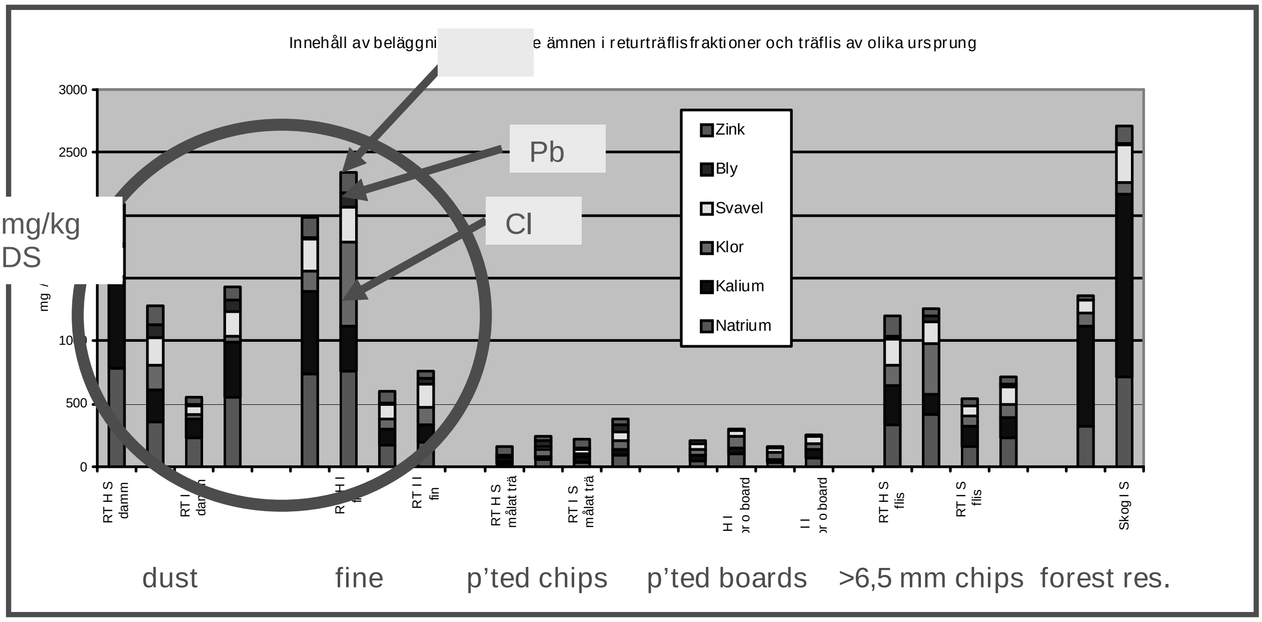

Figure 4. Contents of elements related to deposits/corrosion in different fractions of waste wood of different origin.

Preservative-treated wood is the most important source of increased levels of copper, chromium and arsenic in the waste wood. The contents vary a lot between waste woods of different origins. The arsenic content is higher in waste wood of Swedish origin than in waste wood of German or Dutch origin. This is consistent with the fact that CCA type wood preservatives have been used to a much lesser extent in Germany and the Netherlands. The treated wood is also mainly found in the >6,5 mm fraction, see Figure 5.

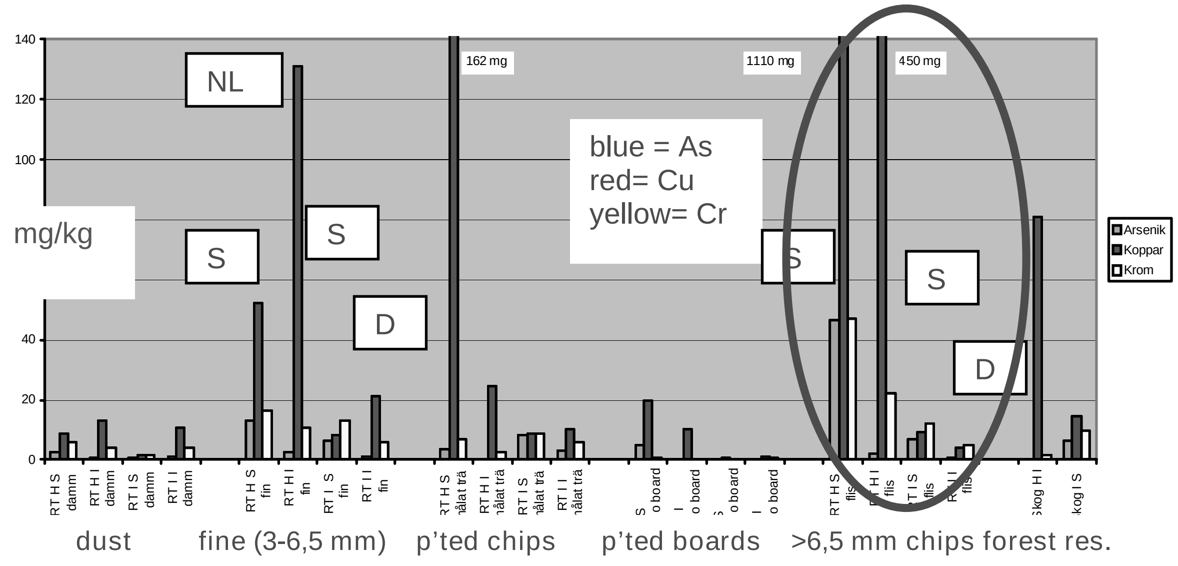

Figure 5. Contents of preservative-treated wood in different fractions of waste wood fuel chips of different origin. D = German, NL = Dutch, S = Swedish.

4 Final words

A fundamental question which requires further consideration is whether one should focus on “upstream” technologies, i.e more careful source sorting, or “down-stream” technologies, i.e. try to reduce the problems with the boilers and handling of the ashes (Krook, 2003).

The most important result from recent research in Sweden is a number of recommendations on different measures on how to minimize the combustion problems with waste wood, see Table 3.

Table 3. Proposals for minimizing the combustion problems with waste wood.

General measure	Specific measure	Effect
Improving the fuel quality	a) Improved source sorting b) Sieving of the fuel	a) Reducing the amount of metals and chlorine b) Separation of the fine fractions and thereby reducing the amount of metals
Combustion modifications	Avoid reducing conditions at the heat exchanger surfaces	Minimizing slagging, fouling and corrosion
Additives or co-combustion	a) Adding sulphur to the fuel b) Injection of sulphur containing substances in the furnace c) Addition of additives or fuels containing silica	a) Sulphating of troublesome metal chlorides b) Sulphating of troublesome metal chlorides c) Adsorption of troublesome substances

Clearly, more careful sorting or sieving will reduce the problems in the boilers and also with the ashes. But is it realistic with the tools available, the amount of work needed and the associated costs, bearing in mind that a substantial amount of the wood in use is contaminated with surface treatments, wood preservatives, fasteners etc? An important question is also, what to do with the sieved out fines?

An efficient waste wood management must be simple to understand and easily applicable for all the parties involved. Incineration with energy recovery (electricity generation, district heating) of unsorted waste wood, i.e. no need to separate painted wood and preservative-treated wood in particular, in plants that comply with environmental regulations seems to be the most promising alternative.

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WASTE MANAGEMENT FOR TREATED WOOD IN DIFFERENT EUROPEAN COUNTRIES

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Table 1: Production of wood wastes

Country	untreated amount [to]	treated amount [to]	CCA treated amount [to]
B	1,200.000	N/D	200.000
CH	N/D	N/D	N/D
D	8,000.000	1.900 – 2.800	-
DK	?	65.000	58.500
FIN (VTT)	530.000	10.000	9.000
F	12,100.000	3,600.000	100.000 – 200.000
GR	90.000 - 100.000	10.000	1.000
IRL	N/D	N/D	N/D
NL	N/D	400.000***	120.000***
N	1,200.000	100.000	15.000
RO*	2,320.000	42.300**	-
UK	7,300.000	80.000	40.000

* Unfortunately there are no available data (N/D) concerning the amount of treated wood waste (including those treated with CCA).
There are no collects (recoveries) of treated wood waste organized at national level.

** This is from the sawdust/shavings from some wood products manufacturing (different chemicals – not preservatives – are also used)

*** Original figures in m^3 (conversion x 0,5 ~ to)

Table 2: Kind of waste wood reuse

Kind of reuse/Country		untreated amount [to]	Treated amount [to]	CCA treated amount [to]
Recycling / reuse e.g. particle board	B	816.000	156.000	N/D
	CH	N/D	N/D	N/D
	D	N/D	N/D	-
	DK	-	0	-
	FIN (VTT)	5.000	N/D	N/D
	F	6,000.000	<100.000	<10.000
	GR	50.000	N/D	N/D
	IRL*	N/D	N/D	N/D
	NL	844.000	127.000	5.100
	N	N/D	N/D	N/D
	RO*	260.000	-	-
	UK	1,470.000**	80.000	40.000
Landfill	B	N/D	N/D	N/D
	CH	banned	banned	banned
	IRL	N/D	N/D	N/D
	D	banned	banned	banned
	DK	-	60.000 in priciple	all – in principle
	FIN (VTT)	5.000	N/D	N/D
	F	3,000.000	1,300.000	100.000
	GR	N/D	5.000	N/D
	NL	0	0	0
	N	20 %	30 % (?)	0 % (?)
	RO*	110 000	-	-
	UK	ca 5,530.000	80.000 to	All - ~ 40.000
Energy production	B	336.000	164.000	N/D
	CH	N/D	N/D	N/D
	D	N/D	N/D	N/D
	DK	-	0	-
	FIN (VTT)	295.000	10.000	9.000
	F	1,500.000	<500.000	0
	GR	20.000	2.000	N/D
	IRL	N/D	N/D	N/D
	NL	200.000	1.200	500
	N	80 %	70 % (?)	100 % (?)*
	RO*	1,520.000	-	-
	UK	unknown		
Special dumps	B	N/D	N/D	N/D
	CH	N/D	N/D	N/D
	D	-	-	-
	DK	-	0	-
	FIN (VTT)	225.000	N/D	N/D
	F	0	?	0
	GR	N/D	N/D	N/D
	IRL	N/D	N/D	N/D
	NL	0	0	0
	N	1,200.000	100.000	15.000
	RO*	430.000	-	-
	UK	Very little		

* IRL - Again, N/D available. However, much of construction waste currently goes into landfill. There are re-cycling programmes underway to reduce this. At present, there is, to my knowledge, any separation of any wood waste.

There is a lot of interest at present at sawmill level to use wood waste for energy production and it is expected that this is a good possibility. At present, sawmill wood residue gose to the various particle-board plants (chipboard, OSB, MDF, hardboard).

There is at least one company which will go to a factory to grind up waste wood eg pallets into chips.

We have no special dumps. If something is classified as a harazdous wood wastel, it will be shipped to eg Finland for high temperature incineration – at a high cost!

** In UK 630.000 tonnes is reused, 840.000 tonnes is recycled to panel production ND = no data

Table 3: Waste wood regulations

Kind of use		Regulation			
		Exists	Does not exist	In praparation	Coming into force (year)
Recycling / reuse e.g. particle board	B				
	CH		X		
	D	X			
	DK		X		
	FIN (VTT)	X			
	F		X		
	GR		X		
	IRL	X (in principle)			
	NL	X			
	N		X		No
	RO*		X		
	UK			X	2004
Landfill	B	X			
	CH	banned			
	D	X			
	DK	X			
	FIN (VTT)		X		
	F	X (9/9/97)		Revision	
	GR	X			
	IRL	X			
	NL	X			
	N		X		
	RO*		X		
	UK			X	2004 (maybe 2007)
Energy production	B	X			
	CH	+			
	D	X			
	DK		excluded		
	FIN (VTT)			X	
	F	X[1]		Revision	
	GR			X	
	IRL		X		
	NL	X			
	N		X	X	1 - 3 years
	RO*				
	UK		X		
Special dumps	B	X			
	CH	banned			
	D	X			
	DK		X		
	FIN (VTT)		X		
	F	X (18/12/92)		Revision	
	G	N/D			
	IRL	X			
	NL	X			
	N		X		
	RO*		X		
	UK	X			

* In RO there is no specific legislation concerning the use of wood waste as you have indicated.
In accordance with EU legislation that covers that field, respectively the Council Directive 75/442/CEE – the European Catalogue for Waste, published by Council Decision 94/3/CE and reviewed by the Decision 2000/532/EC, have been issued:

- HG 155/8.03.1999 – Decision regarding the introduction of waste management and of the European Catalogue for Waste;
- OUG no. 78/16.06.2000 – Regulation regarding waste (storage, manipulating, utilization, destruction);
- HG 128/14.02.2002 – Decision regarding the waste incineration.

PROPOSAL FOR A CONCEPT FOR TREATED WOOD WASTE MANAGEMENT

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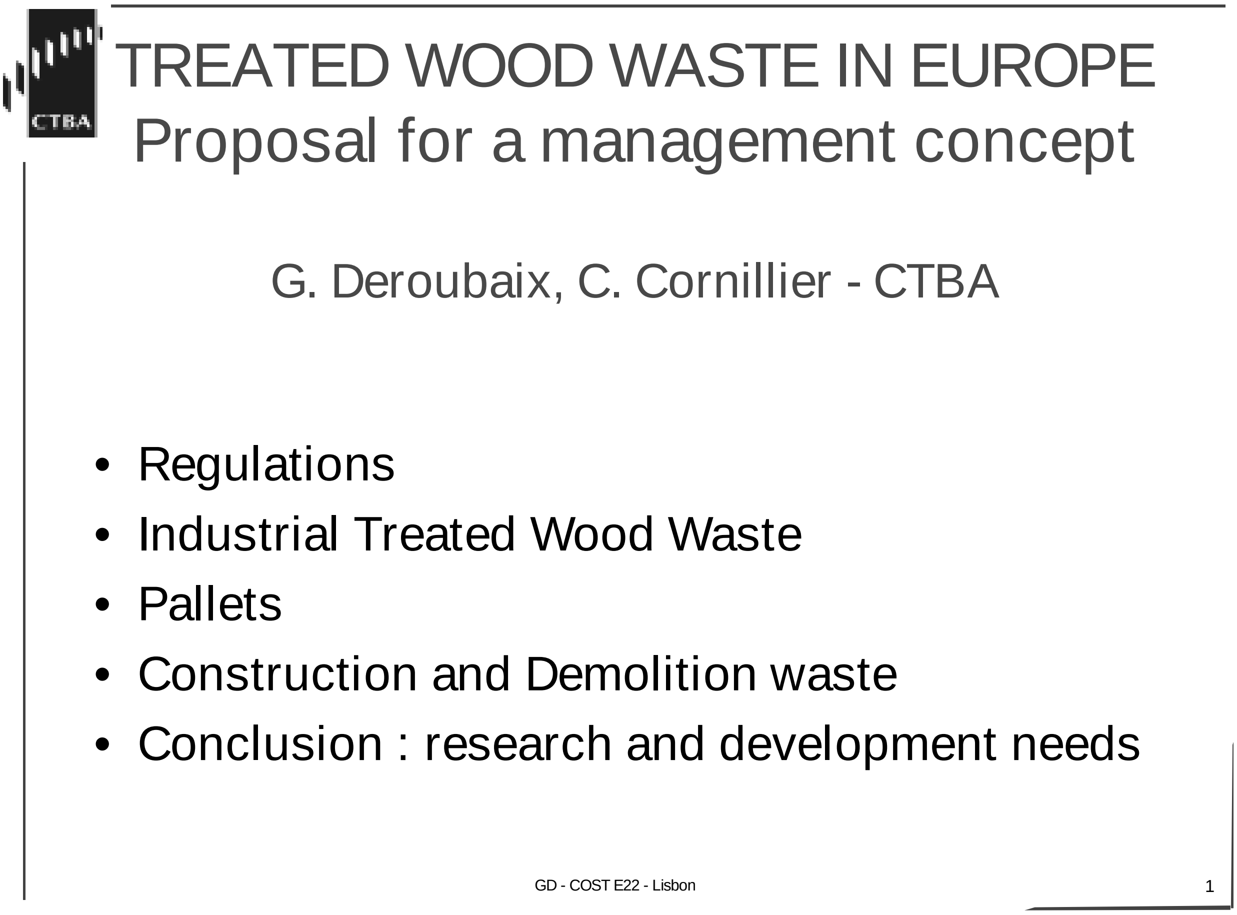


TREATED WOOD WASTE IN EUROPE
Proposal for a management concept

G. Deroubaix, C. Cornillier - CTBA

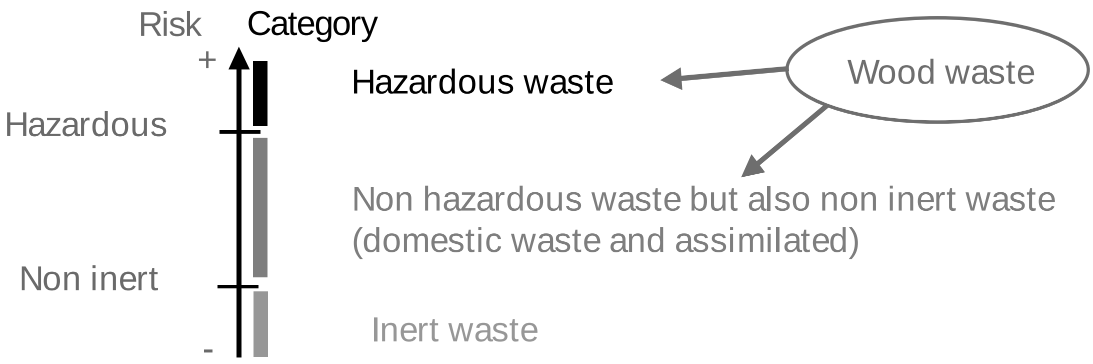
- Regulations
- Industrial Treated Wood Waste
- Pallets
- Construction and Demolition waste
- Conclusion : research and development needs

1- Regulations : Waste classification principles

- Wastes classification according to :
    - risk assessment
    - a codification
    - management pathways (energy recovery or material recycling)

Waste classification according to risk assessment

- Defined at European level :
  - – Directive 91/689/EEC modified
  - – Decision 2000/532/EC modified
- 3 categories according to 2 risk levels :



Hazardous waste

Non hazardous waste but also non inert waste (domestic waste and assimilated)

Inert waste

Definition of the hazardous waste characteristic

- Waste containing any of the hazardous constituents listed in annex 2 of directive 91/689/EEC (51 constituents)

and

- Waste showing any of the hazardous properties listed in annex 3 of directive 91/689/EEC (14 hazardous properties) or containing substances classified as hazardous and at a total concentration exceeding a threshold level

Hazardous qualification for treated wood waste

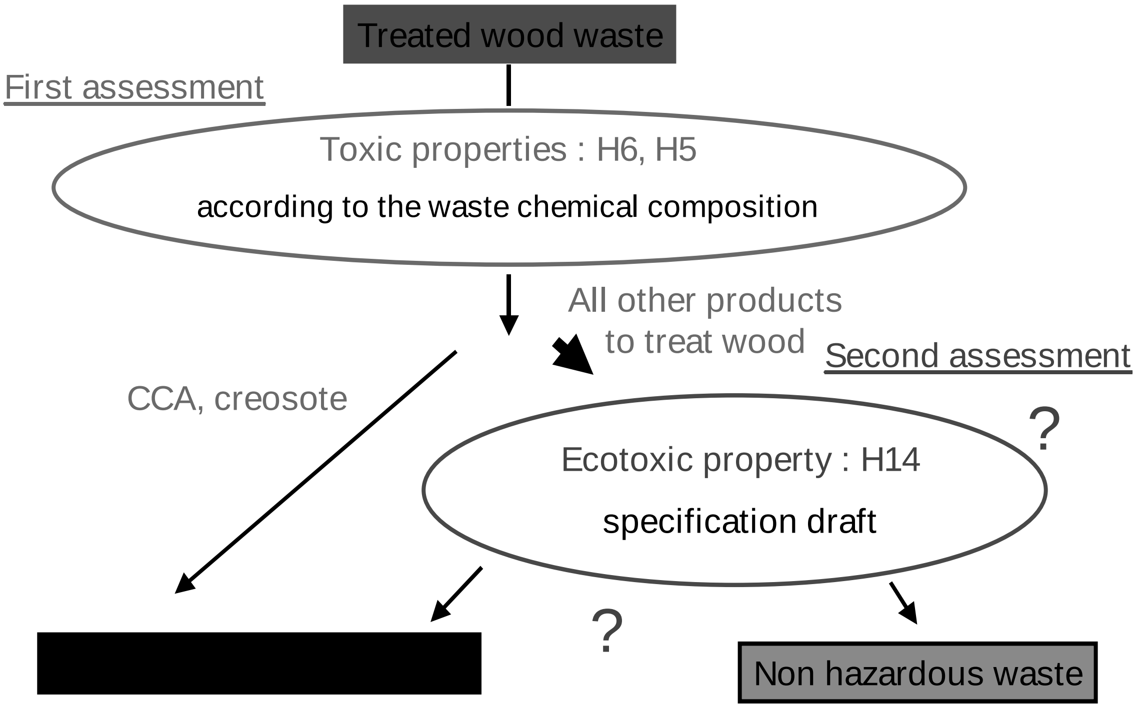
- Hazardous constituents : biocides included

➡ Treated wood waste always have at least one of the hazardous constituents listed in annex 2

- Main hazardous properties :
  - H6 : - very toxic, or containing very toxic substances at a total C ≥ 0,1%
    - toxic, or containing toxic substances at a total C ≥ 3%
  - H5 : - harmful, or containing harmful substances at a total C ≥ 25%
  - H14: - ecotoxic (still no regulation specifications)

➡ The hazardous properties of treated wood waste can be assessed (but ecotoxic)

Assessment of treated wood waste properties



Treated wood waste

First assessment

Toxic properties : H6, H5
according to the waste chemical composition

All other products to treat wood

Second assessment

CCA, creosote

?

Ecotoxic property : H14
specification draft

[redacted]

?

Non hazardous waste

Classification according to an established codification :

(Decision 2000/532/EC modified)

Hazardous wood waste in parallel to non hazardous wood waste

hazardous wood wastes in EC regulation

HAZARDOUS WASTES	NON HAZARDOUS WASTES
Wastes from wood processing and the production paper Cardboard, pulp, panels and furniture	
03 01 04* : sawdust, shavings, cuttings, wood, particule board and veneer containing dangerous substances	03 01 05 : sawdust, shavings, cuttings, wood, particule board and veneer other than those mentioned 03 01 04
Waste packaging, absorbents, wiping, …	
15 01 10* : packaging containing residues of or contaminated by dangerous substances	15 01 03 : wooden packaging
Construction and demolition wastes (including excavated soil from contaminated sites)	
17 02 04* : wood containing or contaminated with dangerous substances	17 02 01 : wood
Municipal waste including separately collected fractions	
20 01 37* : wood containing dangerous substances	20 01 38 : wood other than mentioned in 20 01 37

Classification according to the management pathways

- Wood waste recycling in particle board manufacturing : requirements on final products (European Panel Federation specifications : heavy metals, PCP, BaP)
- Wood waste energy recovery
  - – waste wood fuel defined by the “combustion equipments directive” 2001/80/EC
  - – are excluded : wood wastes containing organochlorinated compounds or heavy metals ; these wastes are in the field of the “waste incineration directive” 2000/76/EC

2- What is treated wood waste : thresholds

- Waste wood fuels : definition through chemical composition, taking into account :
  - incineration and combustion regulations : “regulated substances”
  - biocides, their type and concentration into the wood waste
  - natural content of wood in heavy metals, and in organochlorines (as PCP environmental contamination)
- Waste wood recycling for particle boards : meeting the EPF criteria
- Specifications can be set up on the basis of an analysis of the different waste resources :
  - industrial waste, pallet chips, specified wastes

Chemical composition of wood waste fuels

Substance or compound	Regulated	Additives		Natural concentration		Threshold proposal
		Use	Conc.	Max mean	Max value	
As	Yes	P	< 7000 ppm	1,4 ppm	6,8 ppm	+10
Cd	Yes	F	< 5000 ppm	0,7 ppm	3,8 ppm	+10
Cr	Yes	P, F	< 10000 ppm	22,6 ppm	130,0 ppm	+100
Cu	Yes	P, F	< 6000 ppm	21,9 ppm	400,0 ppm	+400
Mn	Yes	F	?	180,6 ppm	1100,0 ppm	+
(Pb)	Yes	F	< 5000 ppm	29,0 ppm	340,0 ppm	+100
Zn	Yes	F	< 5000 ppm	228,0 ppm	228,0 ppm	+
						Total < 1000 ppm
Cl	Yes	P, C	1%	1100,0 ppm	11890,0 ppm	1%
(F)	Yes	P	?	40,0 ppm	490,0 ppm	0,5%
N	Yes	C	5%	5000,0 ppm	34100,0 ppm	5%
S	Yes	C	?	700,0 ppm	8800,0 ppm	?
Organo-halogénés totaux (PCP, lindane, aldrine, endosulfan Pyréthrinoïdes, triazoles)	Yes	P	< 1000 ppm	3,8 ppm	/	10 ppm
HAP (créosote)	No	P	< 25000 ppm	3,1 ppm	15,6 ppm	1000 ppm
B	No	P	< 1000 ppm	15 ppm	60,0 ppm	1000 ppm
Sn	No	P	< 1000 ppm	1,7 ppm	10,0 ppm	1000 ppm
Quats	No	P	< 5000 ppm	/	/	5000 ppm
Glues and resins	No	C, F	15%	/	/	15%

Chemical composition of waste wood for recycling (EPF criteria)

substances	threshold (mg/kg)
Arsenic	25
Cadmium	50
Chrome	25
Cuivre	40
Plomb	90
Mercure	25
Fluorine	100
Chlore	1000
Pentachlorophenol	5
Créosote (benzo(a)pyrène)	0.5

REGULATORY PRINCIPLES ABOUT WASTE

- To reduce generation
- To sort : no mixing of hazardous waste with non hazardous waste
- To try to recycle waste products or to recover energy
- To landfill only ultimate waste
- To have waste collected and managed by specialised companies
- To have traceability of hazardous waste from the producer to the final treatment

3- Management of industrial treated wood waste : Industrial treated wood waste

- Few heavy metals and organohalogenes treated wood waste
- Reduction of amounts by process changes
- Easily sorted : the source points are easy to identify
- Wood waste (*including minor amounts of treated wood waste ? NO*) meeting the requirements may go to recycling or energy recovery (ex production specifications next slide)
- The remaining part should go to special treatment processes

Industrial wood waste fuel requirements

- In the case of CCA, copper based products, organohalogenes and creosote, any off-cuts after treatment to be discarded
- In the case of metallic pigments (Cd, Cr, Zn) any off-cuts after finishing to be discarded
- PVC containing off-cuts to be discarded
- Panels and glues containing chlorine based hardeners not to be used
- Metals, organohalogenes, PAH, Chlorine levels to be checked according to previous thresholds

Industrial treated wood waste : development needs

- Quick characterisation systems
- Simple thermal treatment equipments for organic treated wood waste (with no harmful emissions)
- Production processes allowing treatment to take place at the very end of the transformation process

4- Pallets

- Small amounts of organohalogenated substances
- Depending on content level, different possibilities :
  - Thresholds for paticle boards
  - Thresholds for combustion
- Need for « pallet chips production sites » able to produce a defined product, either for particle board industry or for combustion

Pallets : development needs

- Characterisation tools: fast, cheap, high sensitivity
- Identification of treated wood pallets ?
- Development of non-organohalogenated biocides for packaging ?

4 - Construction and demolition waste

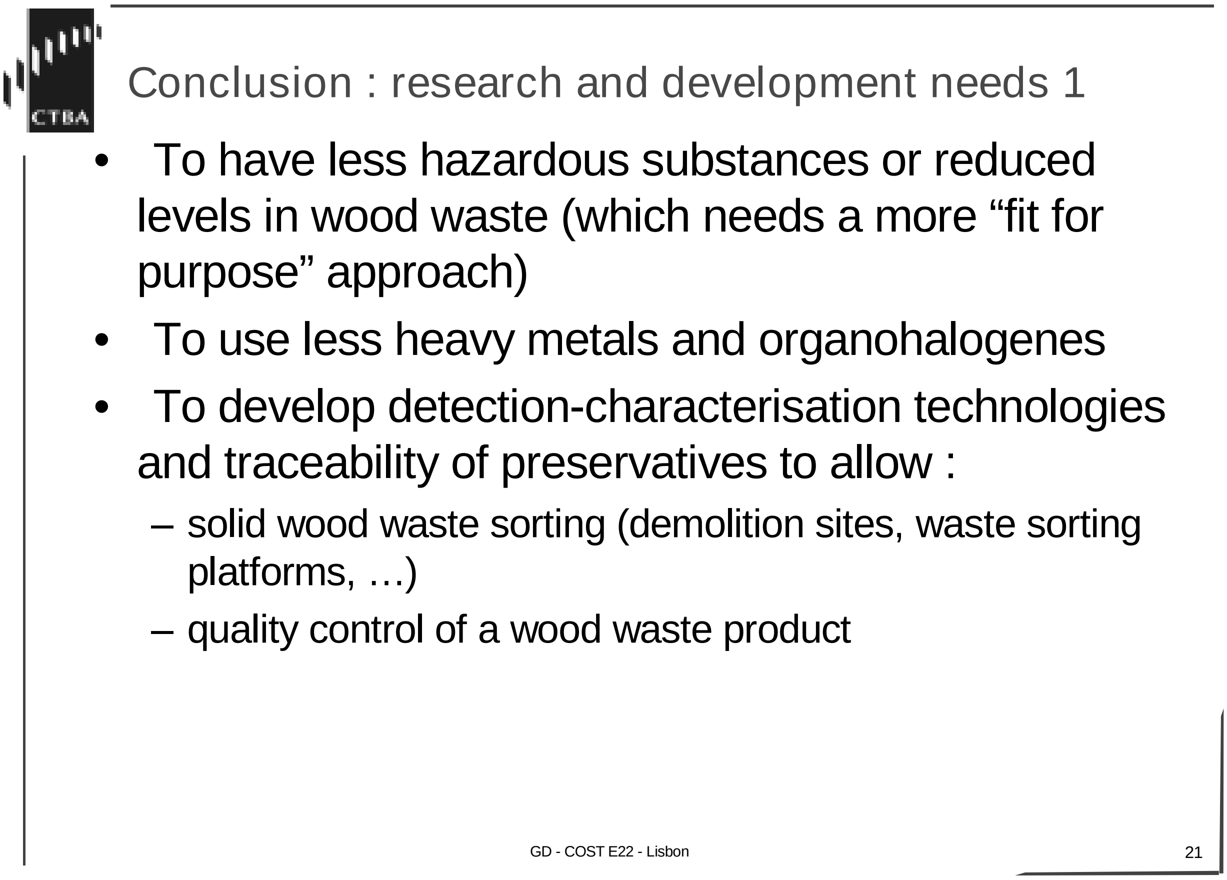
- Any kind of treated wood plus many other kinds of problematic contaminants
- Sorting :
  - Outdoor wood is suspect to include heavy metals
    - > special treatment processes
  - Indoor wood : contaminants concentrations should be lower - > may go to a « sorting site » to be characterised and possibly recycled or burned if requirements are met.

If sorting is not possible and/or contamination levels are too high

– developing high performance combustion equipments with filtration systems to produce energy, (with a regional distribution), allowing an economically acceptable wood waste management,

C and D waste : development needs

- Knowledge of the actual contamination of wood waste
- Characterisation tools: fast, cheap, high sensitivity
- Simplified incineration equipments or enhanced combustion equipments (quality of combustion and filtration)
- Identification of chemicals containing components



Conclusion : research and development needs 1

- To have less hazardous substances or reduced levels in wood waste (which needs a more “fit for purpose” approach)
- To use less heavy metals and organohalogenes
- To develop detection-characterisation technologies and traceability of preservatives to allow :
  - solid wood waste sorting (demolition sites, waste sorting platforms, …)
  - quality control of a wood waste product

ENVIRONMENTAL RISK ASSESSMENT- PROGRESS SO FAR

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Keywords: Environmental risk assessment, treated wood

Abstract: *The environmental risk assessment of wood protection products and treated wood is a key part of the new authorisation process for biocidal products in the EU. This paper considers some of the issues that face those compiling the dossiers for active substances and products under the Biocidal Products regulatory regime with respect to environmental risk assessment and the progress that has been made in dealing with these issues.*

1 Introduction

The ability of companies to place wood preservative products on the European market has, to date, depended on the regulatory control regime present in each country where the product is to be marketed. A wide range of regulations and approval processes can be found in the different countries, referred to from now on in this paper as Member States (MS). These have developed as a result of the national government policies and experiences in the use of wood preservatives in each MS.

As the European Community began to act in a more coherent manner with regard to chemical controls the EC Directive on the Marketing and Use of Dangerous Substances and its implementation was the vehicle used to control the marketing and use of certain dangerous substances in the EU. Examples affecting the wood preservation industry include organotin compounds, creosote and arsenic. This piecemeal approach was an unsatisfactory way to deal with the regulation of chemicals used as pesticides, and biocides.

In the EC Directive 91/414/EC a directive was introduced to regulate the use of pesticides in plant protection products. It was only a matter of time before a directive was introduced to regulate the use of chemicals used in biocidal products, such as wood preservatives. The Biocidal Products Directive 98/8/EC (BPD) was introduced and MS were given two years to enact national legislation for its implementation.

2 The Biocidal Products Directive 98/8/EC

This directive established a regulatory regime in which active substances used in biocidal products, such as wood preservatives, that had successfully cleared a review process would be placed on Annex I for use in the particular biocidal product type. Wood preservative products are called Product Type 8, and there are twenty- three product types in the directive. Industry applicants were required to prepare comprehensive dossiers on their active substances, and write the risk assessments (human health and environmental). The Competent Authorities in the Rapporteur Member States appointed by the EC to carry out the review for that particular active substance were required to assess the dossiers and risk assessments before making recommendations as to whether the active substance should be added to Annex I.

Once the active substance was included on Annex I then industry was required to make an application for the clearance of its products containing that active substance. It could then chose whichever MS it wished in which to file its first application. It was a key provision in the BPD that once a product had been cleared in a MS, the company then made subsequent applications in the MSs in which it wished to market the product. Each MS in which the applications were made was required to recognise by 'mutual recognition' the original product authorisation and grant a product authorisation in its own country.

The matrix of use where different active substances are used in different product types indicated there had to be a prioritisation process and it was decided the active substances in the product types in wood preservatives and rodenticides would be the first called for review.

The justification given for selecting wood preservatives as the priority product type was that the MS believed they had many years of experience in regulating wood preservatives and so the whole process for the implementation of the BPD would be more successful that that achieved for the implementation of the Plant Protection Products Directive 91/414/EC. Following the process of identification and notification by industry the Rapporteur Member State (RMS) for each active substance was appointed by the EC. The dossiers for Product Type 8 active substances are required to be with the RMS by the end of March 2004. It is anticipated that it will take about 1- 2 years for decisions to be made and consequently wood preservative product authorisations in MS will be 2006 /7, at the earliest.

As well as the active substance, industry was also required to consider the risks associated with 'substances of concern' that they might include in their products.

3 Dossier preparation

The dossiers to be prepared for each active substance also required information to be supplied for a biocidal product of the same biocidal product type.

The EC recognised the need to try to develop the procedures that would be understood by industry and regulator and which would be applied throughout the MS. To assist in this process the EC and MS together with input from industry, usually represented through CEFIC, set about developing Technical Notes for Guidance (TnGs), covering such areas as data requirements, dossier preparation, human health exposure, environmental risk assessment and so on. This was a prodigious undertaking, and even now the guidance is incomplete and sometimes inconsistent. Notice the use of the word 'guidance'; these TnGs are for guidance only, and this inevitably means there can still be uncertainty. This state of uncertainty between regulators and the regulated, i.e. industry, is not very satisfactory.

For industry, of particular concern is still the uncertainty as to how the entry on Annex I will be described. For example, at its simplest it could say 'Active substance X can be used in Product Type 8'. Alternatively at its most restrictive 'Active substance X can only be used In Product Type 8 formulations applied by vacuum pressure processes for timber to be used in Hazard Classes 3 and 4, at a maximum application rate of y kg /m3 , with the resulting treated timber only to be used by professional personnel'. Such uncertainty has profound implications on how a dossier is constructed.

As well as having the TnGs for guidance, in April 2003 the EC released the 2nd edition of the Technical Guidance Document on Risk Assessment of Chemical Substances following European Regulations and Directives, including guidance on human health and environmental exposure, and risk assessment.

4 Principles of environmental risk assessment

The EU BPD requires that a biocidal product can only be authorised if it 'has no unacceptable effect itself, or as a result of its residues, on the environment'.

As the intention of the BPD is to operate a legal basis for allowing or forbidding the use of active substances for particular applications, there is a need to have pass / fail criteria which can be used as the basis on which to justify decisions made. In the environmental assessments this is the so-called 'risk characterisation' and consists of the simple PEC: PNEC ratio.

In general if the ratio is greater than 1, then the risk is unacceptable and further refinement of the models or data generated on which the PEC and PNEC values are based is required; or the product requires further risk mitigation measures, for example restrictions in particular areas of application.

The PNEC (Predicted No – Effect Concentration) is the estimate of the relationship between the dose, or level of exposure of an active substance, or substance of concern in a biocidal product; and the incidence and severity of an effect. The effects assessment consists of establishing the PNEC for each relevant environmental compartment. A PNEC is regarded as the concentration below which, an unacceptable effect will most likely not occur. The derivation of the PNEC is a whole subject in itself, but suffice it to say that uncertainty factors are applied to the concentration. So for example, in the case of a dossier assessing aquatic impact effects where there is at least one short-term L(E)C50 from each of three trophic levels (fish, invertebrates and algae) an uncertainty factor of x 1000 is applied. On the other hand, if Long term NOEC (No observable effect concentrations) data is available for the same species then the uncertainty factor applied is x 10.

In the case of the terrestrial compartment, a situation where much treated timber is used, the variety of tests available to support a low uncertainty factor are few and in the absence of any test results with soil organisms, the equilibrium partitioning method developed for sediments can be used as a screening assessment.

The PNEC is clearly an important value, however it is not the main subject of this paper.
The PEC, Predicted Environmental Concentration, forms the exposure assessment part of the PEC:PNEC equation and this is the main subject of this paper.

5 PEC – Predicted Environmental Concentration

The PEC is the exposure assessment, and is the determination of the emissions, pathways, and rates of movement of an active substance, or substance of concern, in a biocidal product and its transformation or degradation in order to estimate the concentration / doses to which human populations, animals, or environmental compartments are or may be exposed. It consists of the three following steps:

- Release estimations;
- Estimations of the environmental behaviour;
- Estimation of environmental concentrations.

At face value the idea of calculating or measuring a Predicted Environmental Concentration is appealing, however, despite its widespread use in the regulation of agricultural pesticides, the application of the concept to the regulation of biocides, and materials treated with biocidal products, is raising issues for which at this point in time there appears to be little willingness on the part of the regulators to address. These issues will be discussed in this paper.

6 Emission Scenario Document

Biocidal products, such as wood preservatives, are applied and used in a wide variety of ways to control differing organisms over differing periods of time. This multiplicity of scenarios potentially makes the human health and environmental risk assessments extremely complex undertakings.
In the year 2000 the OECD established an Expert Group to prepare an Emission Scenario Document (ESD) that would describe the ways in which wood preservatives were applied, and the end uses to which the treated timber was put. The descriptions were then to be used as the basis for describing the methods of application; the potential for environmental impacts from the method of application; and also for representative scenarios covering typical uses of treated wood, selecting representative Use Class applications for treated timber.
The final draft of the OECD ESD was made available in 2003.
Scenarios from the OECD ESD were abstracted and published on the European Chemicals Bureau (ECB) website to be used by applicants in constructing their dossiers.

7 Models v monitoring

The BPD establishes a need for pre-marketing authorisation and, as the costs for generating data to cover the data requirements of the BPD are high, model calculations are used as an integral part of the risk assessment process. These models can be successively refined, i.e. a tiered approach, in the light of more data or experience. The most valuable data is that obtained during the use of a substance and, apart from the possibility of reading across from other methods of application or products, monitoring data is unlikely to be available at the time a dossier or application for a biocidal product is made. Models therefore are of key importance and the default values contained in them must be well defined and understood by those using and interpreting the results of modelling; as well as the regulator in understanding the limitations of the models themselves.

8 Determination of emissions from methods of application

Wood preservatives are applied by a number of techniques ranging from application by brush to application in large industrial timber treatment plants.

Even in the case of the industrial scale of application there are relatively few published papers detailing the losses to the environment from timber treatment installations during the course of their daily operations. Most monitoring carried out in the workplace, whether it is around treatment plants or remedial timber treatments, has been to assess human operator exposure. The methods used to determine such information are not suitable for estimating emissions to the environment.

In most EU countries there is some form of environmental protection regime in place and treatment installations may require a permit to operate. Such permits invariably require some form of offsite monitoring, however, the results of this monitoring are rarely collated and published so that a more informed understanding of the environmental performance of plants a can be made; and in the case of the BPD realistic defaults can be established in models. One consequence is that in the ESD for wood preservatives the default values set are high. For example, 3% of the product is lost daily to a surface drain going to an STP (Sewage Treatment Plant), despite the fact that plants must not have a link to an STP in order to be granted a licence to operate. Clearly such values are unrealistic and even if the 1% default value for point sources, as defined in the TGD on Risk Assessment, is used the quantities of wood preservatives allegedly being lost from timber treatment sites is still too high. Such loss rates would have resulted in a much higher environmental profile for timber treatment operations than is the current situation.

The same general comments are applicable to the other application techniques such as dipping, automated spraying and double vacuum, low pressure, treatment processes.

Some way will have to be found for data on emissions from different methods of application methods to be collated, so that more realistic default values for modelling purposes can be determined.

9 Post treatment conditioning period

The ESD separates out the post-treatment conditioning period, where the chemical and physical interactions take place between the wood and the product after treatment, and before the timber is moved into a post treatment storage facility. The scenario recognises that there will be certain processes and end use applications of timber where the timber will not be placed outside in the open air after treatment. Applicants have to justify such exemptions for such products.

There will however still be substantial quantities of timber that will be placed out in the open and potentially exposed to the effects of rainwater leaching. The scenario does recognise that only a fraction of the timber in a stack is exposed to leaching during the 30 days the timber is held in stock prior to being moved into the supply chain.

A key parameter is to establish the flux or emission rate of the active substance, or substance of concern, from the surfaces of the timber. There is a need therefore to determine a rate of loss based on an area basis. These losses are calculated, and using default values, the amount of substance lost to the soil; its concentration in the soil under the timber and losses off site are determined. The soil concentration is calculated for the dimensions of the soil-receiving compartment, namely the soil area under the timber and the default value selected for depth of soil.

10 Treated wood in service

Mention has already been made of the development of a number of representative scenarios for timbers to be exposed in Use or Hazard Classes 3, 4 and 5. These are described in detail in the OECD ESD. They all have one thing in common, and that is the need to measure the emission of active substance, or substance of concern, from the surface of the treated timber; then to try to characterise the emission profile with time, bearing in mind the service life of some treated timber commodities may be several decades.

11 Determination of emissions from treated timber

Lebow, in the conclusions of his literature review on the leaching of wood preservative components and their mobility in the environment, concluded that relatively little research has been done to quantify or evaluate leaching of preservatives from treated wood after it has been placed in service. Although numerous laboratory studies, and small-sized field studies have been conducted to evaluate the effect of various parameters on preservative fixation and leaching, these studies often have little applicability to in-service leaching rates. The majority of past research on preservative leaching has been laboratory studies designed to compare the effects of various factors on leaching or the leaching rates of various preservatives.

Willeitner and Peek (1998) presented an excellent reflection on how to determine what is a realistic emission from treated timber covering both evaporative losses and leaching by water.

In essence, the techniques used to determine the emissions from the treated timber must be representative of that found over the service life of the treated wooden article.

12 Evaluating the emission

Even if a realistic emission is achieved there are still considerable issues to be considered.

Chemical analytical techniques can be very costly and experience has shown that the quantities of water and emissions of substances required for robust analytical results are often considerably more than the emission tests deliver.

The ecotoxicity testing of the emissions from the treated wood have been shown to yield information that is ecologically relevant, however proposals to make such data and risk assessments based on ecotoxicity testing acceptable in dossiers have met with resistance by the EC and competent authorities.

13 OECD and CEN co-operation

It soon became clear to the OECD Expert Group that a key issue that had to be tackled was that of determining a realistic representative emission rate. A ground breaking co-operative exercise was set up between the OECD and CEN with the remit to develop one or more methods that would generate realistic emissions from treated wood and that could be used to determine the PEC for the relevant treated wood scenario. It was intended that these methods would be further developed into OECD Guideline methods. The CEN input was to be provided by CEN TC 38, and a new Working Group Number 27 was established. The WG produced two laboratory scale draft methods that the OECD put out to consultation and requested comment from OECD member countries in 2003. These methods are described in CEN documents.

A relatively small number of OECD countries commented on the draft methods; and these comments were considered by CEN TC 38 WG 27, in October 2003, with the view to replying to the OECD. The comments to be made by TC 38 WG27 were informed by the work being carried out at BAM, in Berlin, looking at the impact of modifications to standard techniques such as EN84. The final report of this work is due in March 2004, but at the time of writing this paper it is understood that the report will probably be delayed.

As well as laboratory scale methods a number of companies and institutes have been experimenting with using panels or palisades to simulate HC3 out of ground contact exposure and with exposure to rainfall and other chemical and physical degradation processes. It is considered that emission rates from HC 3 treated timber should be lower than those for HC4 and 5 uses of the same active ingredient. The results of some of these experiments are being included in the dossiers being prepared for the BPD and are subject to data protection constraints.

14 So what will happen to the draft methods prepared for the OECD?

It seems likely that the two methods will be merged into one, and the differences between HC3 and HC 4 / 5 exposures will be reproduced by differences in the time of immersion in water. If the method(s) is to become an OECD Guideline it will require validation and this could take some time. Similarly if CEN TC38 does further work in this area any proposals or draft standards will require validation and again this will take time.

At the moment industry has no idea whether any data generated according to the draft protocols prepared for the OECD will be accepted and there is already one MS publicly stating that any data generated by the methods is a waste of time and money.

It will no doubt be argued that provided industry can substantiate the emission flux values it uses in the model calculations for each scenario, this will be sufficient for the purposes of reviewing a substance for entry into Annex I. The situation may not be quite the same when it comes to the authorisation of the wood preservative product at the MS level.

So the tools to produce realistic representative emission and emission rates are still lacking.

15 Receiving compartment size

As well as needing to generate a realistic emission, the characteristics of the receiving compartment are also crucial in the determination of the Predicted Environmental Concentration. The models contained in the OECD ESD set out the dimensions of the volume of soil or water into which the emissions from unit areas of treated wood surface are lost.

The original default distance from the edge of the treated timber used to calculate the receiving compartment size was 10cm in the case of soil. However, following a recent workshop carried out at the ECB on training the experts in the MS Competent Authorities to interpret the scenarios contained in the ESD on wood preservatives, it has been decided to introduce a 2.5 cm distance. Clearly if the dimensions of the receiving compartment are small then the concentration of the active substance / emission will be correspondingly higher than a receiving compartment whose dimensions are larger.

During the development of the ESD, the importance of the size of the receiving compartment became an area of serious disagreement between the members of the Expert Group. The Foreword on page 2 of the OECD Series on Emission Scenario Documents No.2, Emission Scenario Document for wood preservatives, contains the following statement: ‘ A fundamental issue considered in the development of the ESD was the size of the receiving environmental compartments. There are no agreed scientific criteria for choosing this and, although there was no unity within the Expert Group, most members agreed to use the values proposed by the Secretariat that appear in the document. These default values are not “ fixed in concrete” and if users of the ESD have other more valid values, then these should be used instead.’

The concept of the receiving compartment is intended to represent a particular environmental compartment so an assessment can be made of the potential for risk to that compartment. It would seem, however, that the sizes of the receiving compartments defined in the ESD are not ecologically relevant. Despite the importance of this issue it seems the EC will wait and see what effect the size of the receiving compartments will have on the PEC values obtained for active substances and substances of concern during the review programme before deciding what to do. This situation is unacceptable.

16 Relevant time periods

Mention has already been made that decisions will be made on the basis of the PEC: PNEC ratio, but which PEC is relevant? So far the guidance is to calculate the PEC values for three time periods, namely 0-30 days, 100 and 3650 days.

The first 30 days is probably intended to relate to the life cycle of some of the species used for ecotoxicity testing such as *Daphnia.* Is it relevant to the other species such as fish and algae?

The first 100 and 3650 days periods have no scientific basis to them, but they are considered to give some idea of what happens either at the site of application of the product, or where the treated timber is installed in service. What they do is to introduce the need to consider the fate and behaviour of the active substance/substance of concern in the receiving compartment over these periods. Some idea of how the substances behave can be gained from the tests required to characterise the substance, such as the octanol -water partition coefficient; transformation processes; bio-availability; migration through the soil etc.

The OECD ESD gives some suggestions on to how to deal with this aspect, mainly relying on mathematical models.

17 Further considerations

It can be seen that an essential aspect of being able to carry out an environmental risk assessment of a wood preservative product is to be able to characterise the emissions from the wood.

Environmental impacts from treated timber are related to emissions of the active substance from the surface of the treated timber into the environment over time. Destructive sampling techniques to determine losses from the treated wood do not yield the data needed.

18 In summary

Much time and effort has been expended by organisations, institutes and industrial companies in evaluating data and experiences over many years concerning wood preservatives and treated wood.

The output of all this collective effort is contained in the draft methods that are now with the OECD. There is a need to recognise that, for the purposes of environmental risk assessment, highly refined testing techniques are not required. Environmental risk assessment is a process of describing and quantifying impact on a relatively large scale. Much time has been lost through the slow process by the regulatory authorities of accepting and validating the techniques that have been developed. Consequently industry finds itself in an impossible situation where any data generated using these draft methods may or may not be acceptable to the Rapporteur Member State, bearing in mind the dossiers for active substances in PT8 are required to be with the RMS by the end of March 2004.

In spite of this, the work and discussions held between organisations, institutes and industry during this COST Action has highlighted a number of key issues that must be dealt with. These require better scientific input to ensure legitimate product authorisation regimes are in place, not only to recognise the environmental optimisation of wood protection, but also the relevance of the key issues in the regulation of all other biocidal product types.

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Working Group 3 : *Innovations*

WORKING GROUP 3 : *INNOVATIONS*

WG 3 has been concerned with innovative wood protection systems which can contribute to the environmental optimisation of wood protection. The main objectives were defined in the MOU as:

- non-toxic treatments
- insect growth regulators
- chemical modification systems
- natural biocidal products and systems
- new chemical wood preservatives

During the Action, particular attention was given to the applicability of new protection systems, including the challenges of scaling up these systems to make them suitable for commercial, industrialised use. In the Final Conference special attention (in a joint workshop with Working Group 1) was given to performance issues of new systems, many of which are not suitable for testing in accordance with standardised procedures for predicting and quantifying likely effectiveness in service. Furthermore, several treatments were described which have the potential to protect the wood without the use of conventional biocides (these included in particular heat treatments and hydrophobic treatments). The presentations in the Final Conference complemented special sessions held earlier during the Action and published in individual Proceedings on Heat treatments and on Oils and Resins.

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Chairman Working Group 3 ‘*Innovations*’

IMPROVEMENTS IN THE BIOLOGICAL DURABILITY OF UK GROWN TIMBERS BY VARIOUS WOOD MODIFICATION TECHNIQUES

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Keywords: wood modification, pine, spruce, durability test

Summary: *The use of wood modification techniques has gained acceptance as a means of improving some of the properties of European timber species. Among the main improvements are increased dimensional stability and natural durability. Whilst results from various European studies may be inferred to UK grown timbers, there have been very few specific tests carried out to date.*

The work used the recommended guidelines for Basidiomycetes testing described in CEN TC38/WG23/N34 for assessment of natural durability using Coniophora puteana and Poria placenta on Scots pine and Sitka spruce for the following treatments:

- *Acetylation*
- *Furfurylation*
- *Heat treatment*
- *Hot oil treatment*

Results are also compared to EN350-1. Results to date indicate that furfurylation and acetylation are capable of improving the durability classification of UK grown Sitka spruce from Not Durable, Durability Class (DC) 5 for control samples to Very Durable (DC1). More interestingly, lower durability classifications were obtained when using UK grown Scots pine, where the treated samples could be regarded as Moderately Durable (DC3) or Slightly durable (DC4). Normally higher levels of modification would be expected for Scots pine compared to Sitka spruce. This may be due to a more effective envelope treatment with the Sitka spruce samples

In addition a brief summary of some other properties tested is given.

1 Foreword

This paper presents preliminary results obtained by BRE for various modification treatments applied to UK-grown timber species.

The results reported in this paper conflict dramatically with those reported in previous research work. Coming from a single experimental test the results presented here cannot be considered definitive. In order to get a definitive picture, more tests would need to be carried out, and compared with the peer-reviewed data reported by institutes worldwide. Wood is a highly heterogeneous material, and variations between material properties of samples from different sources and even from the same tree have been well documented. Until a re-evaluation or additional tests are conducted the results presented here should be considered preliminary.

Work needs to be carried out to ascertain why the results seem to conflict with previous research, to ascertain realistic levels of treatment afforded and also degree of variation that may be expected. It is the intention of the authors to initiate this work in co-operation with international centres of expertise in wood modification. This work will initially be reported at the International Research Group on Wood Preservation 35th conference in June 2004.

2 Introduction

There has been a considerable drive towards sustainability within the timber industry. However such demands are often met with the need to use a species of lower qualities than, for example, a traditional tropical hardwood species. For several decades, there has been an interest in using wood modification as a means of altering the properties of a 'common' softwood species, so that some or its properties mimic those of a tropical hardwood. Whilst early work in this field was carried out in the US and Japan, more recent developments have taken place within Europe.

Within the UK, the Building Research Establishment (BRE) are among a group actively promoting wood modification as a means of improving timber properties. Currently BRE have several projects involving the use of wood modification techniques to upgrade UK grown timber. There are a variety of treatments that may be considered and these are summarised below.

Acetylation

This is recognised as the most common form of chemical modification, having been demonstrated on laboratory scale several decades ago. The process involves the reaction of wood with an acetylating agent (such as acetic anhydride or ketene) resulting in the esterification of many of the hydroxyl groups within the wood. These then become more hydrophobic, making the wood more dimensionally stable. Recent developments have seen attempts at commercialisation in two European countries – The Netherlands (Figure 1) and Sweden (Figure 2).

Figure 1: Acetylation plant in The Netherlands

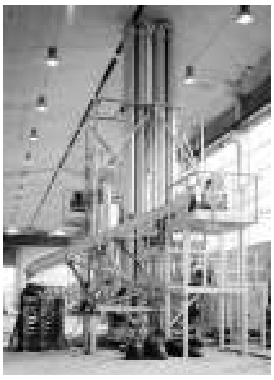

Figure 2: Acetylation plant in Sweden

More recently, considerable financial investment has been placed into acetylation within The Netherlands, with the establishment of a new company, TitanWood.

Furfurylaytion

A process that has emerged on the market place is wood treated with furfuryl alcohol, a compound that may be produced in biomass conversion. This reagent can react with the wood hydroxyl groups as well as undergoing polymerization reactions, resulting in polymeric blocking of the hydroxyl groups. Development of this process has been done in Norway. It has been shown that furfurylation can prevent decay, increase dimensional stability and decrease hygroscopicity among other properties. Figure 3 shows the pilot scale treatment vessel, and full commercialisation has now been established at Porsgrunn in Norway.

Figure 3: Pilot scale furfurylation plant

Heat treatment

There are a variety of companies currently working on thermal modification of wood. Heat treatments are based on the limited thermal cleavage of active groups within the wood structure, which may be released as volatile agents, or trapped within the cell matrix and repolymerized into more stable forms. Treatments are usually carried out in the absence of oxygen, due to the excessive thermal degradation processes that can occur in the presence of oxygen. Thus, atmospheres of nitrogen, steam or treatment *in vacuo* are used. Since this, in theory, is a simpler process than chemical modification, plant construction costs are generally lower, resulting in a slightly lower priced product. Companies working on heat treatments include New Option Wood of France, Plato Wood of The Netherlands, whilst the largest company is a Finnish consortium, which produces ThermoWood®.

Hot Oil treatment

A treatment that is increasing in popularity is the use of a reactive hot oil. BRE are currently working with SHR, Bangor University and several industrial partners on a Fifth Framework CRAFT project for the development of a commercial process using a modified linseed oil system. Other hot oil treatments have been developed in the past, most notably the Menz Holz system in Germany.

3 Method

3.1 Selection of treatment

The majority of work carried out to date has been with Sitka spruce. The following modification processes were used in study, though it should be noted that none were optimised for the timber species under test. Thus, this work represents the first stage in an evaluation process.

The treatments considered were:
Acetylation
Furfurylation
Thermal treatment (New Option Wood)

A range of batches of Scots pine and Sitka spruce samples were treated for these trials. The prepared material was sent for the following treatments:

- Acetylation – to SHR, The Netherlands
- Furfurylation – to WPT, Norway
- Heat treated – to New Option Wood, France
- Hot oil treated – to SHR, The Netherlands

3.2 Timber used

For all results within this paper, either Scots pine (*Pinus sylvestris*) or Sitka spruce (*Picea abies* L. karst) sapwood has been used. Dimensions, unless otherwise stated, relate to dimensions quoted within the standard tests used.

3.3 Durability testing

In order to ascertain improvements in the durability of the treated timbers, it was necessary to run experiments following descriptions laid out within CEN/TC38/WG23 N34 (2003). This is a working document intended as a replacement standard for assessing the natural durability of timber species. Such documents have been drafted due to the need to assess materials such as modified wood in ways different to conventionally preservative treated wood.

Two different sample sizes were used during these experiments. The majority were carried out using standard EN113 (1997) block sizes (50x25x15 mm), though some experiments used blocks of slightly smaller dimension (50x20x10mm).

4 Results and Discussion

4.1 Biological durability tests

The following section shows results achieved to date for UK grown timbers that have been modified by various processes. Results have been derived using CEN/TC38/WG23 document N34. In some cases there is a comparison of these results to EN350-1: 1994.

4.1.1 Durability testing of acetylated, furfurylated and heat treated samples

Samples were treated as listed in 2.1 and the levels of modification measured by weight percent gain (WPG) following treatment is shown in Table 1.

Table 1: Overview of weight changes following modification

Treatment	Weight Percent Gain (WPG) Sitka Spruce used in biological test for named fungal species		Weight Percent Gain (WPG) Scots Pine used in biological test for named fungal species	
	C. puteana	*P. placenta*	*C. puteana*	*P. placenta*
Acetylation	19.6	19.6	17.8	16.2
Furfurylation	35.5	34.6	22.5	22.5

Samples used to test heat treatment had no weight loss data (as thermal treatments result in an overall weight reduction), so these could only be used as a qualitative evaluation. An interesting observation from Table 1 is that there is a higher WPG for Sitka spruce than Scots pine. This goes against conventional thinking, whereby the pit closures of Sitka spruce usually result in a difficulty to treat. Similar trends in weight gains were noted for samples acetylated for EN252 testing, where the ease of acetylation appeared to be:

Douglas fir > Sitka spruce > Larch > Corsican pine > Scots pine.

It is well known that UK Sitka spruce is a fairly fast grown timber species. Any suggestions that this higher than expected weight uptake may be due to this fast growth seem to be dispelled by evidence from another BRE project where there appears to be very little variation in preservative uptake between fast and slow grown timber when impregnated using the same solution and treatment cycle.

Tables 2 and 3 give summaries of the durability data gathered for the Scots pine and Sitka spruce test blocks respectively.

Table 2: Summary of biological test data according to document N34 for Scots pine samples

Treatment	Mass Loss Tested According to CEN/TC38/WG23/N34		Durability class	
	C. puteana	*P. placenta*	*C. puteana*	*P. placenta*
Acetylation	20.2	16.1	4	4
Furfurylation	11.1	13.4	3	3
Heat treated	18.6	18.1	4	4
Control	38.7	22.2	5	4

Table 3: Summary of biological test data according to document N34 for Sitka spruce samples

Treatment	Mass Loss Tested According to CEN/TC38/WG23/N34		Durability class	
	C. puteana	*P. placenta*	*C. puteana*	*P. placenta*
Acetylation	<0.02	<0.02	1	1
Furfurylation	5.6	1.3	2	1
Control	53.2	33.9	5	5

It can be seen that there is a considerable improvement in the durability of Sitka spruce samples. This may be related to the higher weight gains already noted, and the recognised fact that treatments with organic anhydrides (such as acetic anhydride) require WPG's of around 20% to establish high levels of protection against decay fungi (Goldstein *et al* 1961). More recent work (Beckers *et al* 1994) suggested that acetylation levels for Scots pine to 18% WPG were required for effective protection against *Coniophora puteana* and over 20% WPG against *Poria placenta*. An overview of some other trends for different timber species has also been published (Papadopoulos and Hill, 2002).

Document N34 of CEN/TC38/WG23 calculates the durability class of a timber on the mass loss for a given test. This differs from other natural durability standards, such as EN350-1 (1994), where the classification is derived from the ratio of mass loss between the tested material and mass loss of control material. In order to see if there were any differences in results obtained, calculations were carried out according to EN350-1 (Tables 4 and 5 respectively).

Table 4: Summary of biological test data according to EN350-1 (1994) for Scots pine samples

Treatment	Mass Loss Ratio '*x*' According to EN350-1 (1994)		Durability class	
	C. puteana	*P. placenta*	*C. puteana*	*P. placenta*
Acetylation	0.52	0.72	3	4
Furfurylation	0.28	0.60	2	4
Heat treated	0.48	0.82	3	4

Table 5: Summary of biological test data according to EN350-1 (1994) for Sitka spruce samples

Treatment	Mass Loss Ratio '*x*' According to EN350-1 (1994)		Durability class	
	C. puteana	*P. placenta*	*C. puteana*	*P. placenta*
Acetylation	<0.02	<0.02	1	1
Furfurylation	0.14	0.06	1	1

From Tables 4 and 5, it can be seen that Scots pine has a durability classification of 4 (slightly durable) when acetylated, furfurylated or heat treated, whilst Sitka spruce has a durability of 1 (very durable). It would appear that for both acetylation and furfurylation, there has been insufficient weight percent gain (an indication of the level of modification) to achieve a successful treatment. Observations by SHR suggest that the acetyl content within tested samples of Sitka spruce and Scots pine are fairly similar, whilst there is a higher acetyl content for the surface of Sitka spruce (19.6%) compared to Scots pine (16.4%). Thus, there may be a more effective envelope treatment protecting the Sitka spruce.

4.1.2 Durability testing of hot oil treated Sitka spruce

Four different batch treatments were used for the preparation of Sitka spruce used in these trials. Half the samples of each batch were impregnated prior to treatment with the hot oil. The averaged weights and weight gains achieved are summarised in Table 6.

Table 6: Weight percent gains of samples treated

Batch No.	Impregnated*	WPG after impregnation	WPG after curing
6	Yes	N/A	87.6
6	Yes	N/A	47.3
TG2	Yes	27.6	27.3
TG2	No	N/A	26.2
TG3	Yes	27.5	29.3
TG3	No	N/A	27.3
TG4	Yes	26.3	51.9
TG4	No	N/A	53.4

* samples impregnated in reactive oil prior to reaction in hot reactive oil

The four batches used had differing processes, and may be summarised as follows:
Batch 6 – using reactive oil (UZA) throughout
TG2 Linseed oil is being used as heating medium instead of UZA.
TG3 – UZA used as heating medium
TG4 - Pressure applied during the heating phase. Approx. 10 bar for 1 hour. UZA used as the heating medium.

Batch 6 was split into two sections due to a distinct difference in weight percent gains of samples. This may have been due to the positioning of the samples in the reaction vessel, with some blocks (the lower weight gains) not being completely immersed in the reactive oil (for example, within the foam at the top of the vessel).

The natural durability tests ran for the required period of 16 weeks. The results obtained (Table 7) are an indication of the improvement in the natural durability. There is a considerable improvement in the natural durability of Sitka spruce compared to the Scots pine controls following hot oil treatment. Concurrent work showed that untreated Sitka spruce would also be classified as durability class 5, with weight losses in the region of 50% and 30% for Coniophora puteana and Poria placenta respectively.

Table 7: Summary of biological test data according to document N34 (2003) for hot oil treated Sitka spruce.

Batch number	Impregnated	Fungal species	% weight loss	Durability class
Scots pine: None – control	No	C. puteana	35.8	5
Scots pine: None – control	No	P. placenta	22.1	4
Sitka spruce: None – control	No	C. puteana	53.2	5
Sitka spruce: None – control	No	P. placenta	33.2	5
6	Yes	C. puteana	<1.0	1
6	No	P. placenta	1.6	1
TG2	Yes	C. puteana	8.4	2
TG2	No	C. puteana	17.7	4
TG2	Yes	P. placenta	8.5	2
TG2	No	P. placenta	11.0	3
TG3	Yes	C. puteana	4.2	1
TG3	No	P. placenta	8.0	2
TG4	Yes	C. puteana	2.4	1
TG4	No	P. placenta	6.6	2

Whilst further testing is required to demonstrate the comparison between Poria placenta and Coniophora puteana for individual batch treatments, there is a definite improvement of natural durability of at least 3 classifications. It is not possible to suggest which fungal strain will be the more aggressive towards the hot oil treated samples, as each fungus has the potential of behaving differently to a given substrate. However, results for a ‘conventional’ treatment (pre-impregnation, followed by vacuum process, then heating in reactive oil with a pressurised step at the end of the heating cycle) allows Sitka spruce to achieve durability class 1. In some cases, only one fungal species has been used (for example the differentiation of impregnation and non-impregnation prior to curing indicated by TG3 and TG4 in Table 7). Since it is uncertain whether that particular fungal species produces the most aggressive decay for the hot-oil treated wood, it is uncertain if the quoted durability rating is the correct classification (i.e. durability standards indicate the lowest durability rating, suggesting a ‘worst case’ scenario). Thus, these results, whilst giving an insight into the improvement of the natural durability, cannot yet be viewed as definitive results as to the benefits of this treatment.

For the high weight gains samples within batch 6 (where the average WPG was 87.6%), it was noted that the blocks have leached what appears to be some of the reactive oil. This is interesting since this should be polymerised and fixed within the wood structure. This ‘leachate’ may be due to:

The breakdown of the polymerised material in a humid / wet environment
The breakdown of the polymerised material as a result of fungal attack
Material that had not been sufficiently polymerised

For all samples tested to date, the polymerised oil coating was left on the surface of the test blocks. This might create an artificial barrier to fungal decay, and may not be present in treated wood used in actual products. This may be due to secondary profiling of products following treatment removing the polymerised surface on the wood. In order to see if this is the case, a series of tests will determine if there is a difference between tests blocks that have had this polymerised coat removed (by sanding).

Work will continue on testing material treated by various impregnation and curing processes, both for Sitka spruce and Scots pine.

4.2 Other properties

To date, whilst there appear to be trends in the properties of acetylated, furfurylated and heat treated timbers, an insufficient number of replicates have been tested to draw any significant conclusions. There has been more work carried out with hot oil treated Sitka spruce.

4.2.1 Mechanical properties

Following on from preliminary tests, batches of modified Sitka spruce samples were tested to determine their modulus of elasticity and modulus of rupture. In order to achieve comparable results, matched samples were used, of dimension 16 * 23 * 250 mm with an untreated reference sample matched to each treated sample. These dimensions were selected on the maximum dimensions available from samples.

In total 4 sets were produced: A, B, C and D. Set A matched to the treated matched sample of C and set B the treated matched sample of D. Tests were carried out using a 3-point bending test system. Table 8 shows the average values achieved.

Table 8: Average mechanical data achieved for matched treated and untreated Sitka spruce samples

Sitka spruce samples	Modulus of elasticity E (N/mm^2)	Modulus of rupture f (N/mm^2)
Hot oil treated	7700	49
Untreated	7300	62

From these results we can see that the treatment has resulted in a moderate increase in stiffness of around 5% which could be contributed to natural variability within the sets of timber samples. At the same time there is a reduction in strength of around 20%, so some damage has been incurred by the samples. However, for UK grown Sitka spruce the strength tends to be higher than required by the strength class. The limiting factors tend to be stiffness and density, which are not negatively effected.

4.2.2 Hardness testing

A series of matched samples were prepared from the undamaged sections following the 3-point bending testing and the hardness determined. The results are shown in Table 9.

Table 9: Average hardness data for matched Sitka spruce samples

Sitka spruce samples	Hardness in radial direction (N)	Hardness in tangential direction (N)	Average hardness (N)
Treated	1910	1660	1785
Untreated	2100	2000	2050

It can be seen from Table 3 that there is a slight decrease in the average hardness of the surface of treated samples (a 9% reduction in the radial direction and a 17% reduction in the tangential direction.

4.2.3 Assessing Dimensional Stability (Water soak tests)

The aim of undertaking a water soak test is to determine if dimensional stability has been enhanced. Average results for matched samples (derived from undamaged sections of samples from mechanical testing outlined earlier) are shown in Table 10. These are shown for the radial and tangential directions respectively. The Anti-Shrink Efficiency (ASE) is more usually expressed as a percentage.

Measurements for the full cycle of wetting and drying are ongoing for this test, but the initial results are shown in Table 10.

Table 10: Summary of initial coefficients of swelling and anti-shrink efficiency for modified Sitka spruce.

Grain orientation	S (%) treated	S (%) untreated	ASE (%)
Radial	1.7	4.0	56
Tangential	2.7	8.1	67

The preliminary results shown in Table 10 clearly show that the hot oil treatment has produced a far more dimensionally stable material than the original Sitka spruce. Good dimensional stability (low movement) is an essential property for higher value added applications of timber, so these results are very promising.

5 Conclusions

From work carried out to date, it would appear that:
Work has been carried out using acetylation, furfurylation, thermal treatment and reaction with hot oils.
Dramatic improvements in the durability of Sitka spruce may be achieved through the use of wood modification techniques.
Moderate improvements in improvements have been noted for Scots pine.
Field trails according to EN252 are also ongoing.
The reason for this reversal in expected behavior is uncertain.
It would appear to be linked to the level of treatment on the surface of the test blocks (in effect an envelope treatment).
Reaction with hot oils appears to have limited effect on other properties of Sitka spruce (e.g. mechanical properties).

Work will continue:
To further evaluate the modification of UK-grown timbers, with the preparation of demonstration products placed and tested under service conditions.
Establish the suitability of modification systems for different timber species.
Continue to evaluate the durability of modified timber.

6 Acknowledgements

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INFLUENCE OF UP-SCALING PROCESSES ON DEGREE AND GRADIENT OF ACETYLATION IN SPRUCE AND BEECH

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Keywords: Up-scaling, acetylation, spruce, beech, treatability

Abstract*: Acetylation at laboratory scale is a well-known and relatively easy chemical process. In up-scaling the process, the dimension of the wood and the wood species play an important role. Since with acetylation the wood swells, sharp gradients in acetyl contents in the wood should be minimised in order to prevent huge internal tension resulting in cracking.*

In the MP-3 reactor (2500 l, 4 m length) pilot plant, experiments have been conducted with spruce and beech of different sizes (length and thickness). By chemical analysis (saponification followed by HPLC) the acetyl content at several depths and distances from the cross cut sides has been determined.

The results show that process conditions and the wood species have a clear influence on the acetyl content gradients to be expected in the wood.

In order to obtain acceptable products of acetylated wood, it is important to be able to control the process conditions in such a way that gradients are limited. This is valid for both the impregnation and for reaction conditions. Though very different in treatability, both a refractory species like spruce and a very open species like beech, require well-defined and controlled processes.

1 Introduction

The acetylation of wood with uncatalysed acetic anhydride has been studied extensively and shown to be one of the most promising methods for improvement of the technical properties of wood products. The treatment has shown to result in a very durable, dimensionally stable and UV-resistant material with all mechanical properties of the untreated wood maintained. (Beckers *et al.*, 1998; Beckers and Militz, 1994; Beckers *et al.,* 1994; Goldstein *et al.,* 1961; Larsson and Simonson, 1994; Larsson and Tillmann, 1989; Larsson-Brelid *et al*., 2000; Militz, 1991; Rowell *et al.,* 1989; Singh *et al.,* 1992).

In order to be acetylated (see reaction equation figure 1), the cell wall material needs to be in intimate contact with the acetylation agent (acetic anhydride). The crucial part of the acetylation process therefore is the impregnation step before acetylation (figure 2). The penetration of liquid into the wood is a process that plays at three different levels. At the first level it is important to get the liquid from the wood surface deeper into the wood. At this level the cell lumina are filled. Also at this level, the treatability of wood comes to expression. In species that close their cell structure, like spruce, liquid penetration is difficult. The second level is the penetration of the liquid from the cell lumina into the cell wall (ultra structural level). Even within the same genus the width of cell wall cavities may differ. This may influence the maximum molecule size that can enter the cell wall. At the third level the penetration of the molecules between the cell wall polymers takes place (chemical level). The size and nature of the molecules are a key factor at this level.

At laboratory scale impregnation with acetic anhydride is a well-known and relatively easy process. Complete impregnation can easily be reached in small samples. In up-scaling, the process, the dimension of the wood and the wood species play an important role. Sharp gradients in acetyl contents in the wood can be a result of poor penetration. Since with acetylation the wood swells, this can result in internal tension and cracking. Treatability of a wood species, as well as the acetylation process are critical. Refractory (difficult to impregnate) wood species, like spruce and Douglas fir, are therefore in general not suited for acetylation where large dimensions are considered. Methods to improve the impregnability of a wood species, such as fungal pre-treatment, oscillating pressure methods and incising (Messner et al., 2003), could be a solution to upgrade refractory wood species and allow them to be treated by acetylation. Treatment with gas instead of liquid can be an other, since the diffusion of gases through cell wall material is an order of magnitude faster than that of most liquids. The treatability classification of wood species, as given in the EN 350-2, is mainly based on impregnation of waterborne systems or on creosote oils. In the case of acetylation, acetic anhydride is used, which behaves different compared to water. But even so, there is a clear need to improve the penetration of the liquid in refractory species.

As part of a research program on how to improve the treatability of wood with respect to acetic anhydride, it was needed to gain a good impression on the normal permeability of beech and Norway spruce for liquid anhydride. Therefore boards various thicknesses and lengths of Norway spruce and beech were treated in a number of not optimised acetylation processes.

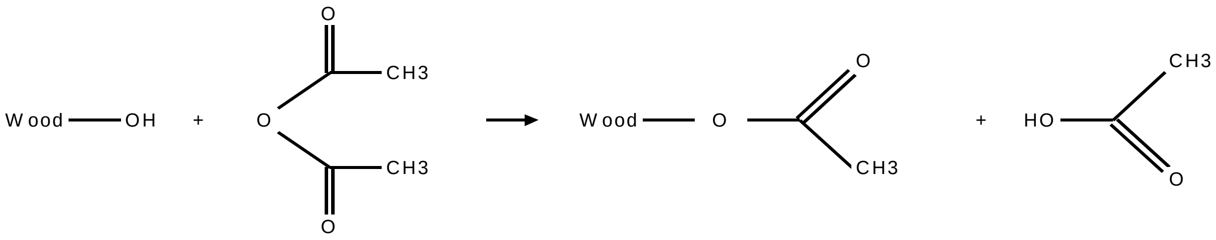

Figure 1. The reaction of wood with acetic anhydride

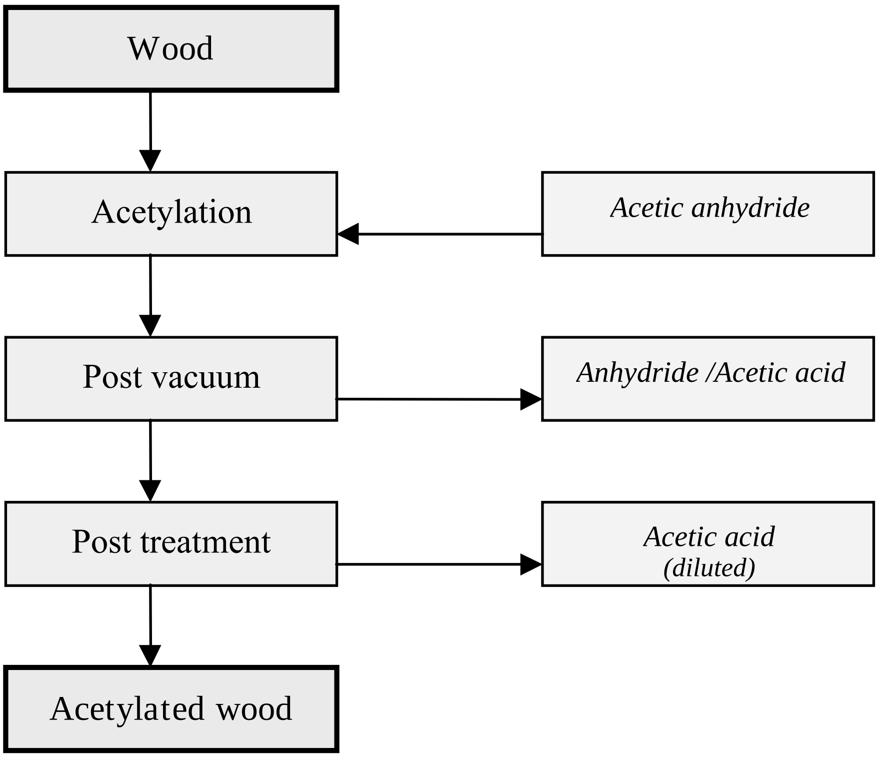

Figure 2. Steps in the acetylation process

2 Materials and Methods

Boards of Norway spruce (*Picea abies* (L.) Karst) and beech (*Fagus sylvatica* L.) of different sizes (length and thickness) were acetylated in the MP-3 reactor (2500 l, 4 m length) pilot plant. In order to get good comparison data the impregnation process for this research was kept constant. It consisted of a period of 30 minutes vacuum and a period of 15 minutes of 1,0 MPa pressure. This is, on purpose, not an optimised system. In the first batch samples with various thicknesses have been acetylated (see table 1), and in the second batch the length as well as the thickness of the samples was varied.

Table 1. Batch 1 - Norway spruce and beech (width 120 mm, length 1.2 m) of various thicknesses.

Wood species	Thickness (mm)											
	3	5	7	9	11	13	15	20	25	30	40	50
Norway spruce	X	x	x	x	x	x	X		x[1]			
Beech	X	x	x	x	x	x	X	x	x	x	x	x

[1] The 25-mm board of Norway spruce had the radial side on the largest plane, the other samples had the tangential side on the largest plane.

Table 2. Batch 2 – Norway spruce and beech of various lengths and thicknesses.

Wood species	Thickness (mm)	Length (mm)
Norway spruce	27	500, 1500
	40	500, 1500
Beech	27	500, 1500
	60	500, 1500

After the impregnation, acetylation reaction, post-vacuum and post-treatment (see figure 2), samples were taken of the boards. The samples have been obtained by using a top router. The shavings were collected in plastic sample bags and then further processed for the analyses. At least two boards per category were sampled in this way and analysed. The analysis method consists of a saponification step followed by High Performance Liquid Chromatography (HPLC) conform SHR Internal Standard Operation Procedure WVS_SHR_056-EN. The method has been partially published (Beckers et al., 2003). The samples of batch 1 have been taken at various depths in the middle of the board's length. The samples of batch 2 (length experiment) have been analysed over a length gradient by analysing the core of the sample. In this way the gradient in acetyl content can be determined both in the direction of the height of the board and in the length of the board.

From the acetyl contents and the depths of the samples gradients have been calculated. Both the acetyl content as a function of depth and the gradient (i.e. the difference in acetyl content per unit of length) as a function of depth have been presented in graphs.

3 Results

The acetyl content in the middle of the sample's length at various depths is graphical expressed in figures 3 and 4 for respectively beech and Norway spruce acetylated in batch 1. The values represent the average of two boards per category. In figures 5 and 6 the results of batch 2 are plotted. The length of the board is set along the X-axis. A clear effect of the longitudinal penetration can been seen for spruce, and especially in the 40 mm thick samples.

The penetration in depth of acetic anhydride in Norway spruce during the first batch is lower than that in beech. Under the used process conditions, a steep gradient in acetyl content (in depth) is observed for Norway spruce dimensions of 13 mm and up, while sharper gradients in depth are starting at 30 mm for beech.

In the graphs (figure 7 and 8) the acetyl content as a function of depth are represented respectively for beech and for spruce. In the last graphs (figure 9 and 10) the gradient (in -% / mm) is plotted against the depth of the board.

In figures 4 and 10 the effect of sawing patron is visible in the 25 mm board of Norway spruce, which was quarter sawn while the other samples are plain sawn.

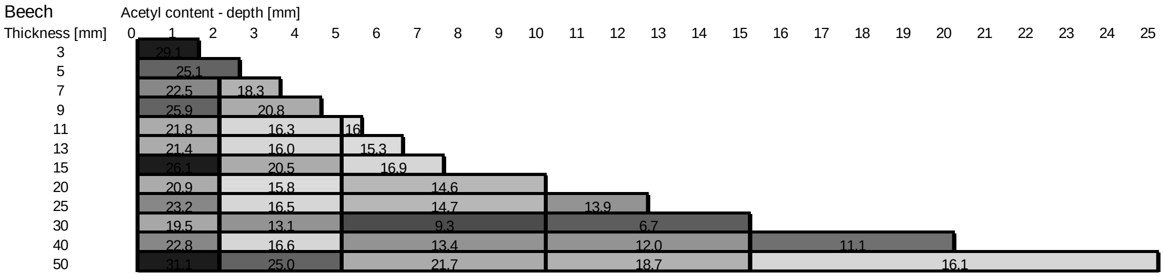

Figure 3. The reached acetylation content in depth for various wood dimensions of beech acetylated within one batch.

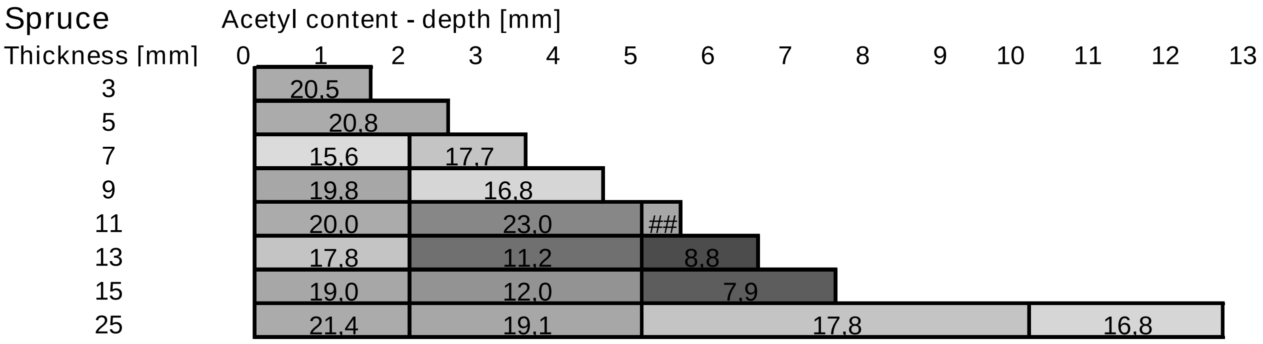

Figure 4. The reached acetylation content in depth for various wood dimensions (up to 25 mm) of Norway spruce acetylated within one batch.

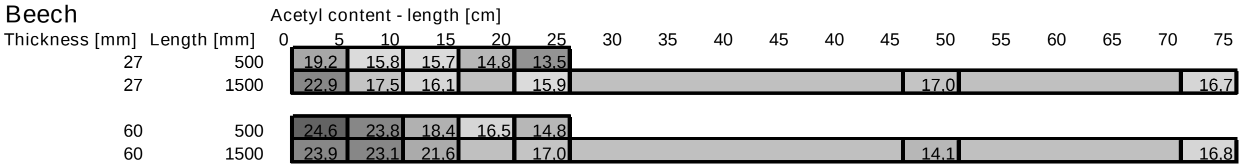

Figure 5. The reached acetylation content in length for 27 and 60-mm thick and 500 and 1500 mm long beech acetylated within one batch.

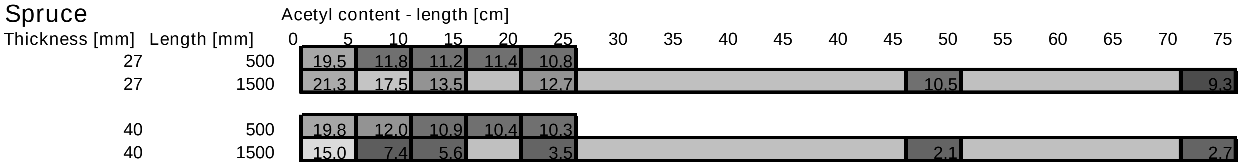

Figure 6. The reached acetylation content in length for 27 and 40-mm thick and 500 and 1500 mm long Norway spruce acetylated within one batch.

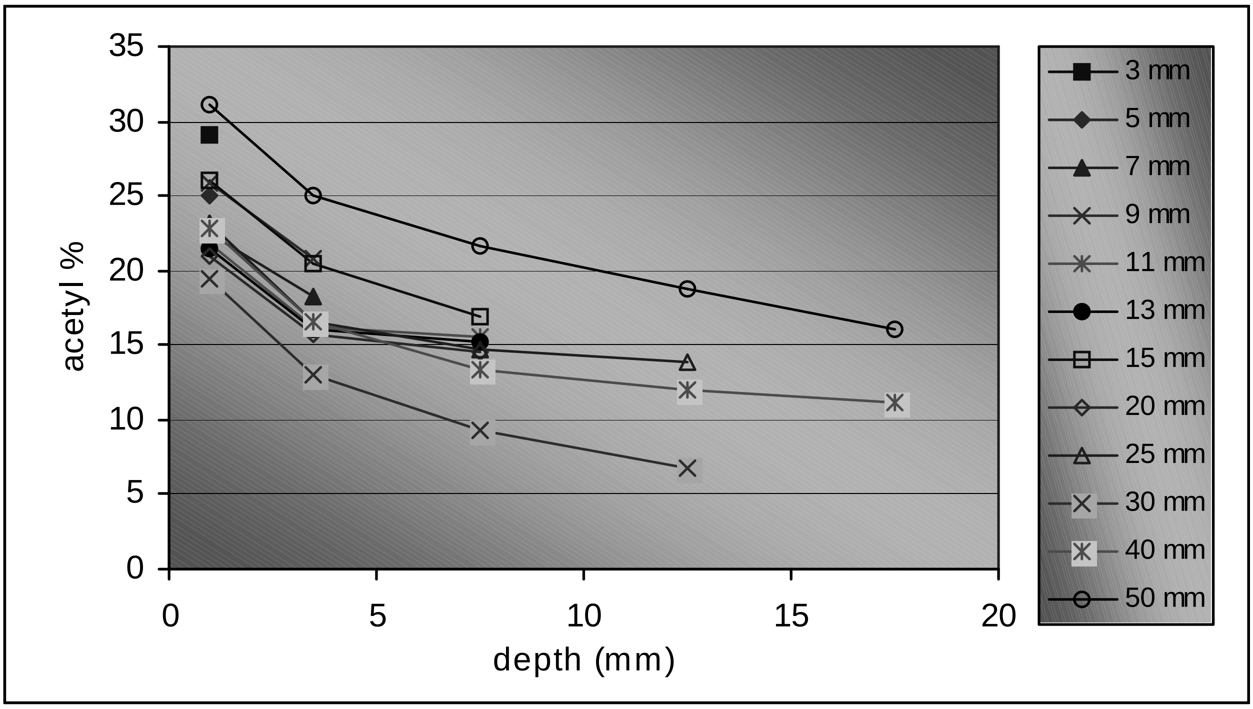

Figure 7. Acetyl gradient in beech

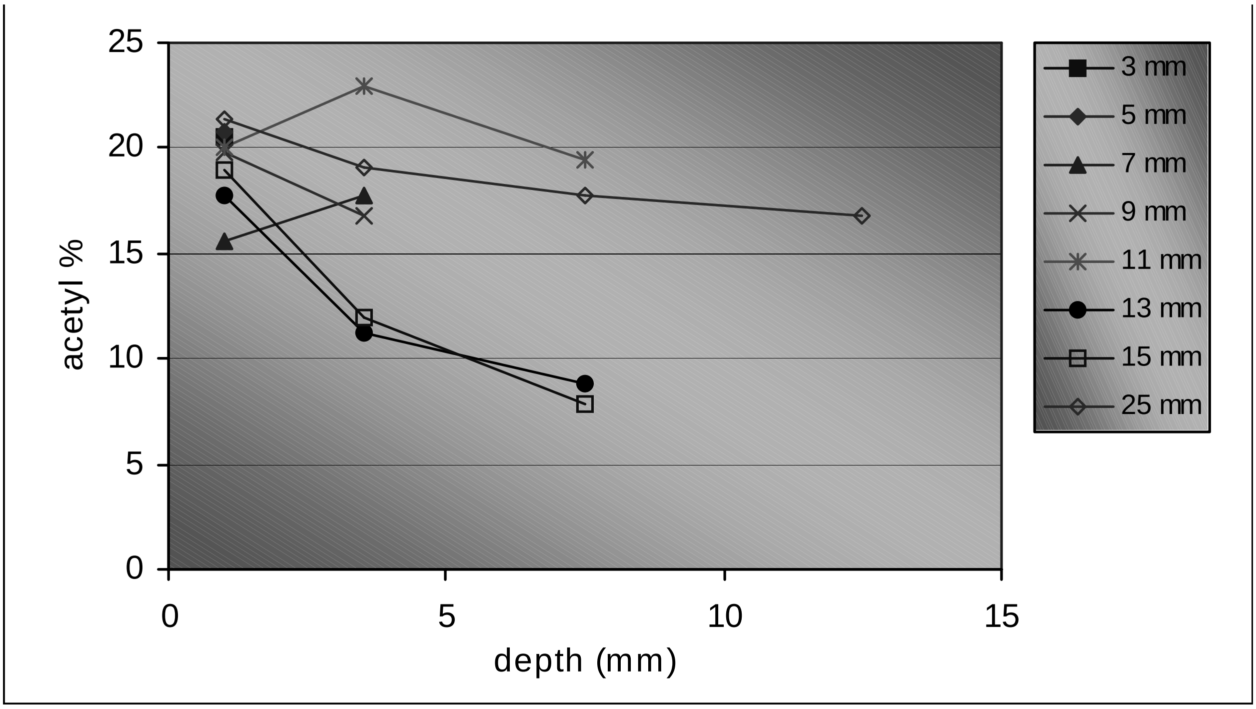

Figure 8. Acetyl gradient in spruce

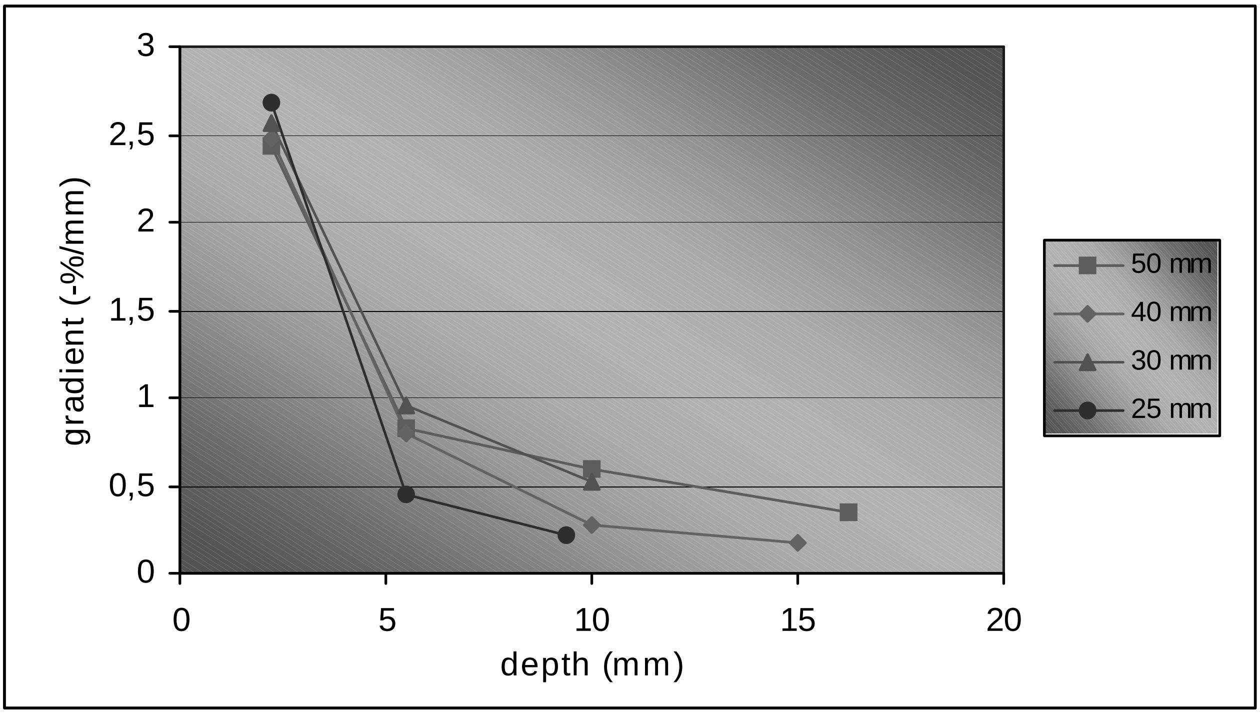

Figure 9. Acetyl gradient as a function of depth in beech

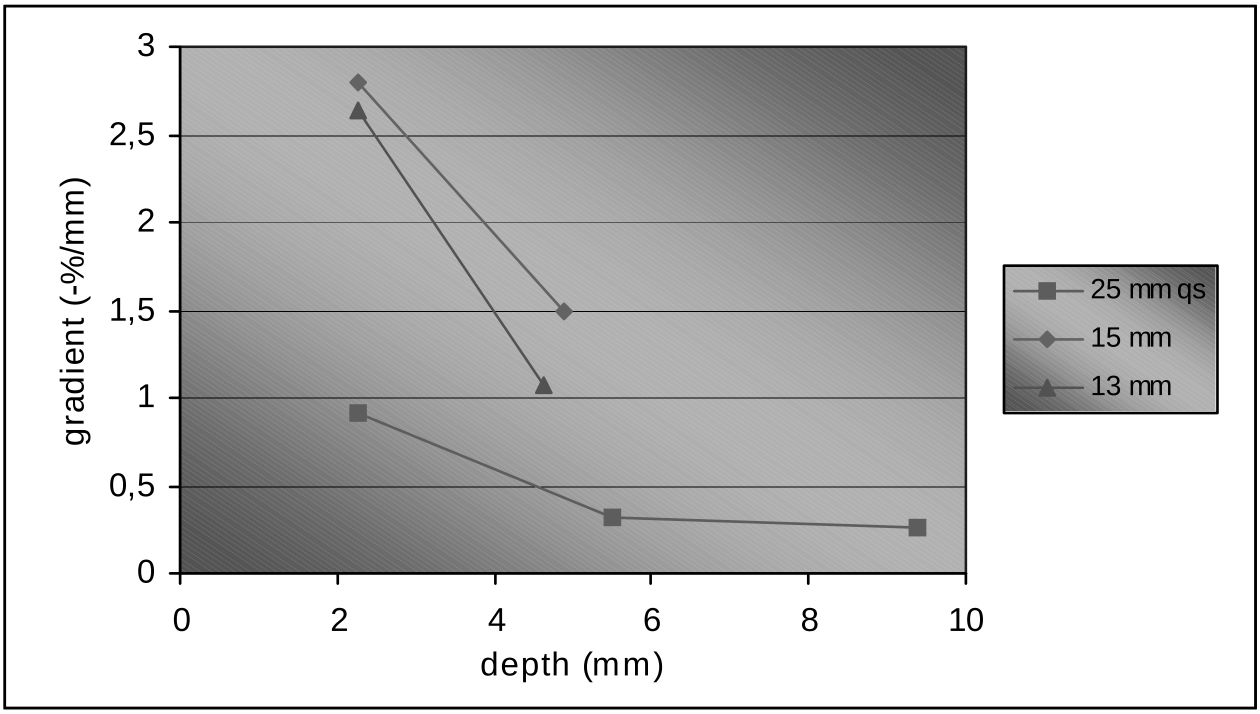

Figure 10. Acetyl gradient as a function of depth in spruce. Remark: The 25 mm sample is quarter sawn.

4 Discussion

Known differences in treatability of wood species, related to liquids, also reflect in liquid acetic anhydride impregnation. According to EN 350-2 the treatability of Norway spruce is classified as 3-4 (sapwood 3 variable, 3-4 heartwood), while beech heart- and sapwood are classified as 1 (4 if red-heart is present). Treatability class 1 gives good penetration of the anhydride, treatability class 4 poor. Liquid acetic anhydride seems to penetrate deeper into wood than water as can be seen from the analysis results in spruce. Here even in the core increased acetyl contents are found, whereas waterborne systems in spruce usually result in an envelope pattern.

The main difference with water borne systems is that, probably during the reaction step, acetic anhydride penetrates the cell walls as gas and migrates deeper into the wood structure than the liquid. Even with temperatures below the boiling point of acetic anhydride this occurs. Locally in the wood, because of the exothermic reaction of the hydroxyl groups with the anhydride and the reaction of water with anhydride (also exothermic), the temperature is likely to be well above the boiling point. This means that locally the pressure of the anhydride gasses, combined with that of the acetic acid that is formed, can be raised and that the anhydride can penetrate the cell walls and adjacent cells as a consequence. There it will react easily with the available hydroxyl groups in the wood, thus, because of the exothermic reaction, even increasing the temperature. Because of the difficult transport of heat through wood this may lead to internal burning if not well controlled. At those places where liquid is present in the wood the heat can be lead away relatively easily. However in the core of the wood where the liquid has not penetrated these burning effects can be seen, if the process is not well adapted to control this phenomenon.

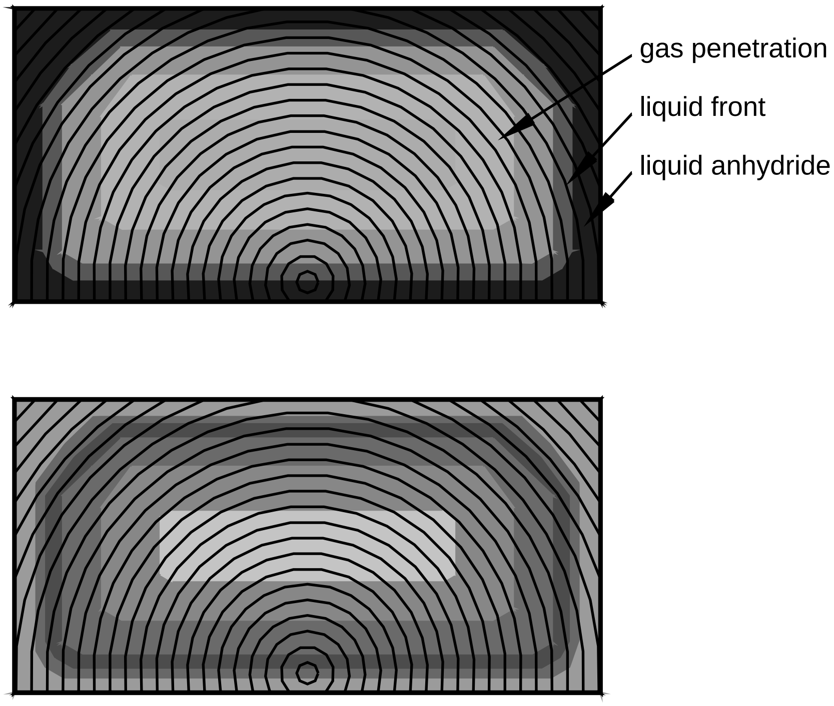

Figure 11. Liquid and gas penetration (upper drawing) and thermal profile (lower drawing) during reaction phase

A typical liquid front situation, as known in envelope treatments, does not appear in acetylated wood. Even in a refractory species like Norway spruce the acetyl content and gradient is not very steep, albeit that in the outer mm’s the gradient is the highest. In a more open species like beech the highest concentrations are still to be found in the outer layers, but even so, the gradient is rather smooth. Consequently, it is very unlikely that tensions in the wood, caused by the differences in swelling, will be raised to such an extent, that cracks or distortions will occur.

However, the acetyl content in the deeper layers of the spruce is still not at a desirable level. This means that the chosen process conditions are not ideal. Experience with long processes learns that even in spruce of bigger dimensions the wanted acetyl content can be obtained. In order to limit the process times and thus making the processes more economical, acceleration of these processes is wanted. Further optimisation of the acetyl processes is still going on. The focus is on gas diffusion and on methods to improve the accessibility. An other way to overcome treatability problems, which can be easily implemented, is to treat wood of small dimensions only and use all sorts of wood engineering techniques to obtain the desired products. For instance, to date it is already very easy and economically viable to make real exterior plywood. The strongly reduced swelling and shrinkage behaviour of such a product allow leaving away dilations. With a board like that, using a transparent coating, the attractiveness of wood really comes to expression.

5 Conclusion

The results show that process conditions and the wood species have a clear influence on the acetyl content gradients to be expected in the wood. The treatability classification of wood species as given in EN 350-2 does not apply when systems other than strictly liquid penetrations are considered. Even the liquid front with acetylation penetrates deeper than the waterfront with waterborne systems. In spruce the effects of the cross-cut sides is limited to the outer 10 cm. In general it is very complicated to separate the cross-cut side effect and the lateral effect. Especially with the more open wood species this is a research methodology challenge. With acetylation there is evidence that at least part of the penetration takes place in the gas phase.

When scaling up processes in wood modification, it is important to be able to control the process conditions in such a way that gradients are limited. This is valid for both the impregnation and for reaction conditions. Though very different in treatability, both a refractory species like spruce and a very open species like beech, require well-defined and controlled processes. The steering of the process conditions in order to control and limit the temperature in the wood, is the most important challenge here.

6 Acknowledgement

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WOOD HYDROPHOBICATION BY ACETYLATION AND OIL TREATMENT

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Keywords: Wood, acetylation, fatty acid ester

Summary: *Enhancement of hydrophobic properties of acetylated wood with plant oil or plant oil ethyl esters was investigated. Pine (Pinus Sylvestris) heartwood samples were acetylated and then impregnated with rape oil (R) and rape oil fatty acid ethyl esters (REE) at elevated temperature. Water vapour absorption dynamics and liquid water up-take were used to estimate hydrophobic properties of treated wood. Wood acetylation increases wood treat ability with non-polar natural oil or it ethyl ester approximately 2 – 3 times. The main impact in reduction of wood swelling had wood acetylation, but in respect of water absorption – impregnation of acetylated wood with seed-oil or its ester. Impregnation with vegetable oil or ester without acetylation had less effect on reduction of water absorption and swelling of wood.*

1 Introduction

Durability of wood can be considerably improved, when hydrophilic hydroxyl groups of wood components are converted in the larger and more hydrophobic functional groups to provide wood to be in permanently swelled condition that decreases wood's disposition to change dimensions. Customary acetic anhydride is used to acetylate hydroxyl groups of wood (Kumar, 1994).

$$Wood-OH+CH_3CO-O-CO-CH_3 \rightarrow Wood-O-COCH_3+CH_3COOH \qquad (1)$$

Where: *Wood-OH* represents wood' reactive hydroxyl group; $CH_3CO\text{-}O\text{-}COCH_3$ is acetic acid anhydride; $Wood\text{-}O\text{-}COCH_3$ represents acetylated wood; CH_3COOH is reaction by-product – acetic acid.
Acetic anhydride substitute hydrogen atom in hydroxyl group of wood with an acetyl group that makes wood less polar and reduce possibility of interaction of modified wood with water and consequently wood swelling. The hydroxyl group acetylation will increase acetylated wood interaction with less polar natural oils that will ensure better maintenance of paints and varnishes. Reduction of wood swelling and shrinking, coatings durability in changing humidity is very essential for wood manufactures such indoors building constructions as window frames or doors.

Solid wood acetylation with acetic acid anhydride and other reagents were studied in Latvia University of Agriculture since fall of the sixties. Different wood species reactivity with varied reagents has been studied. Magnesium perchlorate catalyst, dichloromethane vapours and high frequency current were used to increase the acetylation degree of wood. Laboratory trials resulted in designing, making up, and putting in action pilot equipment of solid wood acetylation with acetic anhydride in liquid phase in the middle of the seventies. Different trials with various reagents and solvents were performed to fix or to extract residual acetic acid from solid acetylated wood (Morozovs etc., 2003).

Carbonyl groups' oxygen atom increased π-electrons density in acetyl group creates possibility to form dipole-dipole interaction with acetylation by product acetic acid. Such acetic acid chemical binding in acetylated wood persecutes acetic acid removal at the end phase of acetylation process. Acetic acid strong chemical absorption in acetylated wood and permanent slow hydrolysis of acetyl groups with absorbed water during service time creates lack of acetylated wood - smell of acetic acid that can not be eliminated by heating or any other physical impact.

Wood cell wall substance interaction with less polar coatings is based on non-covalent bonds. Water, swelling and mechanical stresses can easily crash these interactions. Exchange of week non-covalent bonding with the more than one order in average stronger covalent bonding will increase durability of the wood coating. Covalent bonds will act as anchors for paintings or other wood finishes fixation on wood surface. Such anchoring will be enough flexible to withstand wood swelling and other stresses.

Background for such modifications of wood and coating interactions analogue can be found in the nature. In living organisms growing oligosaccharide chain is bonded with dolichol phosphate to ensure anchoring on lipid membrane surface during biological synthesis. Dolichols are polyprenols containing 16 - 20 isoprene units (80 – 100 carbon atoms) that ensure solution in membrane lipid layer (Text book of Biochemistry, 1997).

In case of wood we have contrary task to anchor practically non-polar coating to polar wood surface. Unsaturated fatty acid can be imagined in role of such anchor. Probability of direct reaction between fatty acid and wood (reaction 2) can be evaluated near to zero because of heterogenic character of reaction, incompatibility of wood and fatty acid, and lack of strong acid catalyst, because strong acid induces destruction of wood substance.

$$Wood-OH+RCOOH \xrightarrow{H^+} Wood-O-COR+H_2O \tag{2}$$

Where RCOOH is fatty acid; Wood-O-COR is wood and fatty acid ester; H2O is reaction by product – water; H+ is proton from acid catalyst.

A fatty acid moieties exchange between triacylglycerol molecules is well known in lipid chemistry (Gunstone, 1996).

$$R^1-O-COR^2+R^3-O-COR^4 \leftrightarrow R^1-O-COR^4+R^3-O-CO-R^2 \tag{3}$$

Where R1 and R3 are moieties of an alcohol or glycerol; R2 and R4 are fatty acid radicals.
Fatty acid exchange facilitates basic or acidic catalysts, enzymes and elevated temperature. Acidic conditions have to be avoided in case of wood but basic catalyst removal from the modified wood after reaction is unrealistic. Only elevated temperature can be used in this case. Acetyl group exchange with fatty acid moiety could be assumed.

$$Wood-O-COCH_3+R^1-O-COR^2 \xleftrightarrow{\Delta} Wood-O-COR^2+R^1-O-COCH_3 \tag{4}$$

Triacylglycerols (natural oils) have three times spacious molecule than methyl or ethyl esters of fatty acids. The lower molecular mass and volume of these esters will increase possibility of reaction 4. When R1 is methyl or ethyl group in reaction the equilibrium will be displaced in the desired direction because methyl or ethyl acetate is volatile (boiling point: 330 K and 350 K correspondingly) and easily removed from reaction mixture (Gunstone, 1996).

Above mentioned theoretical consideration made background for enhancement of hydrophobic properties of acetylated wood with plant oil or plant oil ethyl esters.

2 Experimental

Pine (*Pinus Sylvestris*) heartwood samples (50 mm in fibre direction and 25 × 15 mm in crosscut) were used in experiments. Series of 20 samples were formed for each treatment procedure and for control. After treatment series was divided in two parties for water vapour and liquid water absorbance tests accordingly.

2.1 Modification of wood samples

Following treatments were used to modify wood samples: 1) Acetylation with acetic anhydride without any catalyst at 390 – 395 K (Ac). 2) Acetylation and impregnation with rape seed oil ethyl esters (AcREE). 3) Acetylation and impregnation with rape seed oil ethyl esters with following extraction with petrol ether (AcREEPE). 4) Natural wood impregnation with rape seed oil ethyl esters (REE). 5) Natural wood impregnation with rape seed oil ethyl esters with following extraction with petrol ether (REEPE). 6) Acetylation and impregnation with rape seed oil (AcR). 7) Acetylation and impregnation with rape seed oil then extraction with petrol ether (AcRPE). 8) Natural wood impregnation with rape seed oil (R). 9) Natural wood impregnation with rape seed oil then extraction with petrol ether (RPE). Designation in brackets is introduced for convenience of data presentation and is used below. Acetyl group content in acetylated wood samples was above 20%. All acetylated wood samples were dried in oven till constant mass. Wood samples were impregnated with rape seed oil or rape seed oil fatty acid ethyl esters by immersing in oil at 395 – 400 K for 24 h. After impregnation the treated samples were kept at same temperature till hardening of oil. Half of the each set of impregnated samples was extracted with petrol ether in the Soxhlet extractor for 48 h to leach unattached rape oil or fatty acid ethyl esters. Natural (untreated) wood (C) samples were used as reference. Samples were weighed and measured before, between and after treatments.

2.2 Fatty acid ethyl ester (REE) preparation

16.94 g (0.302 mole) of catalyst – potassium hydroxide was dissolved by stirring in 1.67 L (28.68 moles) of absolute ethyl alcohol. 1.13 L (0.312 mole) of rape seed oil was added to solution. Mixing was continued for 1 hour after moment when homogeneous solution was obtained. Solid by-products were allowed to precipitate from reaction mixture. Then clear solution of rape seed oil fatty acid ethyl ester (REE) was decanted off in separation funnel. Definite volume of pure water was added and after forming of sharp two phase layers glycerol-containing fraction was separated. Amount of added water was determined from results of titration of reaction mixture with distilled water until vanish emulsion formation. The layer of REE was three times washed with equivalent volume of 5% solution of sodium chloride and then with distilled water. REE solution was dried under solid sodium sulphate and solvent – ethanol was distilled off. Outcome of REE was 84%.

2.3 Water vapour absorption and wood swelling tests

Oven dried wood samples were place in desiccator, walls of which was inlayed with filter paper. Paper underneath edge was in contact with water to ensure desiccator’s volume saturation with water vapours at 293±3K. Periodically samples were weighted on analytical balances in glass boxes and measured with micrometer during 1 month.

2.4 Water absorption and wood swelling tests

Oven dried wood samples were immersed in water and loaded. Each day samples were weighted and measured and water in vessel was changed during 7 month. Temperature was maintained 293±3 K.

3 Results and Discussion

3.1 Results of wood impregnation with oil and fatty acid esters

Acetylated wood or natural wood impregnation with rape seed oil or REE and extraction with petrol ether results are presented in Tab. 1. All changes in the wood sample mass were normalized with respect to corresponding oven dried mass of the wood sample before any treatment in order to compare impregnation results.

Table 1Absorption of fatty acid esters and rape seed oil in acetylated and natural wood. (Mean with standard deviation)

Wood treatment	Oil or esters absorption, %	Non-extractive fraction, %
Acetylation and impregnation with REE	35,8 ± 6,6	10,1 ± 3,0
Acetylation and impregnation with R	32,8 ± 6,2	0,8 ± 1,0
Impregnation with REE	11,7 ± 5,2	10,9 ± 4,8
Impregnation with R	14,1 ± 4,5	3,7 ± 1,1

Wood acetylation increases natural oil or it ethyl ester up-take in wood approximately 2 – 3 times. It will agree with consideration that hydroxyl group replacement with the less polar oxyacetyl group will contribute in wood cell wall substance interaction with oil or REE in acetylated wood. More voluminous fatty acid group will swell wood matrix in case of acid radical exchange in accordance reaction 4. Swelling by 1,5±0,4% was observed only in case of impregnation of acetylated wood with fatty acid ethyl esters with respect to initial volume of wood sample. Smaller in size REE could penetrate in wood cell wall substance and swell it in contradistinction to voluminous molecules of natural oil.

Formation of acetylated wood and with fatty acid ethyl ester solid solution would be another reason for the wood swelling during impregnation. Extraction of REE from wood with volatile non polar solvent would eliminate such kind of swelling, because of no chemical bonding between wood and ester. Chemically bonded fatty acid residues could not be extracted. The third factor in changes of swelling could be rearrangement and relaxation processes of modified wood matrix without any elimination of bonded fatty acid. Acetylated wood swelling with REE and changes by extraction with petrol ether measurements shoved decrease by half to 0,7 ± 1,3% in average. The great variance of swelling reduction could not to allow made choose between acyl radical exchange and solid solution formation hypotheses. Equal ratio of non-extractive fraction of REE in impregnated acetylated and natural wood will raise doubt about contribution of reaction 4 in acetylated wood swelling with REE.

Above mentioned did not contradict possibility to occur reaction 4 with acetyl groups on the inner and the outer surfaces of wood sample. Bonded unsaturated fatty acids could polymerize with rest ester or oil to form chemically bonded polymer layer on wood surface. Such coating would change hydrophobic properties of wood.

Natural oil molecules are more voluminous than REE and have less possibility to diffuse in cell wall. Really acetylated wood swelling by impregnation with natural oil was less than 0.2 – 0.3% in average. Natural wood impregnation with both rape oil and REE without acetylation caused shrinking instead of swelling by 0.2 – 0.3%, but these small changes in sample volume did not exceed standard deviation in both cases. Fatty acid and acetyl group exchange it at all would occur only on wood surface.

3.2 Absorption of moisture

Absorption of water vapours in modified and natural wood was normalized in respect to the corresponding oven dried to at 373K mass before modification (eq. 5).

$$MA = \frac{M_i - M_o}{M_o} \tag{5}$$

Where MA was moisture absorption; Mi was mean of sample mass at the moment of measurement; M0 was mean of oven dried sample mass.

Power function gave the best fit of the moisture absorption (MA) in the wood samples. Function for moisture absorption velocity calculation was obtained by differentiation of the power function (6).

$$V_{MA} = (MA)' = \left(k \cdot t^{n}\right)' = k \cdot n \cdot t^{n-1} \tag{6}$$

Where: VMA was moisture absorption velocity; MA was approximation with power function, k and n were empiric constants, t was time, h.

The mean of moisture absorption velocity in modified wood was compared with the same for control samples. The normalised difference in moisture anti-absorption dynamic (DMAD) of each treatment (i) to the respect of untreated wood was calculated by expression (7) to evaluate wood treatment method efficiency.

$$DMADi = \frac{V_{MAc} - V_{MAi}}{V_{MAc}} \tag{7}$$

Where: VMAc was the mean of moisture absorption velocity of untreated (control) wood samples; VMAi was the mean of moisture absorption velocity of treated wood samples. Calculation results are presented in Fig. 1.

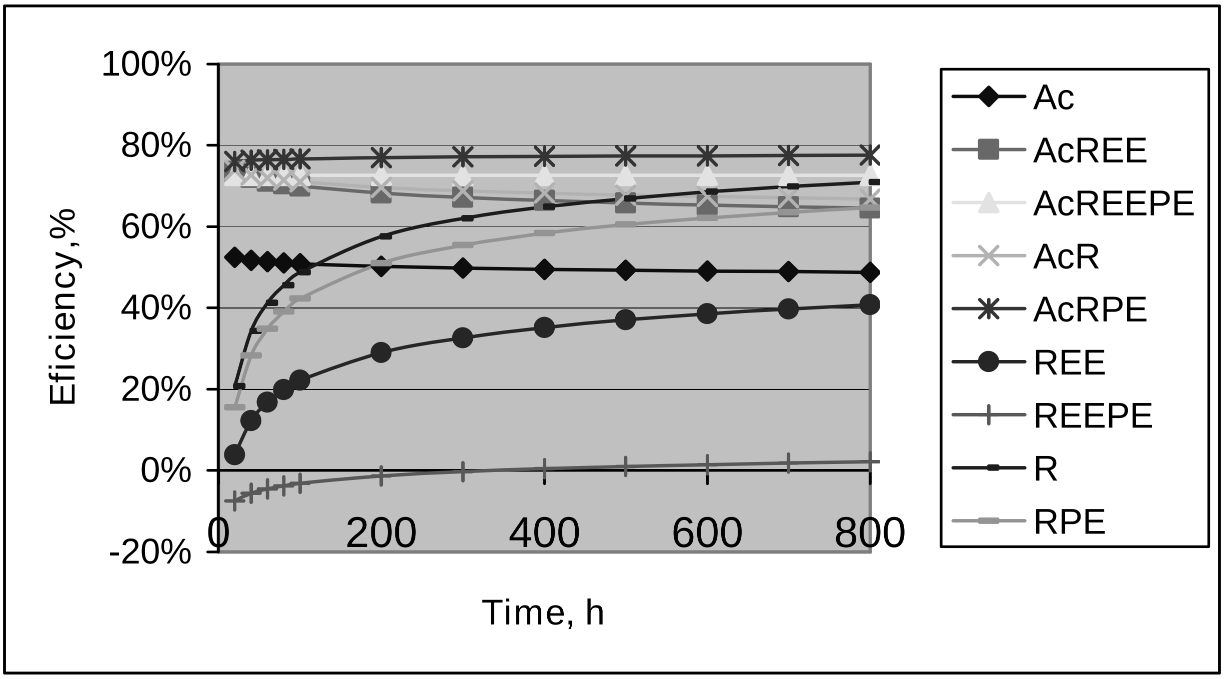

Figure1. Moisture absorption dynamics' change efficiency

Woods' hydroxyl group hydrogen atom replacement with acetyl group would have an impact on water adsorption and diffusion in the wood. Really the velocity of water vapour absorption in acetylated wood is practically proportional to the one of natural wood. Impregnation of acetylated wood with natural oil or REE did not change wood absorption kinetics curve shape but only shifted it. Natural wood impregnation with oil or ester changed kinetics of water vapour absorption in comparison with non-impregnated wood and any kind of acetylated wood. It means that the acetylation had the key effect in moisture absorption reduction in wood. Because reduction in velocity of moisture absorption in impregnated wood without acetylation had more complex character, the effect of impregnation had less effect on reduction of water absorption velocity in first days and increased during experiment. Wood impregnation with fatty acid esters had less effect on protection against moisture contrary to wood swelling. Acetylated wood samples impregnated with REE swelled less in conditions of moisture absorption tests (Fig.2.).

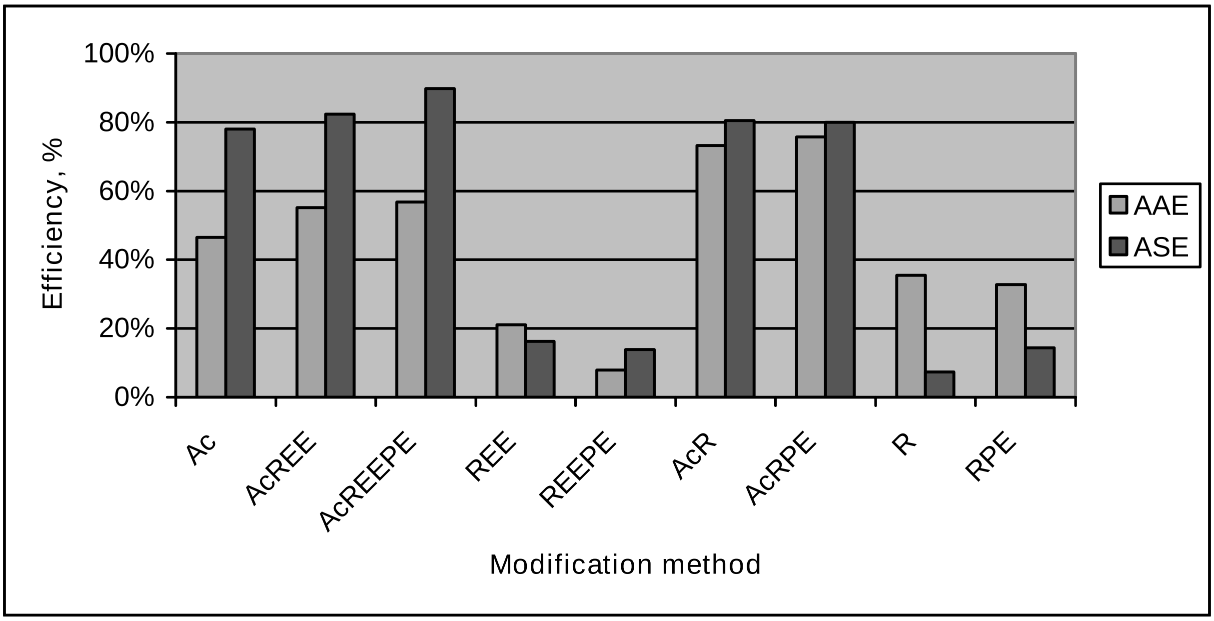

Figure 2. Anti water vapour absorption (AAE) and anti swelling (ASE) efficiency of wood modification

3.3 Absorption of water and swelling

Changes in wood sample mass and dimensions during water absorption tests were used to calculate following indicators. Normalized water absorption (WA) was calculated in analogous way than the moisture absorption by formula (5). Wood sample swelling (S) was calculated than ratio of difference of volume between swelled sample and oven dried sample to volume of oven dried sample. Anti absorption efficiency (AAE) was calculated as ratio of difference between normalized absorption of water (WA) in natural wood and modified wood to WA of natural wood. In the same way was calculated anti swelling efficiency (ASE) as ratio of difference between normalized swelling of control wood and modified wood samples to control wood. Wood void volume fulfilment with water (VVS) was estimated with equitation 8 between volumes of absorbed water to total wood or modified wood void volume.

$$VVS = \frac{m_{aq}}{d_{aq} \cdot m_o \cdot \left(V_s - \frac{m_o}{d_w} - \frac{m_{Ac}}{d_{Ac}} - \frac{m_{impr}}{d_{impr}} \right)} \qquad (8)$$

Where: maq was mass of absorbed water, g; daq was density of water, g.cm-3; mo was mass of oven dried entreated wood sample, g; Vs was volume of treated wood sample, cm3; dw – density of wood substance = 1.5 g.cm-3; mAc was mass of acetyl groups, g; dAc was density of acetyl group, g.cm-3; mimpr was mass of impregnated substance, g; dimpr was density of impregnated substance, g.cm-3. Results of these indicators (AAE, ASE and VVS) calculations are presented in Fig. 3.

Then it is compared with void volume fulfilment in control samples and volume un-fulfilment efficiency (AFE) was calculated in same way as AAE and ASE.

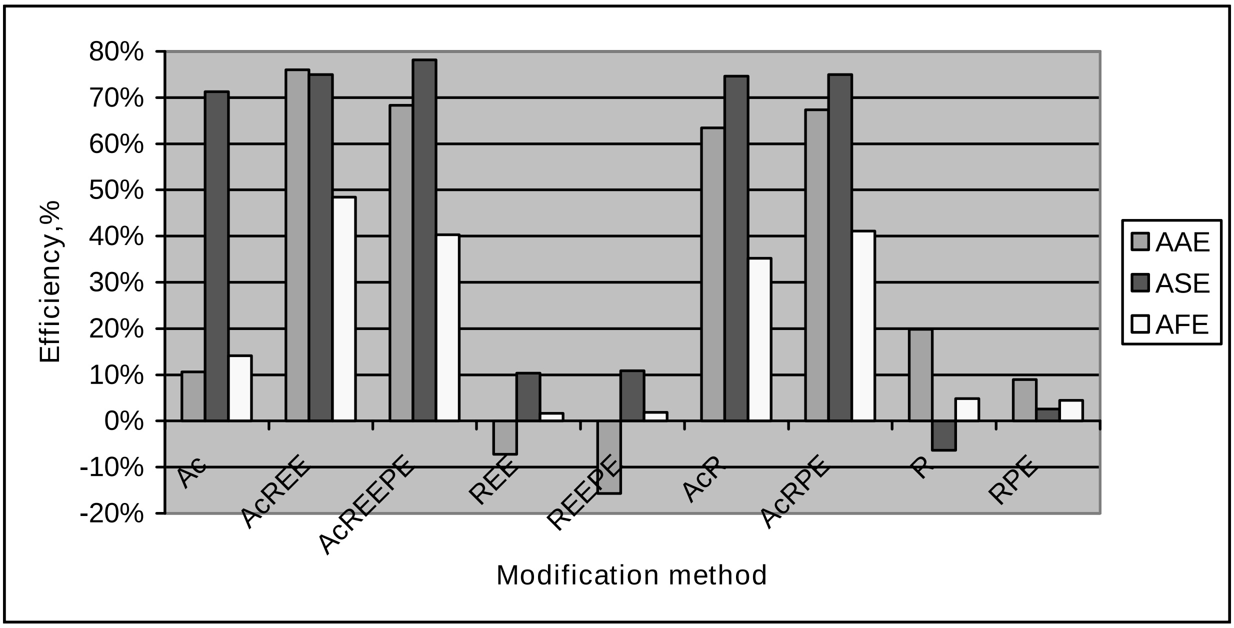

Fig. 3. Comparison of wood treatment method effect on water absorption and swelling in water

Water absorption in acetylated or acetylated and impregnated wood samples did not reach equilibrium even after 7-month soaking in water in comparison with control samples or samples only impregnated with oil or ester. Calculations showed that only all void volumes of non-treated wood or impregnated with rape seed oil or REE samples were filled with water. Acetylated and acetylated and impregnated samples floated but control and only impregnated samples sunk. Wood impregnation with oil or REE without acetylation had many times less protective effectiveness than impregnation of acetylated wood in conditions of permanent contact with liquid water. Acetylation without impregnation with oil or ester gives low AAE. Increased water absorption in case of wood impregnated with REE can be interpreted with influence of traces of monoacylglycerols and diacylglycerols in REE. Diacylglycerols- and monoacylglycerols are normal intermediate products in synthesis of REE from rape seed oil and have properties of detergents. Non-polar solvent – petrol ether extracted less polar fatty acid ester molecules than more polar partly acylated glycerol with free hydroxyl groups. It could be reason why higher rates of moisture and water absorption were observed in beginning of experiment.

Complete wood samples' swelling was obtained within 1 – 1.5 days in water absorption tests. The main effect in swelling reduction had wood acetylation. Impregnation of acetylated wood with oil or REE improved ASE only by few percents. Moisture absorption tests showed that impregnation of acetylated wood with REE significantly decreased wood swelling only in initial period. Effectiveness of acetylated wood impregnation with oily substances is low when acetylation degree of wood was low. Wood impregnation with natural oil or REE alone could not protect against water diffusion in cell wall an it swelling.

Liquid water absorption in wood was faster than from vapour phase and was more convenient to evaluate total wood modification method effectiveness.

4 Acknowledgements

I thank to Laimonis Kulins from the Department of Wood Processing of Forest Faculty of Latvia University of Agriculture for wood sample processing, and I express thanks to Latvian Council of Science for financial support.

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INNOVATIVE MULTI-FUNCTIONAL WOOD TREATMENT PROCESS BASED ON BI-OLEOTHERMY

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COST E22 Final Workshop, Estoril, Portugal

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Old innovative multi-functional wood treatment process based on bi-oleothermy

Bi-oleothermy

Simple multifunctional process that ensures deep wood penetration by hydrophobic products.

The process would make it possible to switch from costly, complex facilities at classified sites to more economical, simpler facilities that will rule out the need for pressure resistance controls or site classification.

Impregnation principles

The bi-othermic impregnation principle is based on the fact that the water vapour in a material condenses as it cools.

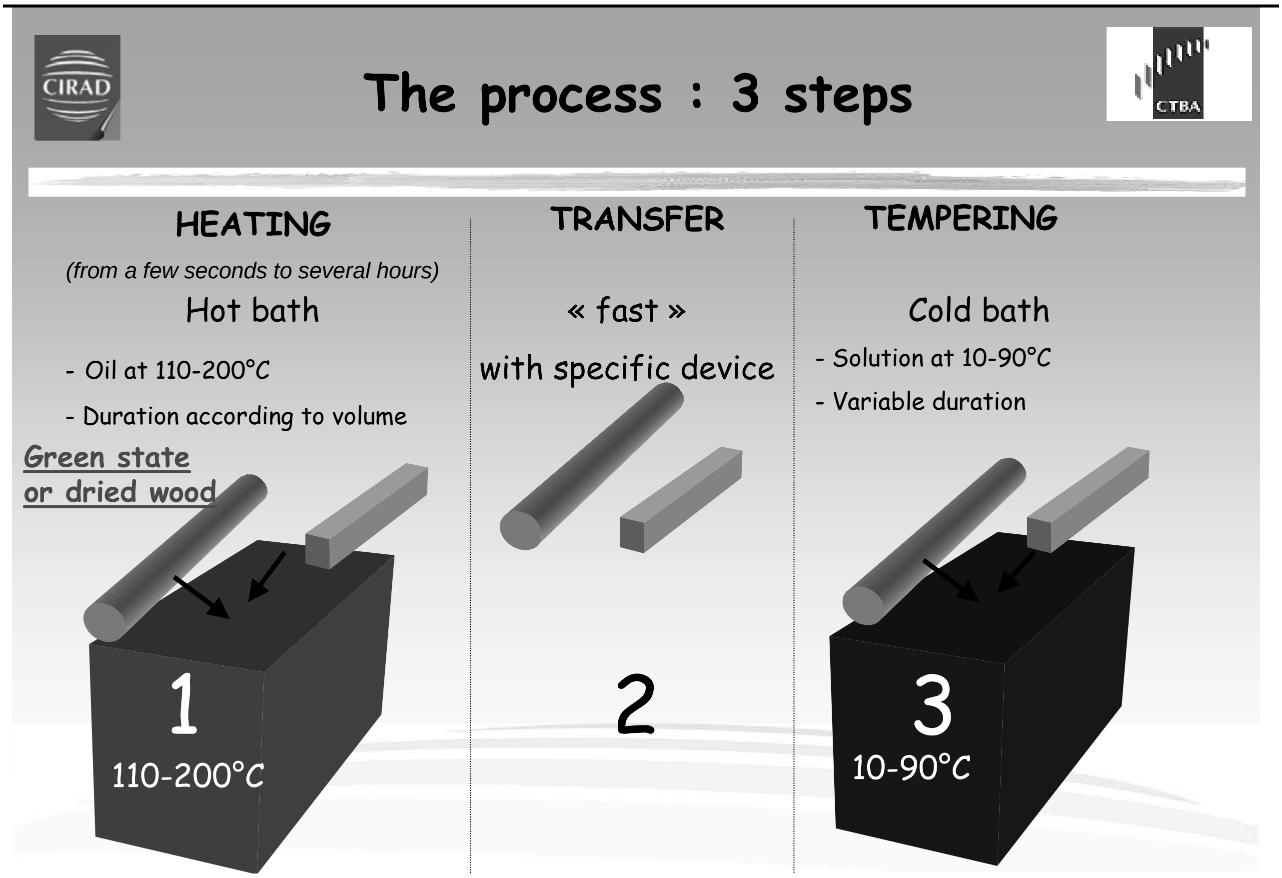
When the material cools, the vapour condenses, creating a vacuum that draws in the liquid from the treatment bath.

When a porous solid such as wood is heated, the water in its lumens (cavities) vaporizes, starting from the outside of the wood and moving towards the heart.

Objectives

To stabilize the dimensions and increase the durability of, and perhaps to confer specific properties (colour, fire resistance, etc) on, green wood pieces, pieces with an intermediate moisture content (> the fibre saturation point) or dried pieces, by:

- A hydrothermal modification that helps to relieve the internal stresses thus ensuring better product stability,
- Intense drying, ie much faster than with the methods currently used,
- Impregnation of the wood with hydrophobic substances that may or may not contain treatment products (biocides, fire retardant, colourings, etc).



The process : 3 steps

HEATING

(from a few seconds to several hours)

Hot bath

- Oil at 110-200°C
- Duration according to volume

TRANSFER

« fast »

with specific device

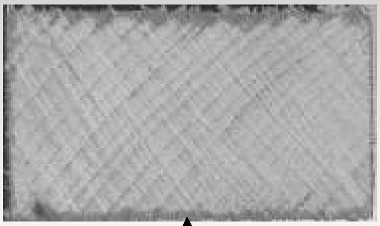
TEMPERING

Cold bath

- Solution at 10-90°C
- Variable duration

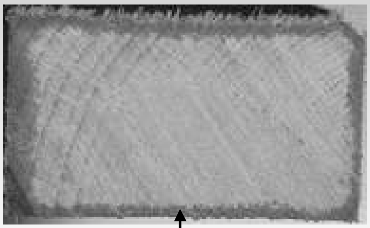
Effect

Piece* of *Fagus sylvatica* (10-11% moisture content) soaked for 1 hour in linseed oil at room temperature



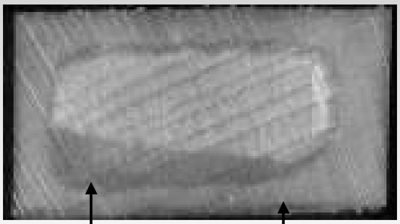
Linseed oil

Piece* of *Fagus sylvatica* (110-120% moisture content) heated for 1 hour in groundnut oil at 180°C



Groundnut oil

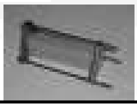
Piece* of *Fagus sylvatica* (110-120% humidity) heated for 30 minutes in groundnut oil at 180°C then soaked for 30 minutes in linseed oil



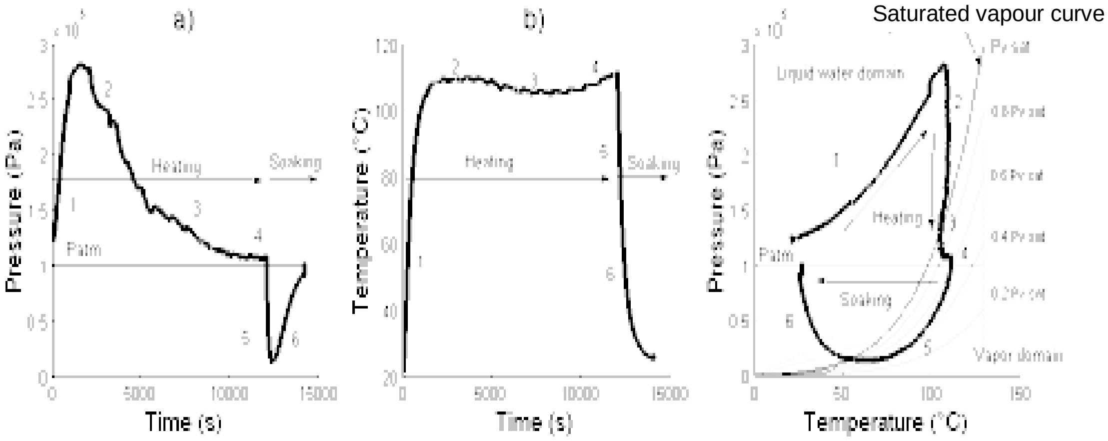
Groundnut oil

Linseed oil

*** Closed ends**



Characteristics curves



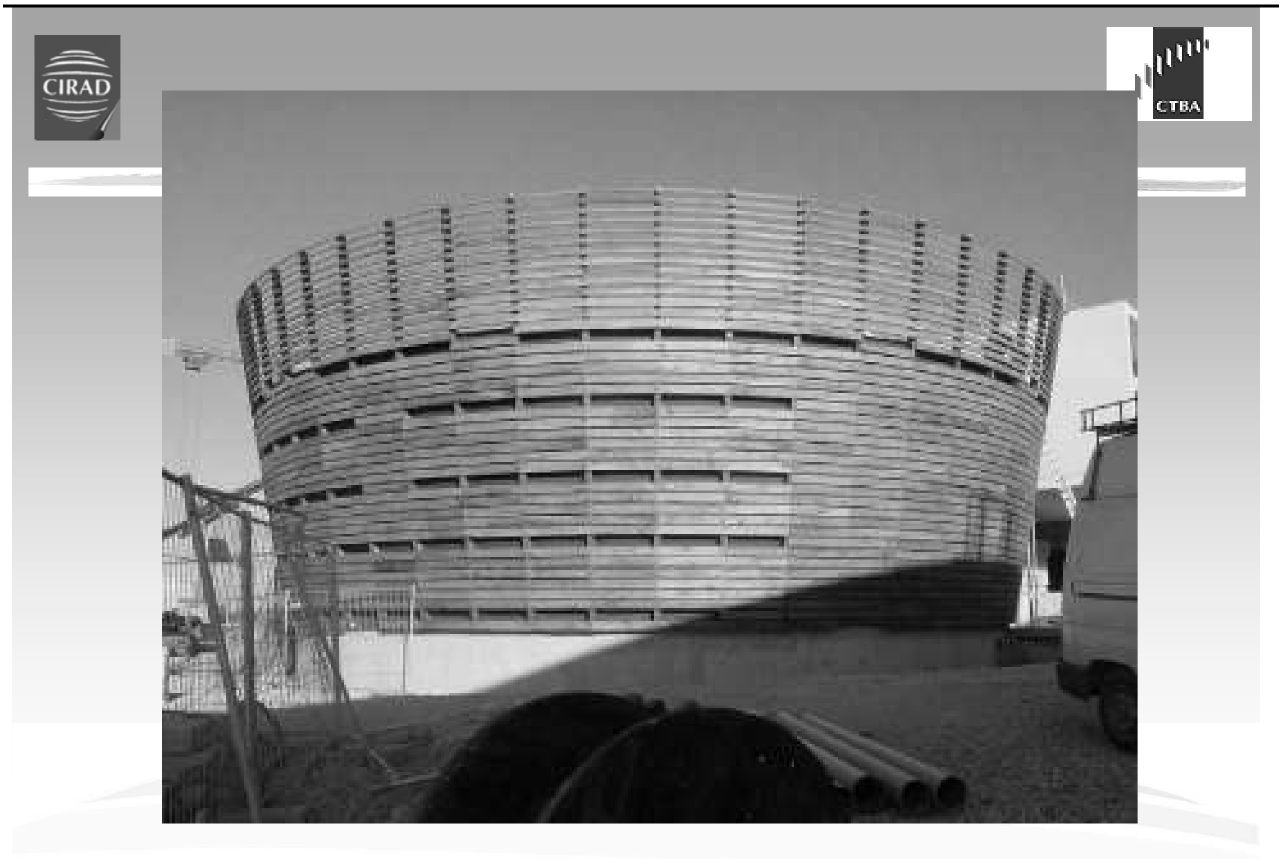
Saturated vapour curve


500 KG

CIRAD

CTBA


CIRAD
CTBA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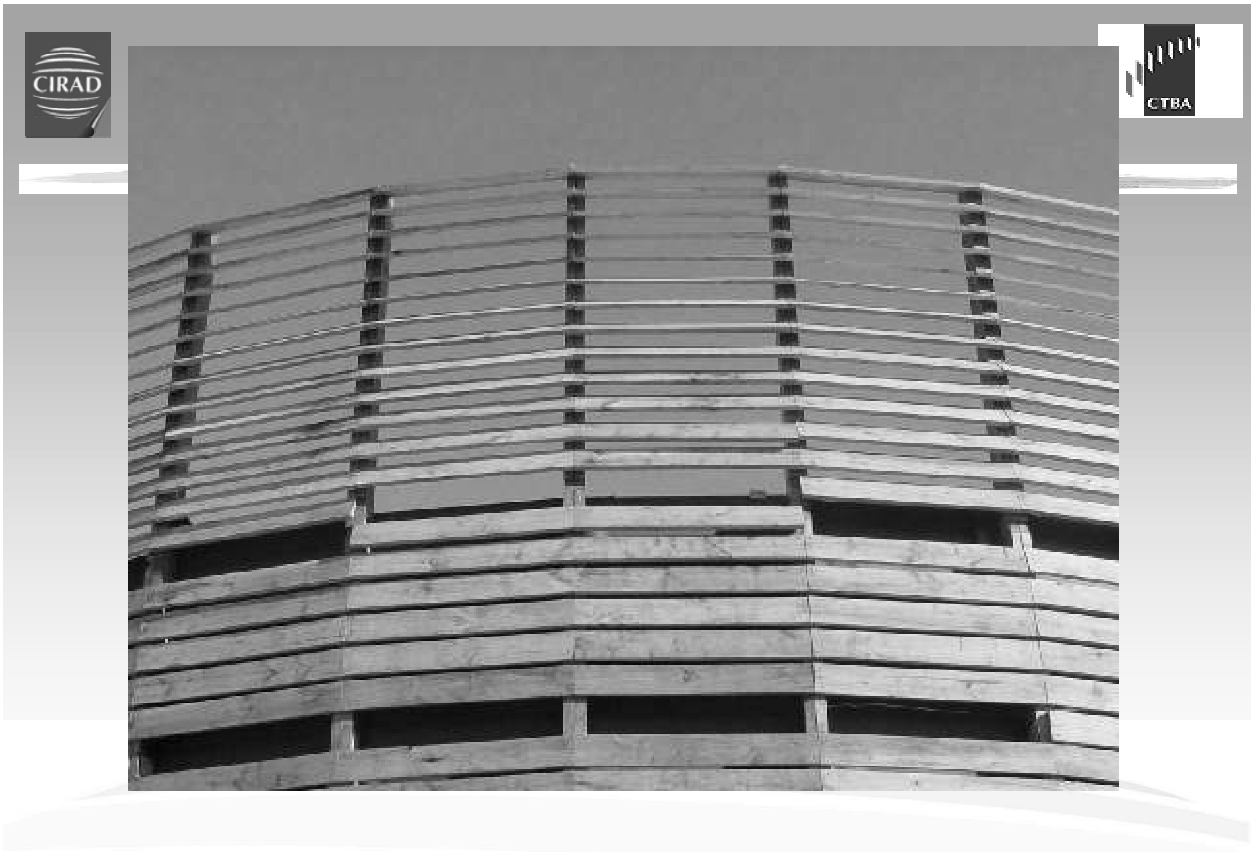
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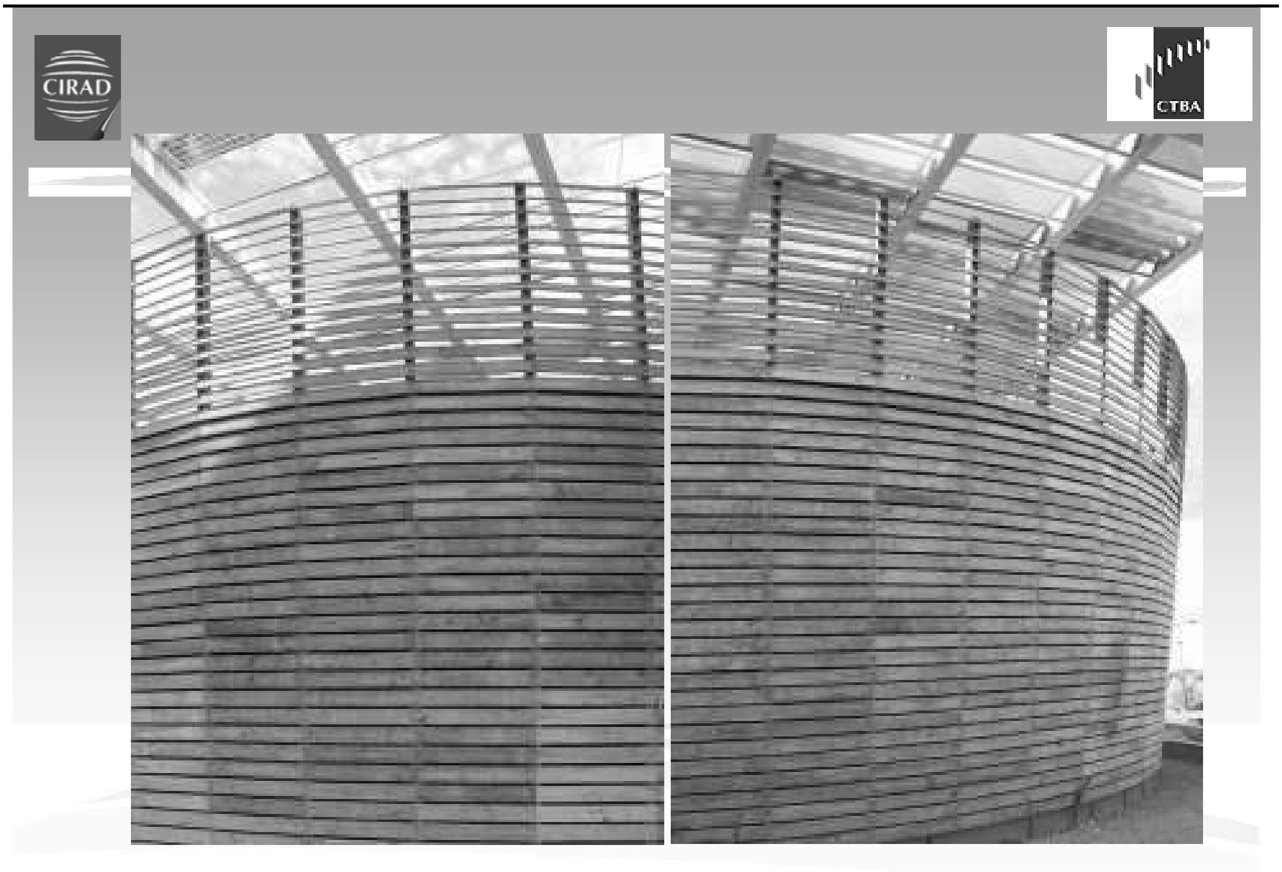
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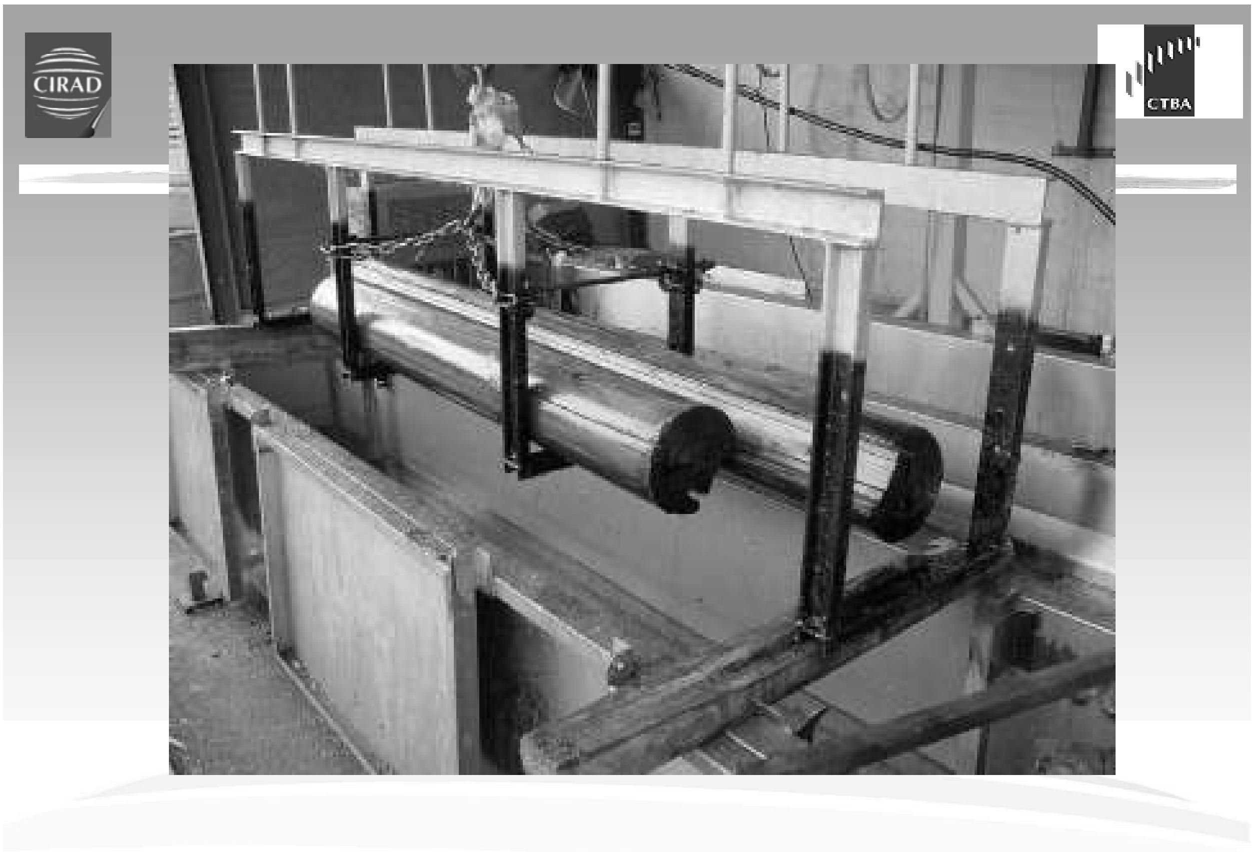
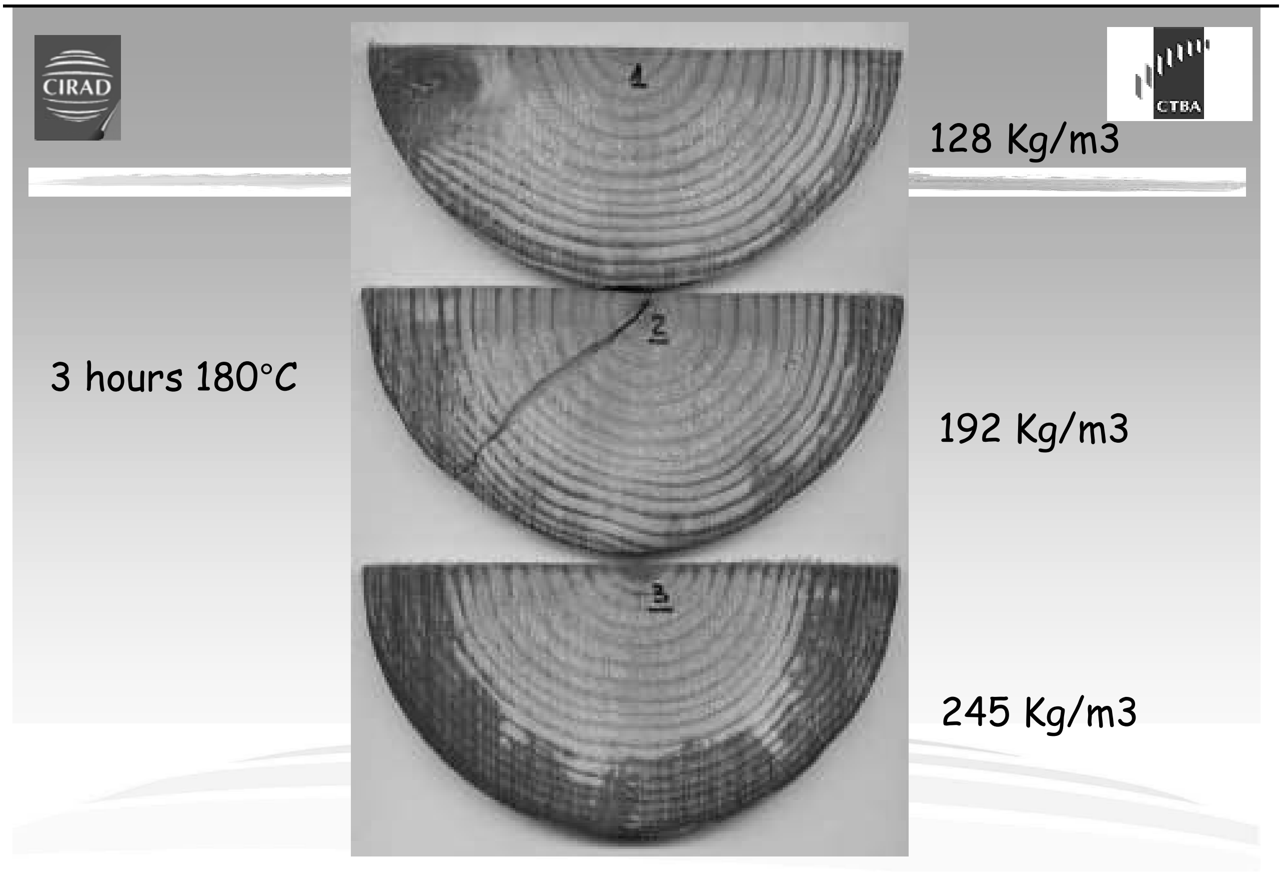


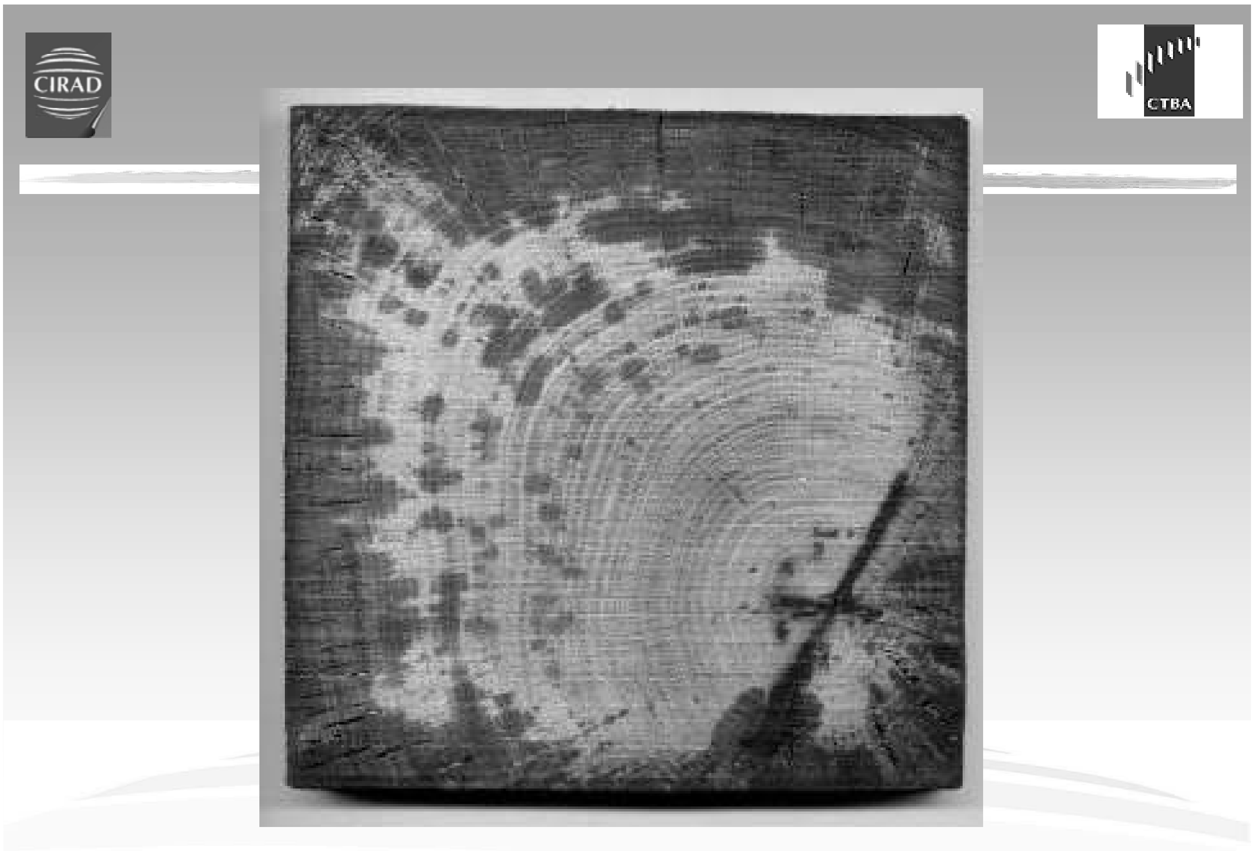


3 hours 180°C

128 Kg/m3

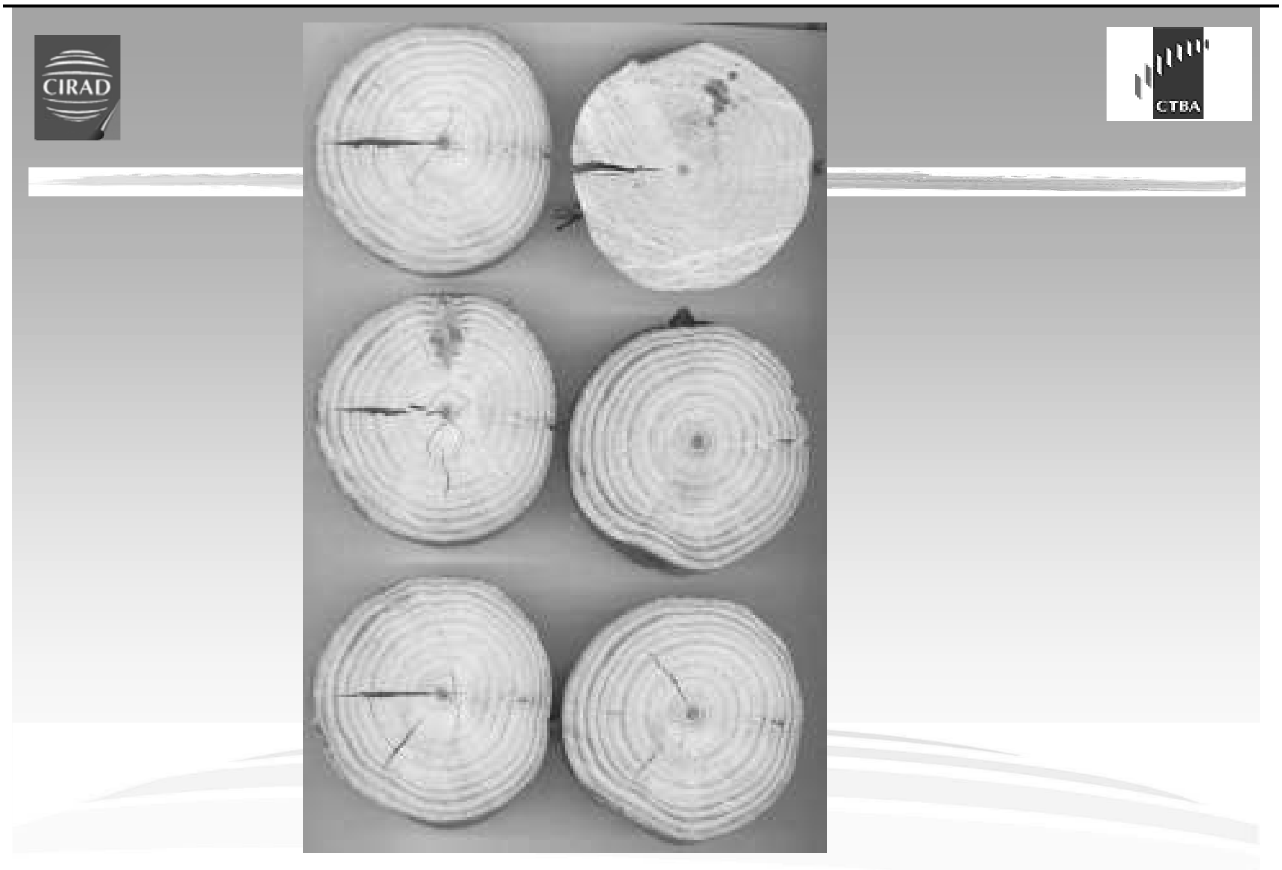
192 Kg/m3

245 Kg/m3



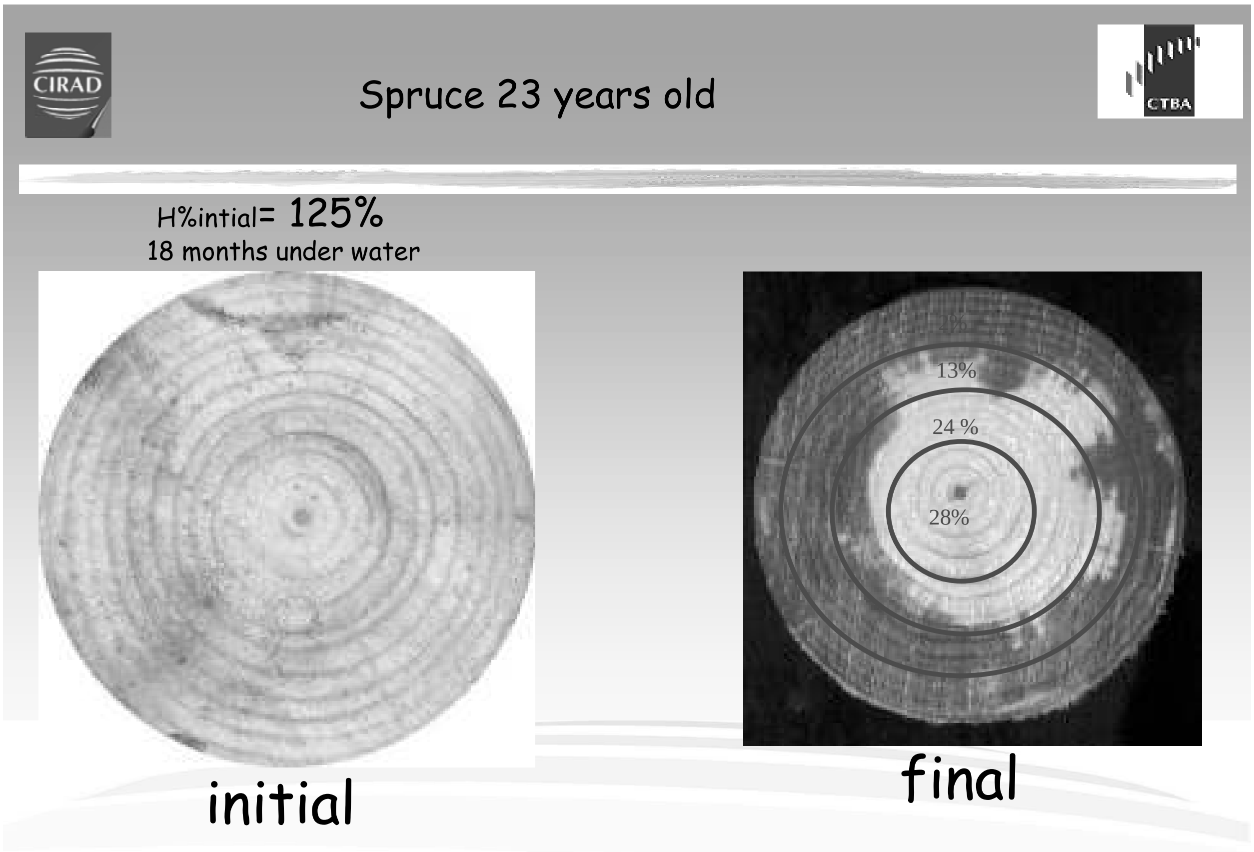
CIRAD

CTBA



CIRAD

CTBA



Spruce 23 years old

H%intial= 125%
18 months under water

Park bench

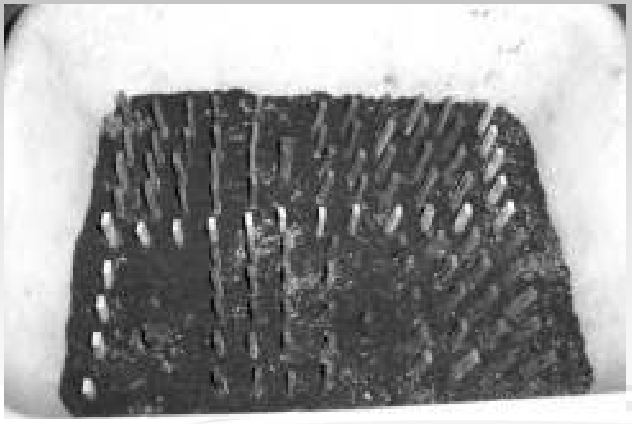

CIRAD
CTBA

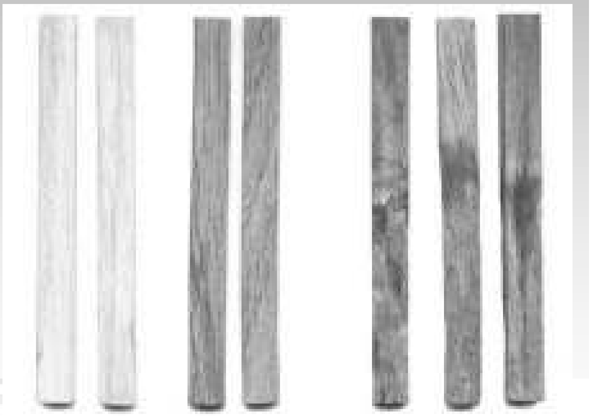

CIRAD
CTBA

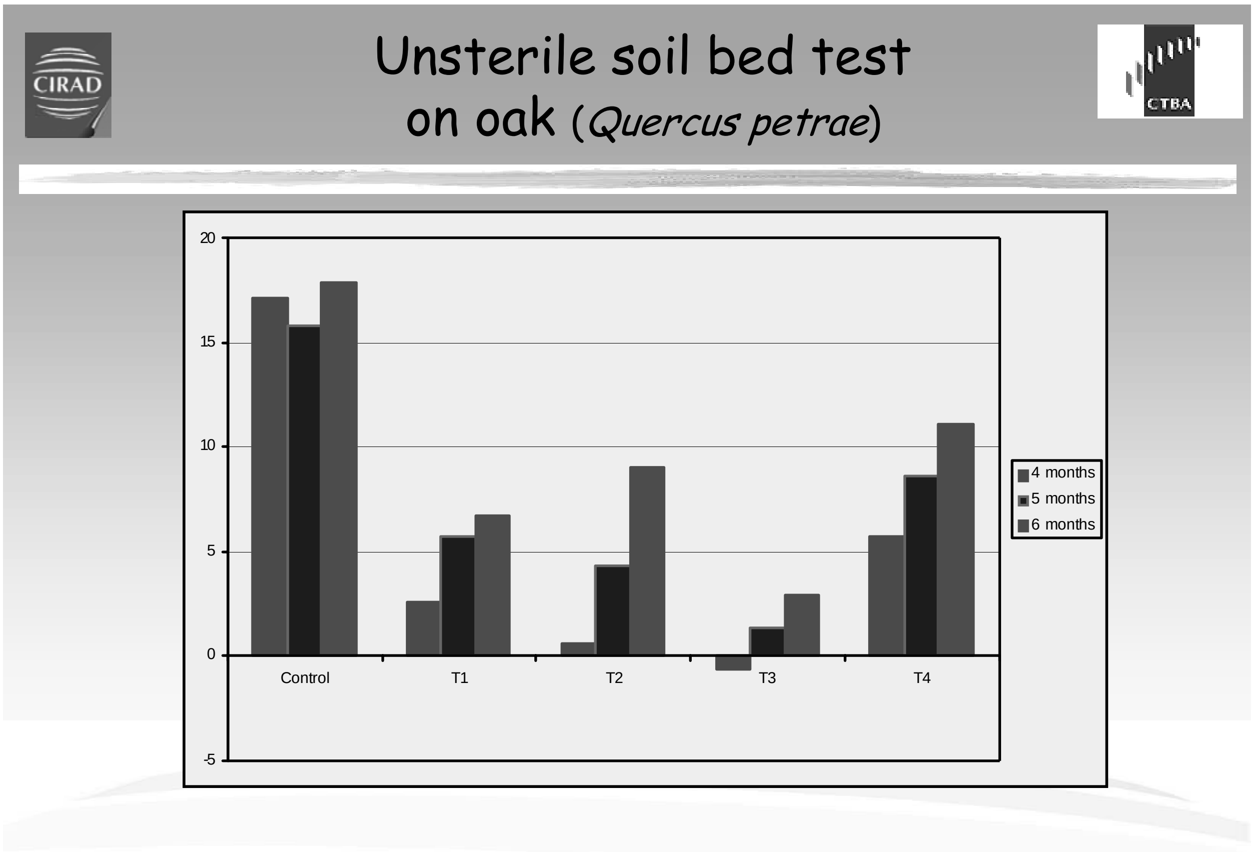
Unsterile soil bed test on oak (*Quercus petrae*)

Tropical conditions

T = 27 °C
Hr = 80 %







Unsterile soil bed test on oak (*Quercus petrae*)

Conclusions - issues

Pre-treatment
Hot oil bath and cold bath wood monitoring
Transfert monitoring
conditionning

Process control

Hot oil technical choice : change, recycle, energy
Drying oils : chemistry

formulation

Costs management

Industrial future

INVESTIGATIONS OF THE ROLE OF CELL WALL MOISTURE CONTENT AND MICROPORE BLOCKING IN THE DECAY PROTECTION MECHANISM OF ANHYDRIDE MODIFIED WOOD

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Keywords: Acetylation, decay protection, solute exclusion, fibre saturation point

Abstract: *This paper reports on the results on an ongoing study into the mechanisms by which chemical modification, thermal modification, and cell wall impregnation with monomers with subsequent polymerization impart decay protection to wood. Results from two preliminary studies, where wood was modified with a variety of anhydride reagents of different molecular weight were presented at an earlier meeting of this action. This paper presents the results of a recent study of the changes of the cell wall volume due to acetylation determined using a helium pycnometer. In addition, the results of two previous studies of the cell wall accessibility of anhydride modified wood are given and discussed. The role of cell wall moisture content and cell wall micropore blocking are considered as possible decay protection mechanisms in anhydride modified wood.*

1 Introduction

Although it has long been recognised that acetylation of wood is capable of providing decay resistance to wood there is, as yet, no consensus regarding the reasons for why such protection is afforded. Briefly, three mechanisms have been postulated for the observed protection, namely:

- Modification alters the chemical nature of the material, so that the enzymes of decay fungi are no longer capable of recognising the substrate.
- Modification lowers the moisture content of the cell wall below the threshold at which decay can take place.
- Modification blocks the cell wall micropores so that degradative agents cannot gain access to the cell wall interior.

It was argued in the previous paper that the first of these mechanisms was not viable, at least during the early stages of decay, because the enzymes responsible for degrading the cell wall polymers are not capable of penetrating the cell wall until decay is well advanced. The inability of these enzymes to penetrate the cell wall has led to the realization that low molecular weight degradative agents (LMWDA's) have an important role to play in attacking the cell wall, resulting in an increase of the dimensions of the cell wall micropores, whereby access by enzymes may be facilitated. Thus, rather than postulating that the substrate is unrecognisable to enzymes, a chemical blocking mechanism may be responsible which prevents the action of LMWDA's. If such a mechanism is responsible, then it would be expected that decay resistance would be related to the extent of OH substitution. Since all studies of the decay resistance of anhydride modified wood have been concerned with acetylated wood, there has been no means of checking whether the putative relationship between decay resistance and OH substitution was in fact true. Only recently have a few studies appeared where the decay resistance of wood modified with a variety of anhydride reagents of different molecular weight has been examined. By modifying with anhydride reagents of different molecular weight it is possible to achieve a variety of levels of OH substitution at the same weight percentage gain (WPG). The wood science group at Bangor has been working with a range of anhydride reagents for a number of years (Fig. 1).

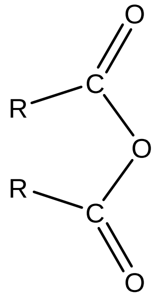

Figure 1: Anhydrides referred to in this paper (R=CH3, acetic), (R=C2H5, propionic), (R=C3H7, butyric), (R=C4H9, valeric) (R=C5H11, hexanoic)

As an illustrative example, results from studies of the decay resistance of Corsican pine modified with a range of anhydride reagents against the brown rot fungus Coniophora puteana is shown below in Fig. 2 (Papadopoulos and Hill, 2002; Farahani, 2003).

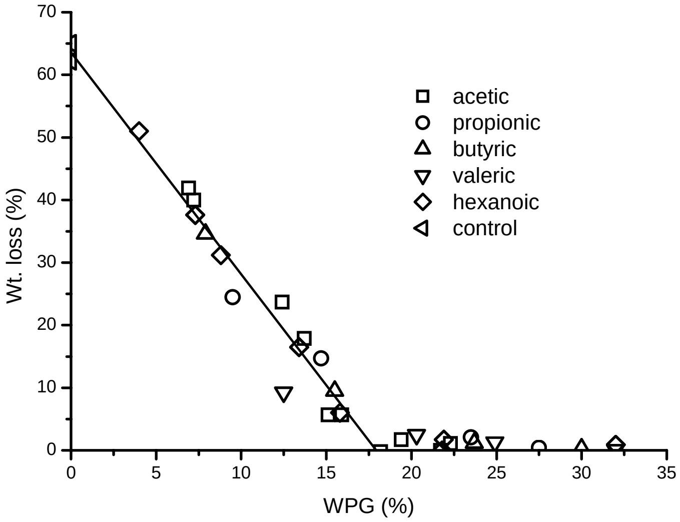

Figure 2: Decay resistance of Corsican pine modified with a range of anhydrides

The above plot shows clearly that the decay resistance is a property of the WPG due to reaction, not level of OH substitution. Similar results have been found for soft rot attack (Suttie, *et al.*, 1999), and preliminary data for exposure to *Phanerochaete chrysosporium* and *Trametes versicolor* (Hill *et al.*, 2003) also indicate that WPG is the primary factor in determining decay resistance. This would indicate that the mechanism is related to bulking of the cell wall. Accordingly, a study was undertaken to investigate this possibility. If bulking is responsible then there are two possible explanations, either a lowering of cell wall moisture content, or blocking of the cell wall micropores. An important part of this study was to determine what the actual change in cell wall dimensions was due to modification. Such information cannot be obtained by determining the external dimensions of wood samples, since there is no reason to suppose that the lumen dimensions remain unchanged during the modification process. The technique of helium pycnometry allows for very accurate determinations of the volume occupied by samples of very complex geometry. This technique has never before been used to determine volume changes of the cell wall due to chemical modification. For determination of the cell wall microporosity of anhydride modified wood, the technique of solute exclusion was used. There have been no reports in the literature where this method has been applied to chemically modified wood.

2 Results

2.1 Helium Pycnometry

The percentage volume change of wood samples (same samples before and after acetylation) as determined by helium pycnometry and by determining external dimensions of the samples are shown in Fig. 3, below.

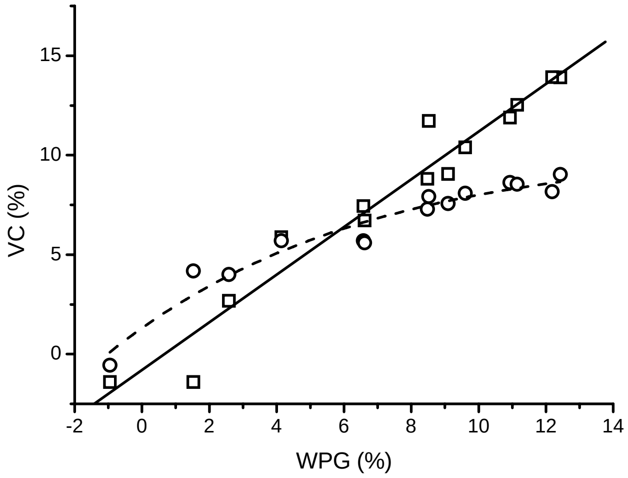

Figure 3: Relationship between percentage volume change of wood samples and weight percentage gain due to acetylation as determined by helium pycnometry (squares, unbroken line) and by measuring external dimensions

The above indicates that there is a linear relationship between percentage volume change as determined by helium pycnometry and WPG, but this is not found when volume change is determined by measuring external dimensions. This shows that the measurement of external dimensions cannot be used to determine changes in the cell wall volume. Up to a WPG of 6%, the percentage volume change is larger than the change in cell wall volume, but is lower above a WPG value of 6%. This phenomenon can be rationalized by consideration of changes in the lumen volume as shown in Fig. 4, below.

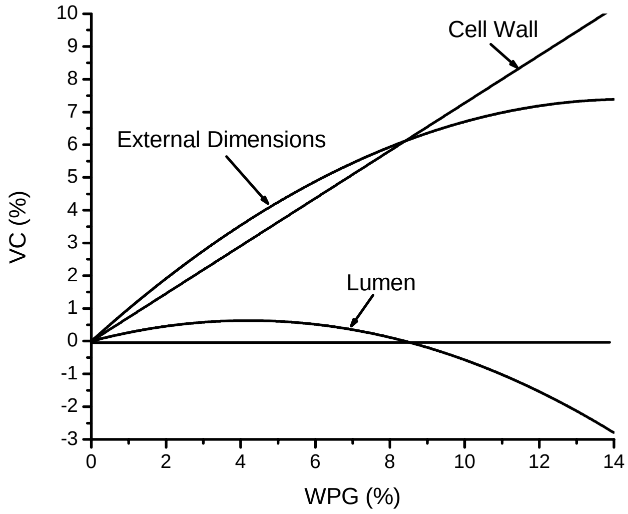

Figure 4: Theoretical illustration of the relationship between percentage volume change of the wood determined by external dimensions, percentage volume change of the cell wall (determined by helium pycnometry) lumen percentage volume change and WPG due to reaction with acetic anhydride

It can be seen from the above example that this behaviour is explained by an increase in lumen dimensions at low WPG levels, followed by an expansion of the cell wall into the lumen at higher WPG's. Expansion of the cell wall into the lumen at high WPG's has been noted by previous researchers when studying acetylated wood using electron microscopy (Evans et al., 2000; Sander et al., 2003).

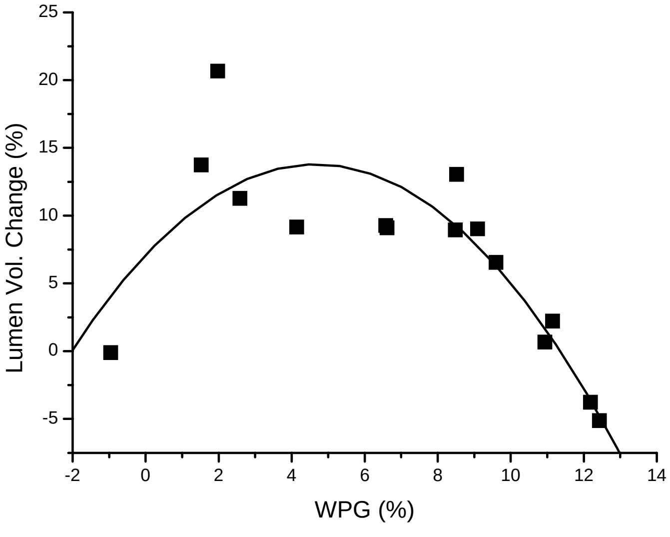

Figure 5: Calculated percentage change in lumen volume as a result of acetylation determined by subtracting helium pycnometry volume determinations from volumes obtained by measuring external dimensions

Figure 5 shows values obtained by subtracting the helium pycnometry volume measurements from the external dimension volume determinations, which whilst being subject to some scatter, nonetheless agree reasonably well with the behaviour predicted theoretically.

Molar volume (the volume occupied by one mole of acetyl groups) can be calculated from the absolute volume change at a specific weight gain. The results from this calculation are shown below in Fig. 6.

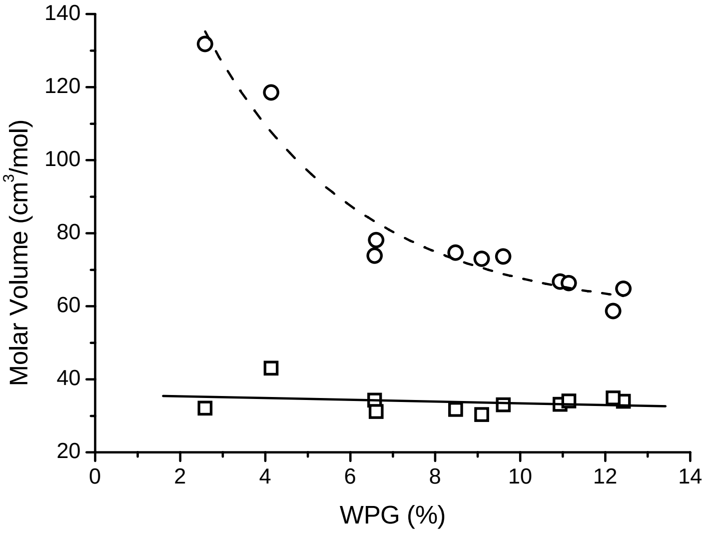

Figure 6: Change in molar volume of acetyl group in cell wall as calculated from external dimension measurements (circles, broken line) and by helium pycnometry

This shows that the molar volume changes very little as WPG increases, and that use of external dimensions is an unreliable method for determining this parameter. The average molar volume of the acetyl group, over the range of WPG's studied, is 33 cm3 mol-1.

2.2 Solute Exclusion

A few examples of the determination of the cell wall micropore accessibility of unmodified and acetylated wood using solute exclusion are shown in Figs. 7,8,9, below (Forster, 1998).

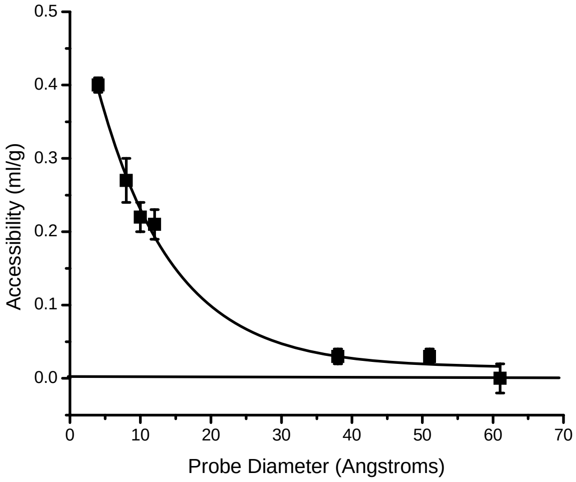

Figure 7: Accessibility of the cell wall of unmodified Corsican pine

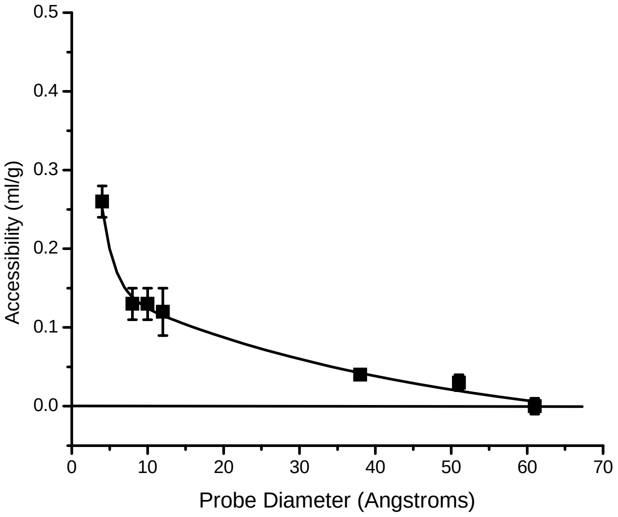

Figure 8: Accessibility of the cell wall of acetylated Corsican pine (12.8% WPG)

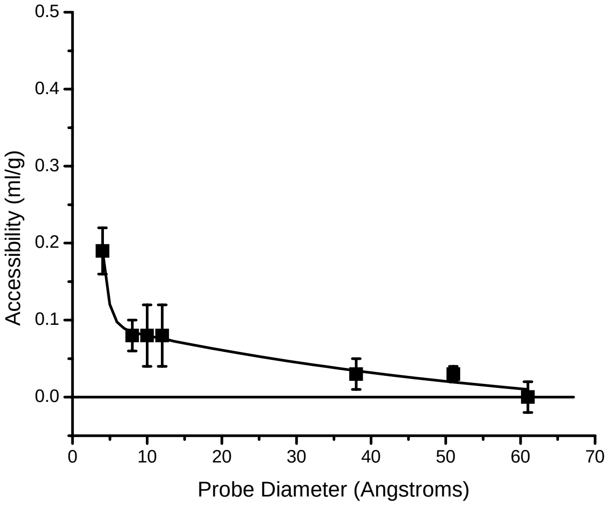

Figure 9: Accessibility of the cell wall of acetylated Corsican pine (20.7% WPG)

It can be seen that the accessibility to the cell wall decreases as the level of modification increases. However, even at WPG levels where full protection against fungal occurs there is still a residual level of accessibility.

Solute exclusion allows for an alternative method for determining the fibre saturation point (FSP) of wood samples. This has been calculated by subtracting the accessibility of the largest probe from the accessibility for water for each of the samples studied (Forster 1998, Farahani, 2003). The results of this analysis are shown in Fig. 10.

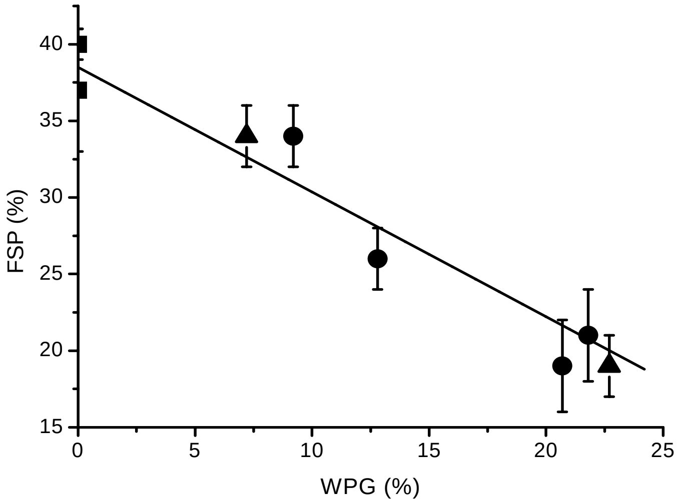

Figure 10: Fibre saturation point versus weight percentage gain for unmodified (squares), acetylated (circles) and hexanoylated (triangles) Corsican pine

The results from this study indicate that there is a linear relationship between FSP and WPG. Furthermore, the change in FSP is not related to degree of OH substitution. The straight line drawn through the data points is a calculation of the FSP based upon the hypothesis that volume occupied by reagent in the cell wall is thereby denied to water molecules (i.e. the reduction in FSP is a bulking phenomenon). The line was calculated from the molar volume values determined in Fig. 6, along with the selection of a value of the FSP of unmodified wood of 38.5%. It can be seen that there is a good correlation between the line and the data points, indicating that the reduction in FSP is indeed due to bulking of the cell wall by the chemically bound acyl groups.

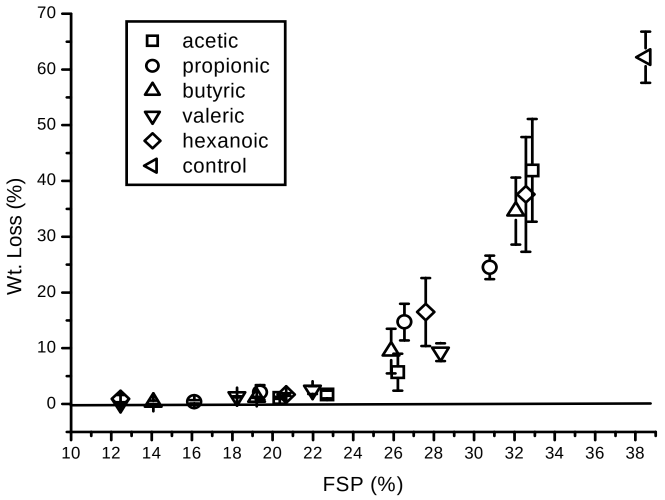

Figure 11: Plot showing weight loss due to decay by Coniophora puteana against calculated fibre saturation point for wood modified with a variety of anhydride reagents

By using the linear fit of WPG versus FSP of Fig. 10, the results from the decay experiments with Coniophora puteana can be plotted in terms of FSP (as determined by solute exclusion). This plot is shown in Fig. 11, above. This shows that weight loss due to decay approached zero at around 22% FSP. In a study of the decay of unmodified Scots pine (Pinus sylvestris) and Norway spruce (Picea abies) by Coniophora puteana, it was reported that the moisture content of the wood had to exceed 25% before decay would occur (Viitanen and Paajanen, 1988). The close correspondence between the threshold FSP values obtained in this work and those reported previously for unmodified wood are of interest.

3 Conclusions

This work has shown that decay resistance of wood chemically modified with anhydride reagents is related to the degree of bulking of the cell wall and not the level of OH substitution. Although the accessibility of the cell wall is greatly reduced by reaction with anhydrides, there is still some residual accessibility even at WPG's in excess of 20%. When FSP is determined using the solute exclusion method, the reduction in FSP is found to be proportional to WPG. The use of helium pycnometry has shown that determination of cell wall volume changes by the use of measurements of external dimensions of the wood is not possible. In addition, it has been shown that at low WPG's the lumen volume increases, but at higher WPG's the lumen volume decreases and the cell wall expands into the lumen. By determination of the actual volume that the acetyl group occupies in the cell wall it has been found that there is a clear relationship between volume occupied by the acyl group in the cell wall and reduction in FSP. Finally, when weight loss due to decay is plotted against FSP (as determined by solute exclusion), decay protection is found to occur at around 22% FSP. However, although there appears to be a clear relationship between FSP and decay resistance, it should be noted that this is also true for extent of bulking and decay resistance. Based on this work, a micropore blocking mechanism cannot be ruled out.

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MECHANICAL AND DURABILITY PERFORMANCE OF THM-DENSIFIED WOOD

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Keywords: Densified wood, mechanical, physical, durability, chemical analysis

Summary: *The mechanical, physical and durability properties of Thermo-Hydro-Mechanically post-treated densified wood were studied. The main objective was to investigate the chemical changes of wood constituents during THM post-treatments of densified wood and optimise the high steam treatment process to obtain stable compressed wood with improved hygroscopicity, strength and durability properties. For these purposes, a multiparameter reactor was developed and small wood specimens were densified and post treated under different moisture conditions at temperatures up to 200°C. The impact of different parameters of THM treatment process like temperature, RH and duration was examined through chemical and physical (DSC, GPC, RMN, capillary viscosimetery of cellulose, sorption isotherms), mechanical as well as biological tests on the products. In this article a brief description of the reactor and the process was illustrated and the experimental results were given and discussed.*

1 Introduction

The concept of moulding the wet wood by heat and compressive force is known since a very long time. Since 1886, the idea of densifying wood in the radial direction exists, Kollmann et al. (1968). At the early of the XX century, the effect of heat and water on wood plasticization was formulated. Soon after, the German exploited this distinctive feature of wood and it was the beginning of a new products, compression wood, called Lignostone, Kollmann et al. (1968). The product was compressed in a hot-press by heat treatment. During World War II a similar product was produced in US under the tradename of "Staypak", Seborg et al. (1945). This type of technology still exists and the production process is very simple. The wood pieces are placed between the heated plates of a hydraulic jack, and they are compressed up to 250 kg/cm2 in the radial direction. In this process the temperature is raised up to around 140°C during two hours. To eliminate the risk of explosion during processing, the wood pieces prior to densification should be stabilized to about 13% moisture contents. This process is an open system and there is no control on the wood moisture contents during processing and the product may be called Thermo-Mechanical (TM) densified wood. This treatment improves certain mechanical properties of wood but the transformed shape (compression deformation) produced during densification is unstable and it can recovered totally or partially by re-moistening and heating. Various wood products such as bentwood, densified wood, moulded solids wood, laminated compressed wood (Lignifol) suffer from this set-recovery or springback. In practice, to prevent the moulded wood from set-recovery different types of treatments chemically, physically or mechanical devices have been used. Heat treatments have been efficase but requires excessive exposure of specimen to high temperature i.e. 20 hours at 180°C or 5 hours at 200°C, Inoue et al. (1993). The use of steam has been recognized as being an efficient method for permanent fixation of transformed compression deformations as shown by Stamm (1946). Based to this work, Inoue et al. (1993) developed a technique by which compressed wood was fixed in a metal frame and steamed in the compressed state. The influence of steaming temperature and processing time on the set recovery for sugi were shown by Inoue et al. (1993).

Since 1997, densification of wood in a closed system has been developed in our Laboratory, Navi et al. (1997) and the permanent fixation of compressed wood was studied. Recently, we have studied the fixation of THM-densified wood using new multiparameter reactor, Heger et al. (2003). The densification and the post-treatment of wood were carried out in a new developed reactor. In this work the effect of post-treatment parameters, temperature and processing time on the fixation of the compressive set were studied under saturated and unsaturated vapor conditions.

2 Densification and set-recovery tests

2.1 THM Densification

Cylindrical spruce specimens with 40 mm in diameter and 50 mm height were densified in radial direction under saturated steam in a multiparameter reactor (Figs. 1&2) with a compression set of about 66%. The compression set value C = (R_o – R_c) / R_o where R_o and R_c are the thickness of the samples before and after compression respectively. A diagram showing the THM densification procedure is given in Fig. 3. It consists in two steps: at first the sample is heated with saturated steam until 140°C during 10 minutes (plasticization) and then densified under controlled displacement mode. The maximum load level was about 22 kN.

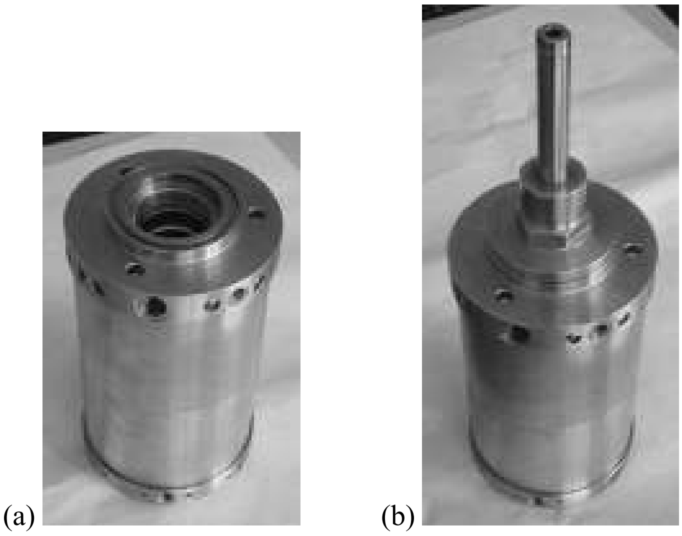


Figure 1: (a) THM reactor without piston and removable cover, (b) Overview of the THM machine

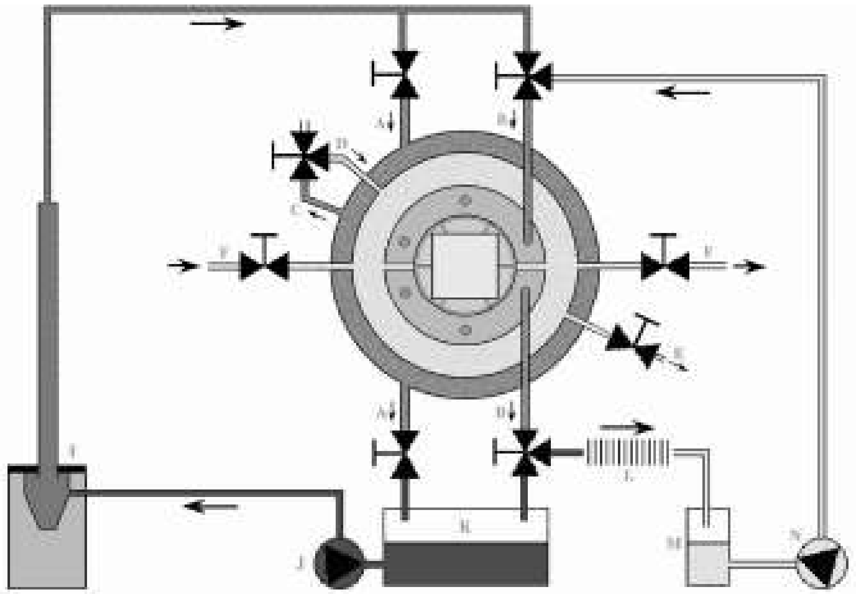


Figure 2: Schematic representation of the THM reactor showing in and out going streams to both saturated steam chamber and treatment chamber

2.2 Post-treatment

After densification, the samples have been post-treated under different temperature (140°C – 200°C). As the post-treatments have been carried out under saturated steam, the temperature of post-treatment was easily controlled by pressure. After densification the piston was stopped and maintained in the same position during the whole duration of post-treatment. At the end of post-treatment the reactor was purged by letting the steam go out of the treating chamber, the sample immediately cooled under 60°C and removed from the reactor.

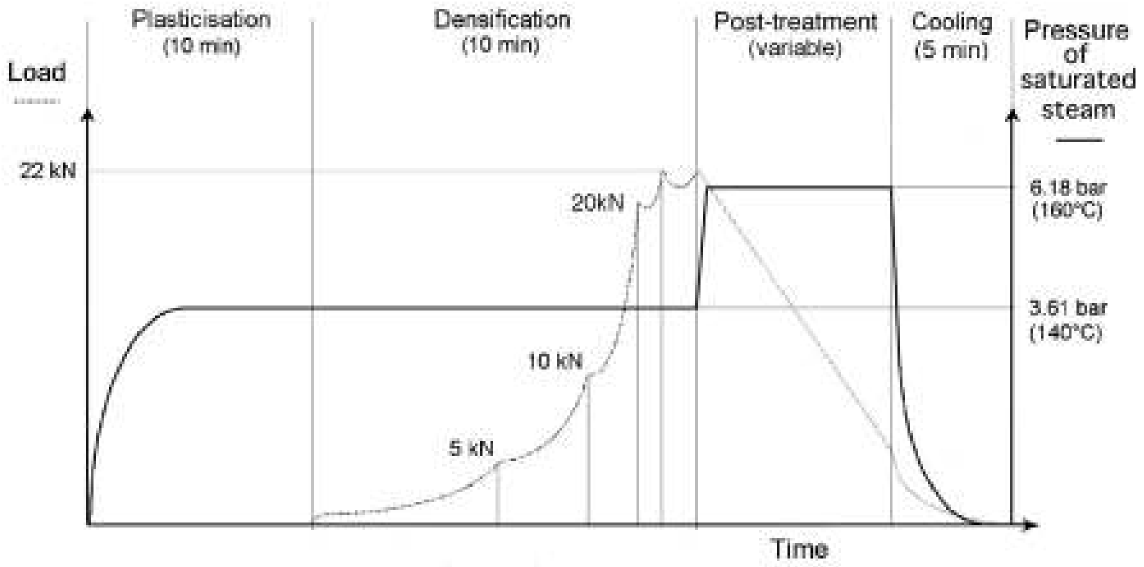


Figure 3: Diagram of the THM densification and post-treatment procedure, external loading and steam pressure variations

2.3 Set-recovery test

To examine the effect of THM post-treatment on fixation of compressed set deformation of densified wood a set recovery test was conducted on each specimen. This test consists of soaking-drying cycles of specimens in a water at 60°C and drying in a oven at 30°C. The set-recovery (R_l) is defined as: $R_l = (R'_o – R_c) / (R_o – R_c)$ where R'_o is the thickness of the specimen after the set-recovery test. In Fig. 4 we have presented the set-recovery of densified wood specimens post-treated by saturated steam at 180°C temperature for different processing times. When the set-recovery value of a densified specimen tends to zero, after several soaking-drying cycles, one could say that permanent fixation of compression set has been achieved. Fig. 4 shows that a complete fixation has been obtained for specimens that have undergone post-treatment at 180°C for about 20 minutes. A complete result of processing time to achieve a permanent fixation at different temperatures under saturated and unsaturated steam are given in Table 1.

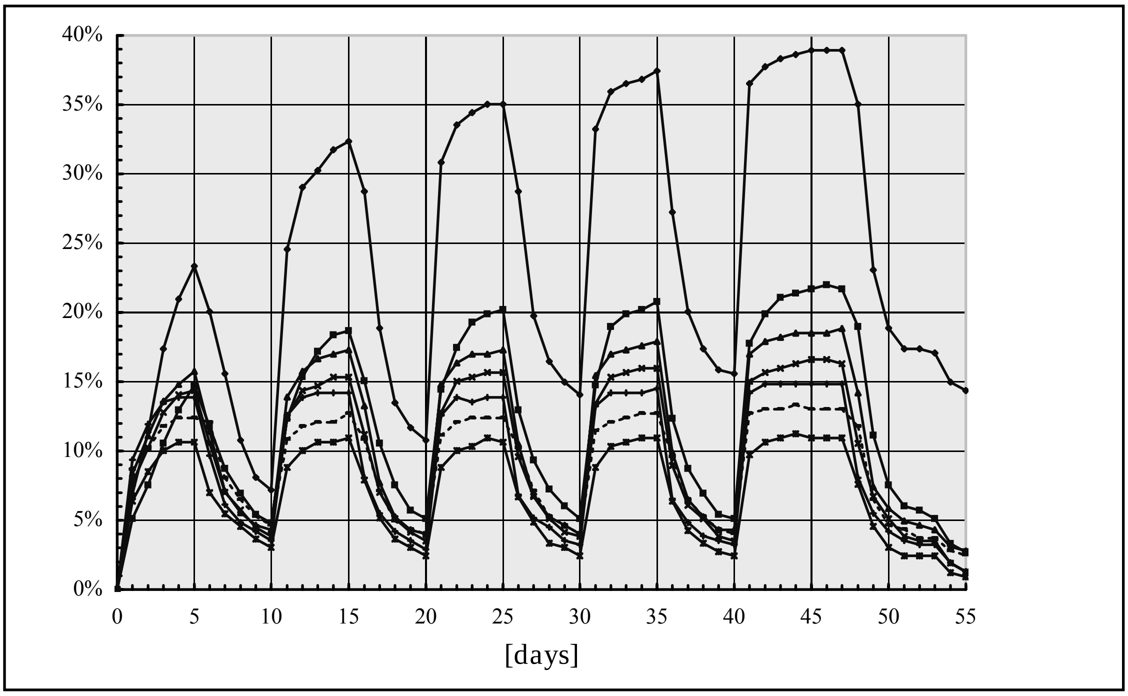


Figure 4: Set-recovery (soaking-drying cycles) curves of densified samples, post-treated by saturated vapor at 180°C under different processing times

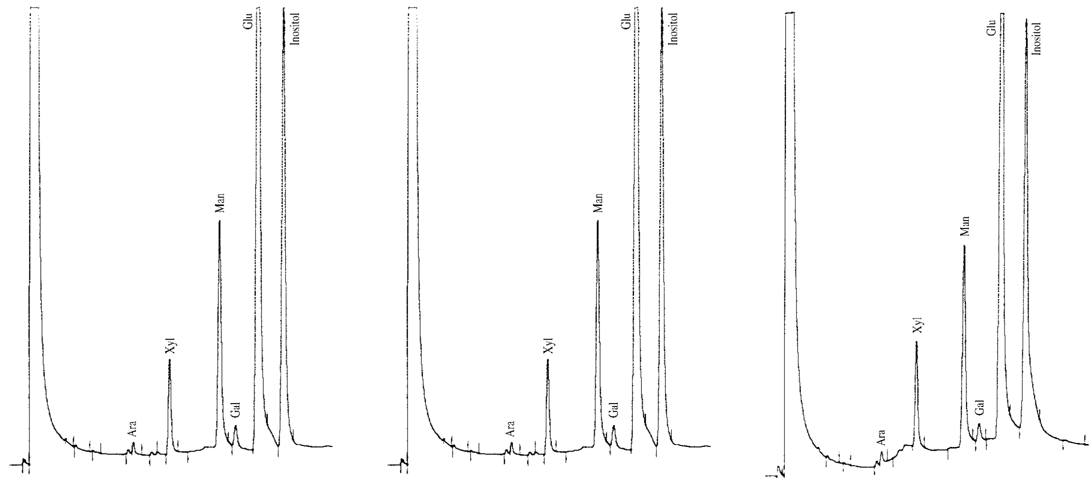
Table 1 Post-treatment processing time for complete fixation of transversal compression set in terms of temperature under saturated and unsaturated vapor

Relative humidity (h)	0	0.4	0.6	0.8	1
140°C	–	4 days 4 h	–	33 h 41 min	3 h 28 min
160°C	16 days 20 h	–	–	2 h 14 min	70 min
180°C	3 days 23 h	6 h 13 min	2 h 47 min	52 min	20 min
200°C	–	–	43 min	18 min	4 min 10 s

3 Experimental methods and results

3.1 Gel Permeation Chromatography (GPC) of polysaccharides

The pH of softwood varies from moderate (~4) to weak (~6) acid range (Sandermann, Rothkamm 1959, McNamara et al. 1970). Thus, during the post-treatments, the pH of water at 200°C is not 7 but 6 and due to the labile bond of acetyl groups on glucomannans acetic acid is produced. Under these acid conditions hemicelluloses are easily hydrolysed and eluted by water, especially xylans. The percentage of neutral monosaccharides (glucose, galactose, mannose, xylose and arabinose) during post-treatment has been measured before and after post-treatment by GPC. The picks are referenced by an internal standard: the inositol. These results are given in Fig. 5.



(a) (b) (c)

Figure 6: GPC spectra of monosaccharides. a) densified wood, b) densified and 180°C, 30min post-treated wood, c) densified and 200°C, 5 min post-treated wood

3.2 Glassy transition temperature (T_g) of lignin using Differential Scanning Calorimeter

As the lignin is a tridimentionnal reticulated amorphous polymer, its degree of reticulation and its Tg might vary during thermo-hydric treatments. So the Tg has been measured by DSC between -30 and 240°C for several post-treated samples. These results are shown in Fig. 7.

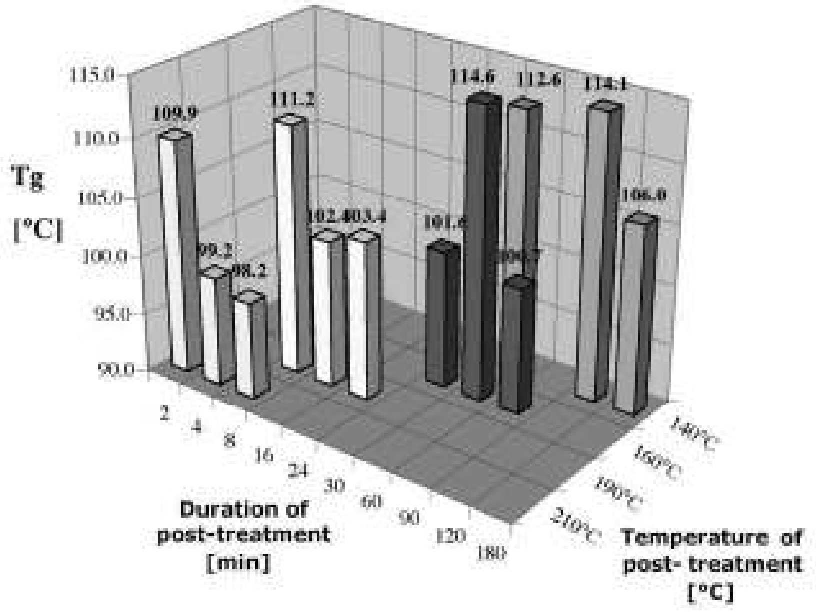


Figure 7: T_g of post-treated densified samples measured by DSC

3.3 Sorption isotherms

Thermo-hydric treatments are well known to decrease the hygroscopy of wood. Sorption isotherms have been carried out in order to quantify the absorption. The samples have been cut in thin chips. The following samples were used :

- natural wood sample
- densified wood sample
- densified wood sample post-treated during 3 hours at 140°C
- densified wood sample post-treated during 16 min at 180°C
- densified wood sample post-treated during 4 min at 200°C

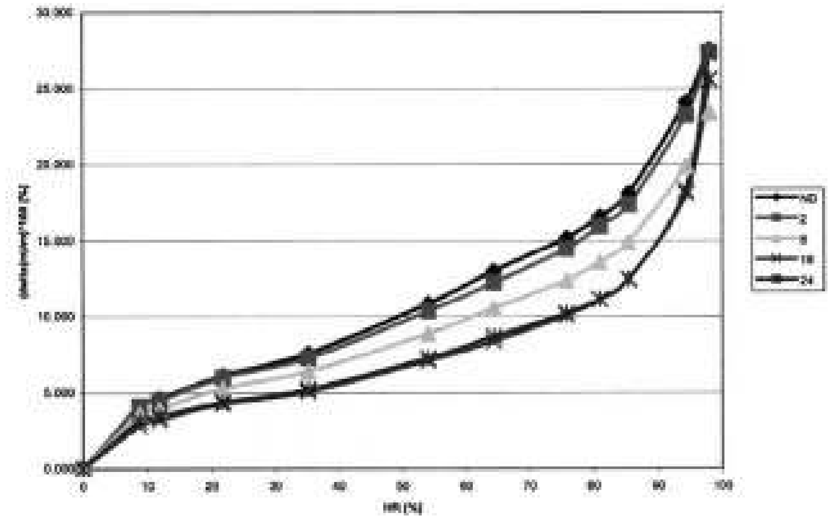


Figure 8: Sorption isotherms. ND: natural wood, 2: densified wood, 8: densified and 140°C, 3 hrs post-treated wood, 18: densified and 180°C, 16 min post-treated wood, 24: densified and 200°C, 4 min post-treated wood

The results of these sorption isotherms are presented in Fig.8. By using Dent sorption isotherm model, we have calculated the influence of post-treatment on the variation of –OH sites.

4 Mechanical properties

4.1 Shear stress resistance tests

Shear stress resistance tests have been carried out on three densified spruce samples. These samples had a squared section (c = 30 mm, h = 40 mm). The load has been longitudinally and parallel to the fibers axis. Table 2 gives the values of this test.

Table 2: Shear stress resistance of non densified and densified spruce sample

Spruce	Shear stress resistance [N/mm^2]
non densified samples	4.7 - 12.0
1st densified sample	121,4
2nd densified sample	107,2
3rd densified sample	103,4
mean value	110,6

4.2 Brinell hardness tests

The decrease of the porosity during the densification process makes the wood much harder in the radial and tangential directions. In order to measure the increase of hardness in these directions Brinell hardness (BH) tests have been carried out on spruce samples. Table 3 gives the results of this hardness test.

Table 3: Brinell hardness of natural and densified spruce samples

Spruce	tangentiel BH [N/mm2	radial BH [N/mm2]
non densified samples	12	12
1st densified sample	70,8	71,3
2nd densified sample	52,8	59,8

5 Discussion

To visualize the effect of densification on wood microstructure and also post-treatments on recovery of compression-set at cellular level, transversal micrographs were made by confocal microscope. From Fig (9a) once can recognize that by densification almost all lumens closes and the density of spruce samples increases from about 0.45 g/cm3 to 1.280 g/cm3. Densification of wood in the radial direction not only closes the lumens but also deforms the wood rays in a 'zigzag shape' as is illustrated in Figs. (9b, 9c). Comparison of microscopic micrographs of densified wood at cellular level before and after soaking-drying tests indicates that the lumens of earlywood cells opened up more widely than the late wood cells, Figs. (10a, 10b). In general, earlywood cells possess larger lumen with thin cell wall than latewood cells. Consequently during densification, earlywood cells undergo to bigger deformation and their microfibrils are subjected to larger internal stress than the latewood cells. During soaking-drying tests, when the physico-chemical bonds are weakened, those microfibrils with higher internal stresses return to their initial form faster. This opening seems to begin at the earlywood-latewood cells interface and progresses as the recovery increases Fig. (10c). It is not clear when latewood cells start to open, but it takes place after the opening of the earlywood cells. Obviously it depends on the distribution of the inner stress in the cells.

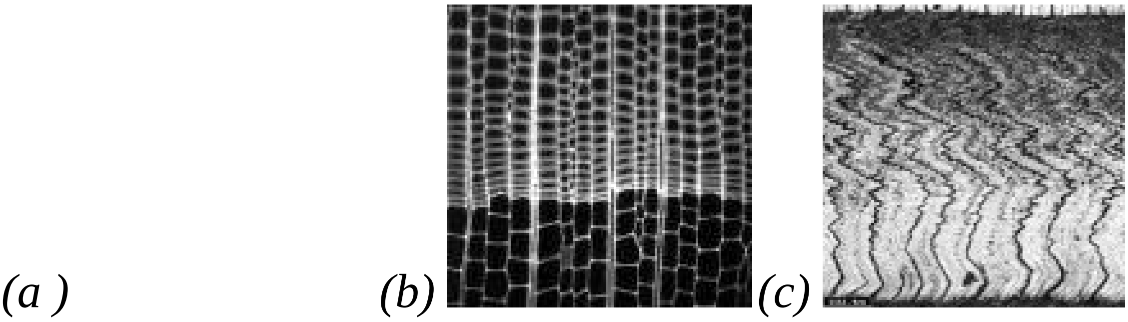


Figure 9: *a)* Residual pores of latewood cells and wood rays in densified wood. *b)* Wood rays in non-densified simple *c)* Densified spruce. Densification has transformed wood rays to a zigzag form shape

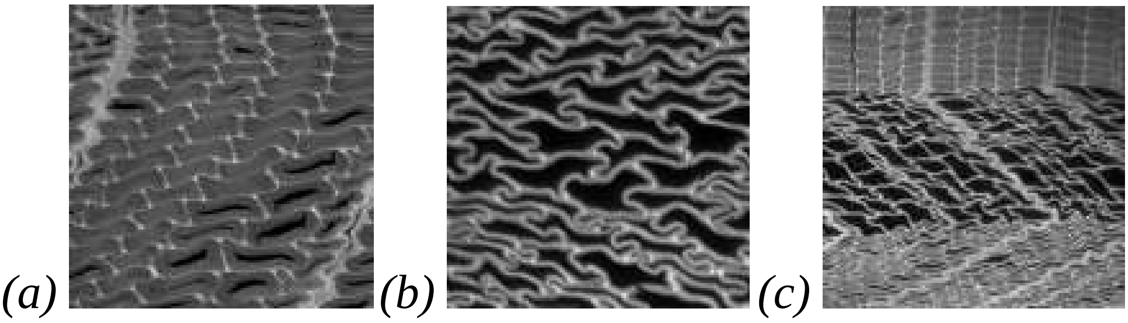


Figure 10: Partly recovered densified wood post-treated at 140°C and 4 hours processing time seen by confocal microscop after soaking-drying test: *a)* Slightly opened latewood cells. *b)* Widely opened earlywood cells. *c)* Widly opened latewood-earlywood cells interface

Table 1 presents the post-treatment processing time to achieve a complete fixation of compressed set under saturated and unsaturated vapor conditions. This table clearly indicates that the temperature of steaming and the vapor pressure have predominant effect on the fixation of the compressed set. Vapor in the range of our steaming temperature plays two important roles in the post-treatment stages: on one hand it contributes to the hydrolysis of the wood components and on the other hand it increases the kinetics of reaction and energy elastic relaxation.

The elastic-strain energy stored during densification in semi-crystalline microfibrils, in hemicelluloses and in the lignin is the main cause of compression-set recovery. The deformed shape of densified wood can remain unchanged as far as the deformed microfibrils are jointed to lignin and cellulose by hemicelluloses through strong covalent and numerous hydrogen bonds respectively, in spite of stored elastic strain energy.

The high-pressure steam conditions are well known to hydrolyse polysaccharides, especially the hemicelluloses, which are less chemically resistant than the cellulose of microfibrils, but to a lower extend the lignin too (Ito et al. 1998a, 1998b). During the post-treatment the hydrolysis reaction is catalysed by acetic acid and the glycosidic bonds are chemically degraded by cleavage of □-O-4 ether linkages (hydrolysis) to short molecules (oligosaccharides: mono-, di- and tri-saccharides) leading to a decrease of the hemicelluloses DP, which weakens the connection between the microfibrils and the lignin. During the abrupt decrease of pressure at the end of the post-treatment, a part of the oligosaccharides is eluted and removed from its original inter-microfibril location by water. The hydrolysis of the hemicelluloses during post-treatment was examined by CP/MAS 13C RMN and confirmed by the intensity increase of carbon 1 and 4 peaks of polysaccharides. Moreover, the elution of the hemicelluloses during post-treatment was confirmed by the decrease in intensity of Ara, Xyl, Man and Gal peaks in gas phase chromatography (Fig. 6).

Consequently, during post-treatment while the compression set of the sample is maintained by the piston, two complementary phenomena are taking place allowing the cellulose microfibrils to relax the inner stresses – 1) The binding between the microfibrils and lignin becomes weaker through hemicelluloses hydrolysis. 2) The hemicelluloses are removed by elution leaving room for the cellulose microfibrils to move.

Furthermore, DSC analysis of milled post-treated samples show an increase of Tg at the beginning of post-treatments (from 140°C to 210°C), followed by an abrupt decrease probably due to the degradation of lignin by hydrolysis of □- and □-aryl and alkyl linkages. Chemical degradations of both the hemicelluloses and the lignin (the matrix) result in decrease of cohesion between wood components, particularly between cellulose microfibrils and lignin. These degradations might be the reason of the brittleness of post-treated densified wood especially for post-treatment temperatures up to 180°C, where lignin is more severely degraded (Tg = 98.2°C after 8 minutes at 210°C).

From a mechanical point of view, one can consider post-treated densified wood as a new brand material as its mechanical properties are highly enhanced, especially the shear resistance and the hardness. The shear resistance increases from 4.7-12.0 N/mm2 to 110.6 N/mm2 and the Brinell hardness from 12.0 N/mm2 to 52.8-71.3 N/mm2 (Navi, Girardet, 2000). The reason for such an increase might be due to the closing of the lumens, which decreases the porosity from 60-75% to 10-15% and to the chemical modification of wood substances. Thus, the hydrous behaviour is improved. This improvement is partially due to the elution of hemicelluloses in which wood becomes less hydrophilic.

6 Conclusion

Densification of wood under saturated steam at 140°C improves the mechanical properties of wood and reduces wood hygroscopicity. Nevertheless the transformed compression-set during densification or moulding is unstable depending on the temperature and time of processing. It has been shown that THM treatment is an efficient process for permanent fixation of the compression-set.

During post-treatments the weakening of the bonds joining the deformed microfibrils to lignin by hemicelluloses allows the internal stresses to relax. During THM post-treatment hemicelluloses hydrolysis and the elution of the hemicelluloses lead to the weakening of the connection between microfibrils and the lignin. Therefore hemicelluloses hydrolysis plays a fundamental role in the relaxation of the stresses in the wood components. Complete fixation of compression-set can be obtained by THM treatment at 180°C for about 20 minutes or 140°C for about 3 hrs 28 minutes. This exponential decrease of processing time with increasing steam temperature, correspond to the exponential increase with temperature of the kinetics of the hemicelluloses hydrolysis.

The potential to reduce the post-treatment period has a strong impact on the practicality and economics of this technology. However, we have found a diminution of the mechanical properties of THM treated wood at temperatures higher than 180°C, which might be the maximum temperature practical for processing. Since our actual studies are carried out with only 4 cm of diameter cylindrical samples, further investigations are in progress to produce larger samples in order to give industry a commercial interest.

7 Acknowledgements

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DECAY RESISTANCE OF THERMO-HYDRO-MECHANICALLY DENSIFIED WOOD

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The paper is presented by [1*]

Abstract: *The decay resistance of Thermo-Mechanically (TM) and Thermo-Hydro-Mechanically (THM) densified wood was studied in comparison to TM- and THM- with additional steam-post-treatment (prepared by Laboratory for Construction Materials, Swiss Federal Institute of Technology Lausanne, Switzerland) and in comparison to untreated wood. The decay resistance tests were performed at the Federal Research Centre for Forestry and Forest Products (BFH) using pure cultures of basidiomycetes on malt agar plates. Significantly lower mass losses caused by Coniophora puteana was found for both modifications (TM and THM) when steam-post-treatment was applied after densification. However with the test fungus Poria placenta this effect was much less pronounced. Thus both modifications (TM and THM) with or without steam-post-treatment led to no better biological durability classification.*

NOTE:
Please note that there is another paper submitted by Dr. Parviz Navi describing the THM-densification process and the mechanical and chemical properties of THM treated wood.

1 Introduction

Densification of solid wood is an established process to improve selected mechanical and physical properties e.g. Young's Modulus, surface hardness, transversal shear strength and dimensional stability (Navi and Girardet 2000). There are mainly two types of densification processes used for solid wood: first one is based on filling the voids with substances, whereas the second one, which is adopted for this examination, compresses the voids in its radial direction by applying mechanical force and temperature. The combination of mechanical load and elevated temperature during the densification-process is conversant since the beginning of the 20th century. Kollmann (1936) named compressed wood out of a densification process under elevated temperatures in a hot-press "Lignostone", whereas Seborg *et al.* (1945) denominated densified wood marketed in the United States of America as "Staypack". Other authors have also reported on densified wood, compressed at elevated temperatures to improve its mechanical properties and dimensional stability (Morsing 1997, Navi and Girardet 2000).

Prior examinations on densified wood predominately deal with the problem of dimensional stabilisation and mechanical properties. In contrast, this study aims on the examination of biological properties of densified wood, in particular the change in natural durability due to a densification process with elevated temperatures and a hydro-thermal post-treatment to stabilise the compression set.

2 Material and Methods

Specimens of $8x8x35mm^3$ cut out of spruce (*Picea abies* L. Karst) and pine sapwood (*Pinus sylvestris* L.) were used in biological tests to determine the resistance against basidiomycetes on malt agar according to EN 113 (1996). The spruce specimens were tested untreated and differently densified. The number of specimens and the densification parameters are listed in Table 1.

Table 1: Densification parameters and number of specimens used in tests according to EN 113 (1996); abbreviations for the differently densified materials in brackets

Species	post treatment	non-densified	Thermo-Mechanical densification		Thermo-Hydro-Mechanical densification	
Spruce	none	36	36	(TM)	36	(THM)
	180°C / 30 min	-	36	(TM+)	36	(THM+)
Pine	none	54	-		-	

All densified specimens were densified at 140 °C for 70 minutes, the percentage of densification was approximately 67%. Post treatment was carried out at 180 °C, total time of densification plus post treatment was 110 minutes.

The test of resistance to basidiomycetes on malt agar was carried out according to a modified EN 113 (1996) test with miniblock specimens (8x8x35mm³) in Petri dishes. The differently densified specimens as well as untreated spruce and pine sapwood controls were incubated for 12 weeks. Following strains were used for the study: Coniophora puteana = (Schum.:Fr.) P. Karsten Ebw. 15; BAM 1/1995, Poria placenta var. Monticula = (Fr.) Gilbertson et Ryv. FPRL 280 BAM, 8/1997.

To assess the grade of durability, the relative durability was calculated as the quotient of mass loss of the modified material and untreated pine sapwood controls (x-value, EN 350-1; 1994), as is normally done for the classification of natural durable timber.

3 Results

3.1 Biological tests with *Poria placenta*

All tested densified specimens showed a slight decreased percentage mass loss by *Poria placenta* compared to untreated spruce references (Figure 1). The highest mass loss was found for untreated spruce references with 36% followed by the untreated pine sapwood controls and virulences with approximately 27%. No significant differences in mass loss were determined for TM (ca. 27%), TM+ (ca. 20%) and THM (ca. 23%) specimens. The slightest mass loss with approximately 17% was found for THM+ specimens.

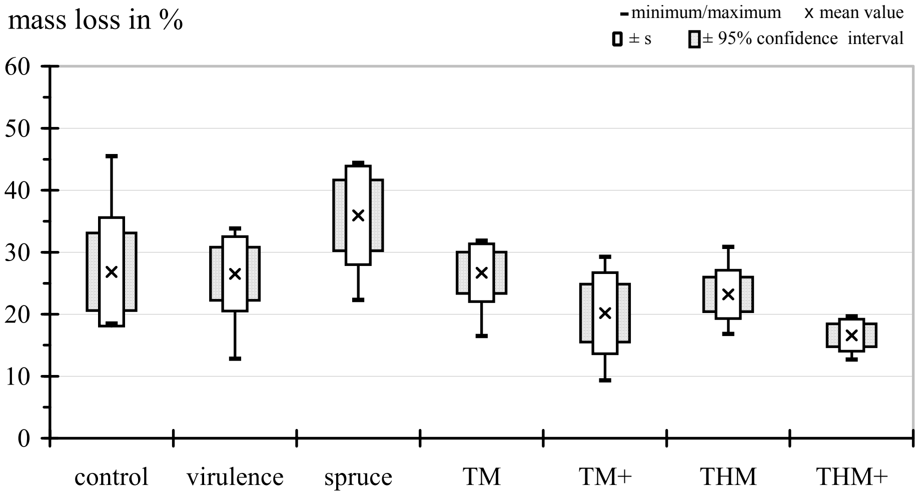

Figure 1: Corrected percentage mass loss after fungal attack by *Poria placenta*

An increase in mass loss of the densified specimens caused by *Poria placenta* compared to untreated references and controls was found, if the absolute mass loss was taken into consideration (Figure 2).

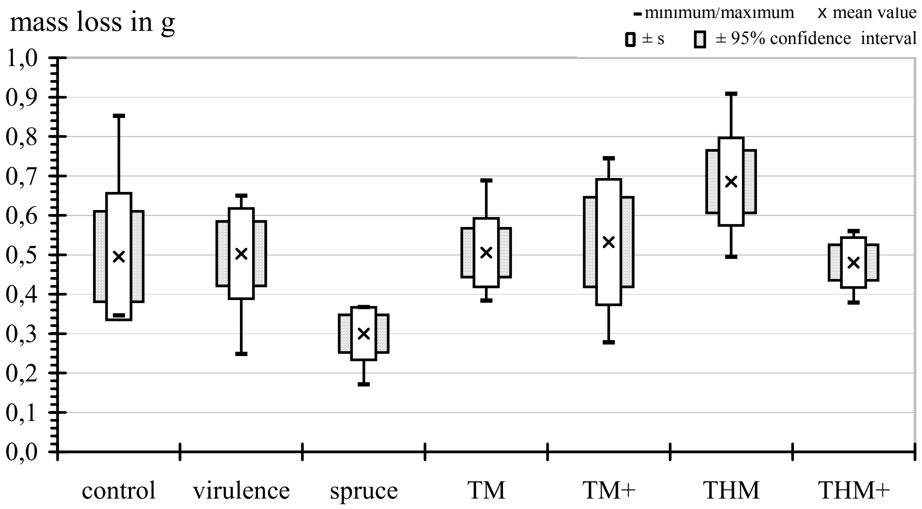

Figure 2: Corrected absolute mass loss after fungal attack by Poria placenta

An absolute mass loss of approximately 0,5g was caused by *Poria placenta* on untreated pine sapwood controls and virulences. The untreated spruce references showed an absolute mass loss of 0,3g. Obviously higher mass loss showed the TM and THM+ specimens with approximately 0,5g. Slightly higher was the average absolute mass loss of TM+ specimens with 0,53g, significantly higher the determined mass loss of THM with approximately 0,69g on average.

All average values of mass loss and moisture content of the specimens after 12 weeks of incubation as well as the calculated x-values and classes of natural durability according to EN 350-1 (1994) for tests with *Poria placenta* are listed in Table 2.

Table 2: Average values of mass loss, moisture content, x-values and assigned classes of natural durability according to EN 350-1 (1994) out of biological tests with Poria placenta

Material	mass loss		MC	x-value based on		durability class	
	in %	in g	in %	%	g	%	g
Untreated spruce	36,0	0,300	50,3	1,34	0,61	5	4
TM	26,7	0,505	52,6	0,99	1,02	5	5
TM+	20,2	0,532	36,6	0,75	1,07	4	5
THM	23,2	0,685	47,7	0,86	1,38	4	5
THM+	16,6	0,480	32,3	0,62	0,97	4	5
pine control	26,8	0,495	49,0	1,00	1,00	5	5
Virulence	26,5	0,503	49,4	0,99	1,01	5	5

The classification to classes of natural durability according to EN 350-1 (1994) pointed to a different grading of the densified specimens depending on the use of the absolute or percentage mass loss. If the percentage mass loss was taken, class 5 (not durable) was assigned for TM densified specimens and class 4 (slightly durable) for the TM+, THM and THM+ specimens. Untreated spruce, pine controls and virulences obtained the classification as not durable (class 5).

If the absolute mass loss was used as the basis for the calculation, all densified specimens and the untreated pine controls and virulences were classified as not durable (class 5). In this case, untreated spruce references were classified as slightly durable (class 4).

3.2 Biological Tests with *Coniophora puteana*

The highest mass loss was found for untreated spruce references with 53% and TM densified specimens with approximately 52% followed by the untreated pine sapwood controls and virulences with approximately 42% respectively 43% (Figure 3). THM specimens showed an average mass loss of 38%, significantly lower was the mass loss of TM+ and THM+ densified specimens with 5% respectively 3%.

The tested TM specimens showed no difference in percentage mass loss caused by *Coniophora puteana* compared to untreated spruce references. In contrast, the found percentage mass loss of the TM+, THM and THM+ specimens was significantly decreased compared to untreated spruce references. The post-treatment of the TM+ and THM+ densified specimens even resulted in an significantly decreased mass loss compared to untreated pine controls and virulences.

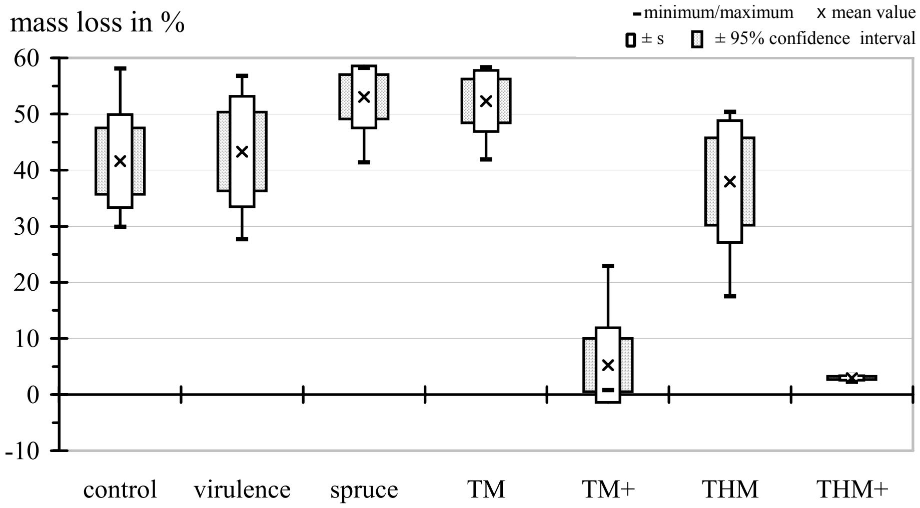

Figure 3: Corrected percentage mass loss after fungal attack be *Coniophora puteana*

Differences were found regarding the ratio of mass loss of spruce references and densified specimens, if the absolute mass loss after fungal attack by *Coniophora puteana* was taken into consideration, compared to the percentage mass loss (Figure 4). If so, TM and THM had the highest mass loss of approximately 1,1g, whereas the untreated spruce references showed only an average mass loss of ca. 0,4g. The average mass loss of TM+ and THM+ with approximately 0,1g in both cases was significantly lower. An average mass loss of ca. 0,8g was found for untreated pine controls and virulences.

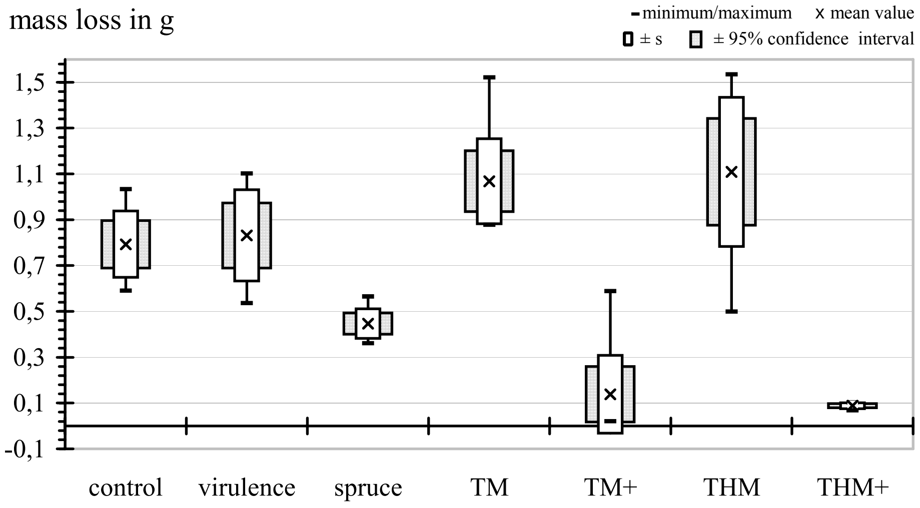

Figure 4: Corrected mass loss after fungal attack by Coniophora puteana

All average values of mass loss and moisture content of the specimens after 12 weeks of incubation as well as the calculated x-values and classes of natural durability according to EN 350-1 (1994) for tests with *Coniophora puteana* are listed in Table 3.

Table 3: Average values of mass loss, moisture content, x-values and assigned classes of natural durability according to EN 350-1 (1994) out of biological tests with Coniophora puteana

Material	mass loss		MC	x-value based on		durability class	
	in %	in g	in %	%	g	%	g
untreated spruce	53,0	0,447	67,2	1,27	0,56	5	3
TM	52,3	1,068	50,1	1,26	1,35	5	5
TM+	5,3	0,138	31,0	0,13	0,17	1	2
THM	38,0	1,109	40,0	0,91	1,40	5	5
THM+	3,0	0,088	28,4	0,07	0,11	1	1
Pine control	41,6	0,793	56,2	1,00	1,00	5	5
Virulence	43,3	0,831	44,1	1,04	1,05	5	5

Analogue to the tests with *Poria placenta,* the classification of the densified specimens to classes of natural durability according to EN 350-1 (1994) pointed to a different grading depending on the use of the absolute or percentage mass loss. If the percentage mass loss was taken, class 5 (not durable) was assigned for TM and THM and class 1 (very durable) for the TM+ and THM+ densified specimens. Untreated spruce, pine controls and virulences obtained the classification as not durable (class 5).

If the absolute mass loss was used, TM and THM as well as untreated pine controls and virulences were classified as not durable (class 5). If so, TM+ was durability class 2 (durable), THM+ was assigned to class 1 (very durable) and untreated spruce references were classified as moderately durable (class 3).

4 Discussion and Conclusions

The differently densified wood specimens showed an dissimilar performance in biological tests subjected to the post-treatment and test fungi used. With respect to the found mass loss after fungal attack, a first differentation regarding the densification process parameters has to be done. The conventionally densified TM and THM specimens exhibited a deviant performance in biological tests in comparison to the post-treated TM+ and THM+ specimens.

In the case of the TM and THM samples, both tested fungi caused an average percentage mass loss equal to the mass loss found for untreated references. On the other hand the absolute mass loss of the TM and THM densified specimens was even significantly higher compared to the references. In contrast, a different behaviour of the post-treated specimens (TM+ and THM+) was found. *Coniophora puteana* caused a significantly lower percentage as well as absolute mass loss compared to references, whereas *Poria placenta* entailed percentage mass loss in a range of the mass loss caused on TM and THM specimens. The absolute mass loss after attack by *Poria placenta* was even increased compared to the references.

In general, *Poria placenta* was able to cause a higher mass loss on the densified material than *Coniophora puteana*. This is explained by the fact, that *Poria placenta* is known as being critical for thermally modified wood. Especially the TM+ and THM+ densified specimens have to be rated as thermally modified, due to the hydro-thermal post-treatment at 180°C. Leithoff and Peek (1998) as well as Kamdem et al. (1999) have reported of high mass losses of thermally modified wood caused by *Poria placenta,* whereas Dirol and Guyonnet (1993) as well as Militz and Tjeerdsma (2000) reported on mass loss of 3% or less after fungal attack with *Coniophora puteana*. Therefore *Poria placenta* is regarded as critical for thermally modified wood and taken to assess the grade of durability according to EN 350-1 (1994). If so, TM specimens are classified as durability class 4 (slightly durable) whereas TM+, THM and THM+ are durability class 5 (not durable) if the percentage mass loss is taken as the basis for calculation. Considering the absolute mass loss as the basis for calculation, all densified materials are durability class 5 (not durable). Nilsson and Daniel (1992) have also mentioned such differences and suggested to take the absolute mass loss for expressing decay rates and durability.

The results of this examination circumstantiate, that the densification, regardless if carried out either as a conventional densification with applied mechanical force and elevated process-temperature or with an additional hydro-thermal post-treatment, are not suitable to improve the naturally durability by compression of solid wood.

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DURABILITY OF HEAT-TREATED WOOD

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Key words: Heat-treatment; thermal treatment, durability; decay; mould

Abstract: *The durability of heat-treated pine (*Pinus sylvestris*) and spruce (*Picea abies*) was investigated.*

The heat treatment was carried out according to a process intended for wood in above-ground end-uses and the durability against decay was tested in the laboratory in "terrestrial microcosm" (TMC) and in the field according to EN 252, and a ground proximity multiple layer method. The latter has been installed in Sweden, Germany and the USA (Hawaii) respectively. The resistance against discolouring micro-organisms was tested in a specially designed "Greenhouse test hut" as well as in the field.

The heat-treated material has performed very well in the TMC test. No decay has yet been observed after two years exposure in the Swedish ground proximity test. The EN 252 stakes show a high rate of failure after two years exposure, but a microscopical analysis reveals no indication of decay. The high rate of failure is probably a consequence of the strength loss caused by the heat-treatment itself, enhanced by the subsequent wetting in the ground and further chemical degradation.

The tested material seems to be less susceptible to discolouring organisms than untreated wood but not as good as wood treated with copper-containing wood preservatives.

1 Introduction

During heat-treatment the properties of wood are changed. The durability is better than that of untreated wood, but the knowledge of heat-treated wood and its suitability for various end-uses where the durability is critical is still insufficient. Heat-treatment is also known to decrease the strength and the stiffness of wood. The objective with this study was to evaluate the durability of heat-treated wood against decaying and discolouring fungi. As comparison untreated and preservative treated wood were used.

Durability testing has been carried out in the laboratory as well as in the field according to various test methods. The tests in the laboratory have been carried out in unsterile soil in Terrestrial Microcosms (TMC). In the field, the stake test EN 252 has been used as well as a ground proximity multiple layer method (Häger 1979, Nilsson 1993). This method simulates exposure near and above the ground. A specially designed Greenhouse test hut (Terziev, Edlund 2000) has been used for testing the resistance against discolouring micro-organisms.

2 Material and methods

2.1 Test material

Spruce (*Picea abies*) beams and pine (*Pinus sylvestris*) beams from South Western Sweden were heat-treated. Out of these beams and from untreated beams from the same area test samples for durability tests were prepared.

In each trial, material as specified in Table 1 has been tested.

Table 1: Material tested.

Material	Specification
Heat-treated pine (*Pinus sylvestris*)	Intended for use class 3 (above ground)
Heat-treated spruce (*Picea abies*)	Intended for use class 3
Pine (*Pinus sylvestris*) control	Untreated pine sapwood
Spruce (*Picea abies*) control	Untreated spruce
Preservative-treated: Kemwood ACQ 1900	Retention 19 kg/m^3 (Nordic class AB, i.e. use class 3)
Preservative-treated: CCA – A (Tanalith CT 106)	Retention 8,8 kg/m^3 (Nordic class A, i.e. use class 4)
Preservative-treated: CCA – AB (Tanalith CT 106)	Retention 5,4 kg/m^3 (Nordic class AB, i.e. use class 3)

2.1.1 The heat-treatment process

The heat-treatment was carried out in Finland at Stora Enso Timber’s plant in Honkalahti. The maximum temperature was 220°C during five hours and the total process time was four days. The process was completed by conditioning so that the moisture content (MC) after the treatment was approximately 6 %. According to the producer this should correspond to the equilibrium MC at 20°C and 65 % relative humidity (RH). This level of treatment was classified as suitable for timber to be used for outdoor exposure above ground, i.e. hazard (use) class 3 according to EN 335-1.

2.1.2 Preservative treatment

The samples were vacuum-pressure treated to a retention corresponding to the Nordic class AB (above ground use).

2.2 Methods

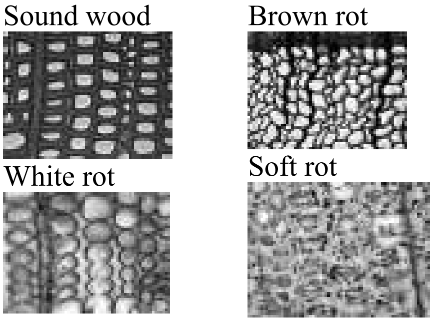
2.2.1 Terrestrial Microcosm (TMC)

Small stakes 5 x 10 x 100 mm were inserted to approximately 80 mm depth in containers with unsterile soil. Four different soils, each characterized by its dominating wood-destroying micro-organism(s) were used. There were 10 samples of each material (Table 1) in each soil. The TMCs were kept at 25°C and 85 % relative humidity during the time of exposure which was 12 months. The samples were dried at 103±2 C and weighed before and after exposure to calculate the weight loss.

Figure 1: Design of Terrestrial Microcosm TMC

Table 2.2. Origin of soils used and related dominating micro-organisms

Soil origin	Dominating micro-organisms
Simlångsdalen old field	Brown rot
Ultuna (Uppsala) test field	Soft rot and tunnelling bacteria
Forest	White rot and soft rot
Compost	Soft rot and bacteria



Sound wood

Brown rot

White rot

Soft rot

Figure 2: Sound wood and wood with different types of fungal attack

2.2.2 Field test EN 252

Ten stakes 25 x 50 x 500 mm of each material (Table 1) were installed in two test fields, the Simlångsdalen test field and SP's test field in Borås. Both test fields are located in South-Western Sweden. The latter has been prepared of soil from Simlångsdalen, compost and soil of unknown origin from the Borås area. Brown rot, white rot and soft rot all occur in that field. The Simlångsdalen old test field has been used since 1943 and was formerly agricultural land. This field is dominated by brown rot, but white rot and soft rot appear as well.

The stakes are inspected once a year and the extent of decay is graded according to the rating in Table 3.

By adding the index of decay for the stakes of each group and dividing the sum by the number of stakes, the average index of decay for each preservative and retention level is obtained. When all stakes in a group have failed (average index of decay = 100), the average life is calculated.

Figure 3: Detail of SP's test field in Borås.

Table 3: Grading system according to EN 252

Definition of condition	Rating	Decay index
Sound – no decay	0	0
Slight decay	1	25
Moderate decay	2	50
Severe decay	3	75
Very severe decay (stake rejected)	4	100

2.2.3 Ground proximity multiple layer field test

Each test unit consists of ten samples 22 x 95 x 250 mm that are stacked two by two in five crossed layers, with the bottom layer on the ground. To avoid weed growth the ground was covered with a geotextile, permeable for micro-organisms.

Tests according to this method are carried out at SP's test field in Borås, at the BAM in Berlin and at an American test site in Hilo, Hawaii. The latter has been chosen to get faster information on the performance of the samples tested. One stack of each material is exposed at each test site. In this trial the evaluation with respect to decay and attack by discolouring organisms is focussed on the top and bottom layers only. The grading systems are presented in Tables 4 and 5.

Figure 4: Ground proximity multiple layer test design

Table 4: Grading system for decay attack

Rating	Description	Definition
0	Sound	No evidence of decay
1	Slight-moderate attack	Visible signs of decay; small areas of decay, typically not more than 3 cm2.
2	Severe attack	Marked softening and weakening of the wood typical of fungal decay; distinctly more than 3 cm2 affected.
3	Failure	Very severe and extensive rot, members often capable of being easily broken.

Table 5: Grading system for attack by discolouring micro-organisms

Rating	Description	Definition
0	No discolouration	No evidence of discolouration caused by micro-organisms
1	Slight discolouration	Individual spots
2	Distinct discolouration	Groups of spots/streaks and/or patches of continuous staining
3	Total discolouration	Entire surface area

2.2.4 The Greenhouse test hut

Samples 22 x 95 x 200 mm were exposed in a greenhouse hanging approximately 10 cm above a berth of soil. By spraying the soil with water a high humidity was maintained in the greenhouse. Five samples of each material (Table 1) were exposed during 14 months. The samples were inspected each month and the discolouration was rated in six grades.

Figure 5: The Greenhouse test hut, designed for testing wood against discolouring organisms

3 Results

3.1 TMC

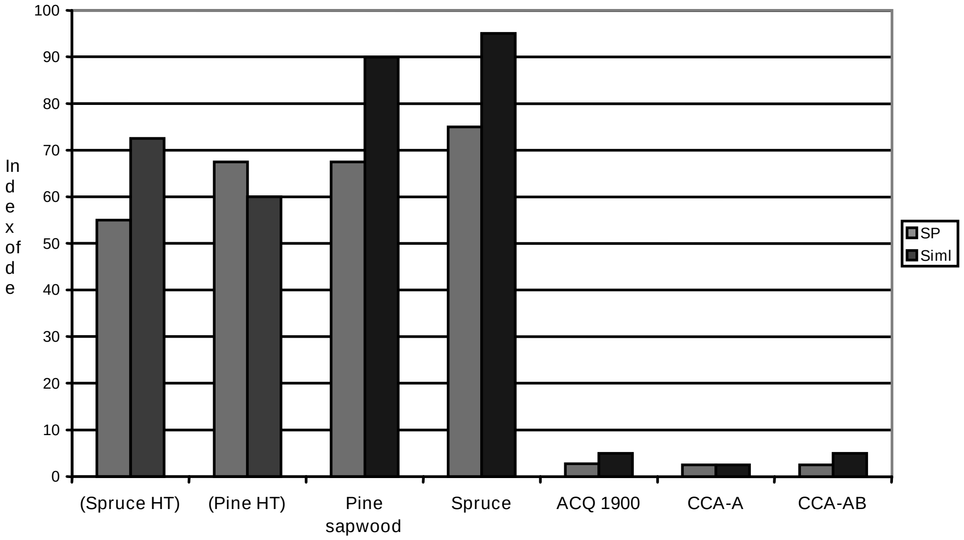
The results from the TMC tests are presented in Table 6. The mass loss is shown for the individual materials tested in each soil. For each material the average mass loss for all soils is also shown. The heat-treated material has performed very well in all soils and the average mass losses are lower than those of the preservative-treated wood.

Table 6: Mass losses for samples exposed 12 months in different soils. The mass losses are calculated as the average for 10 samples.

Material tested	Mass loss, % m/m in different soils after 12 months' testing			
	Simlångsdalen soil	Ultuna soil	Forest	Compost
Heat-treated pine (*Pinus sylvestris*)	2	4	3	5
Heat-treated spruce (*Picea abies*)	3	5	4	6
Pine (*Pinus sylvestris*) control	42	41	17	95
Spruce (*Picea abies*) control	37	38	17	71
Preservative-treated: Kemwood ACQ 1900	4	4	5	30
Preservative-treated: CCA-AB	5	16	3	43
Preservative-treated: CCA-A	2	9	1	25

3.2 EN 252

All stakes have been inspected after two years' exposure and the result is shown in Figure 6. The bars for the heat-treated wood are presented in different colours as the high rate of failure was not caused by decay as confirmed by a microscopical analysis, see 2.5 below.



Figur 6: Decay index for samples in ground contact after two years' exposure in Simlångsdalen (Siml) and Borås (SP).

3.3 Ground proximity test

Only the results from the Borås test site are reported here. The tests in Berlin and in Hilo, Hawaii are in progress and will be inspected and reported at a later stage.

After two years' exposure no decay was found in the heat-treated samples. Minor attack (rating 1) of decay was found in the bottom layer of the spruce controls. Thus, the presentation here is focussed on the results of the microbiological discolouration on the bottom and top layers respectively, see Figures 7 and 8. The upper surface of the top layer and the bottom surface of the bottom layer generally seem to be most susceptible to discolouring micro-organisms. The heat-treated wood has so far shown good resistance against discolouring micro-organisms, equal or better than the preservative references and far better than the untreated controls.

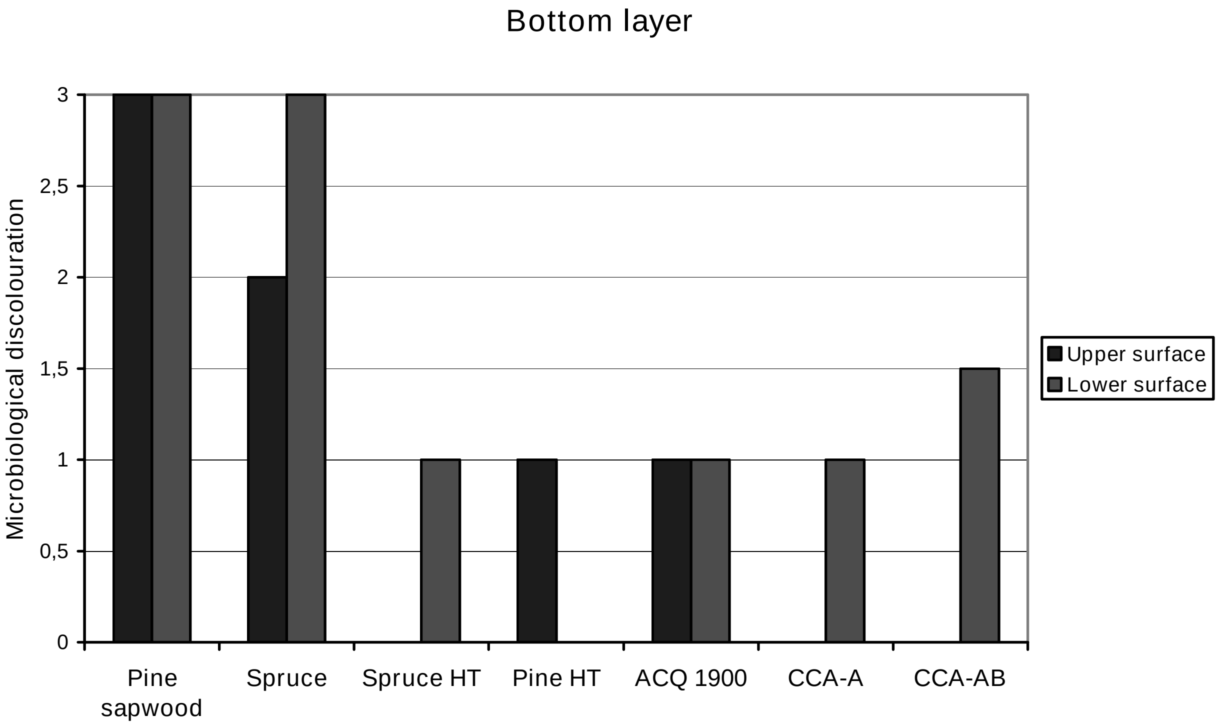


Figure 7: Microbiological discolouration on the upper and bottom surface respectively of the bottom layer. Average for the two samples in the layer.

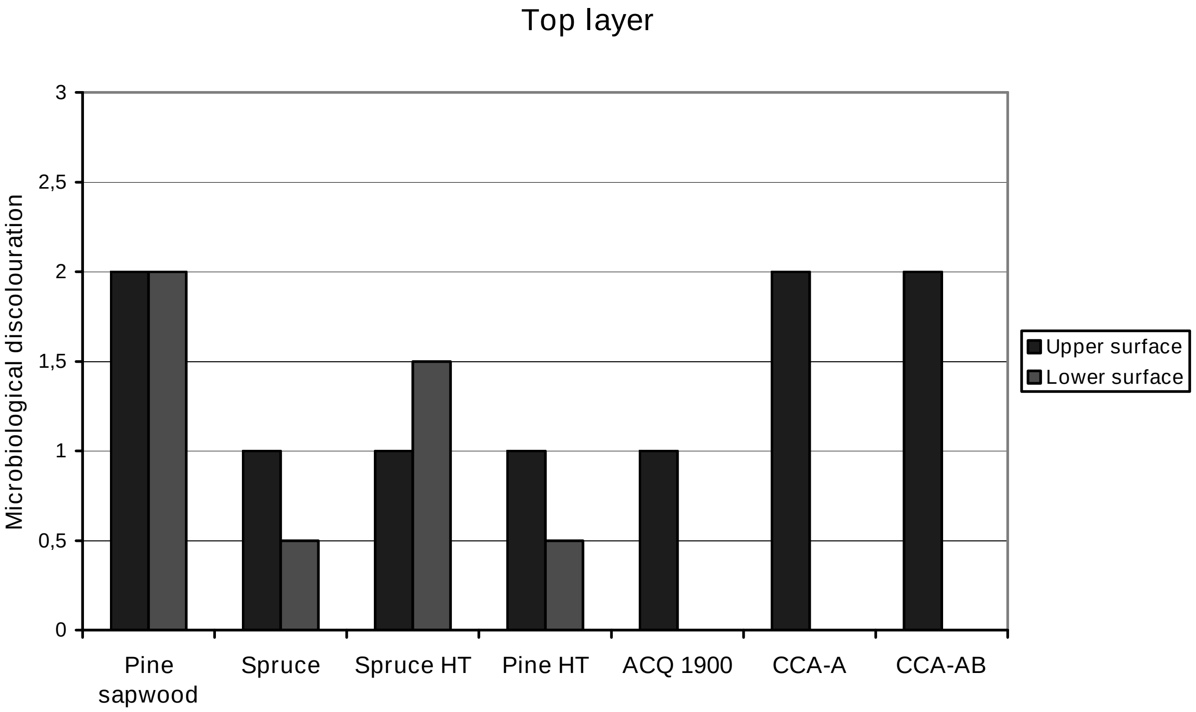


Figure 8: Microbiological discolouration on the upper and bottom surface respectively of the top layer. Average for the two samples in the layer.

3.4 The Greenhouse test hut

Figure 9 shows the development of the discolouration during the 14 months' test period. The preservative-treated references have performed better than the heat-treated material and untreated controls.

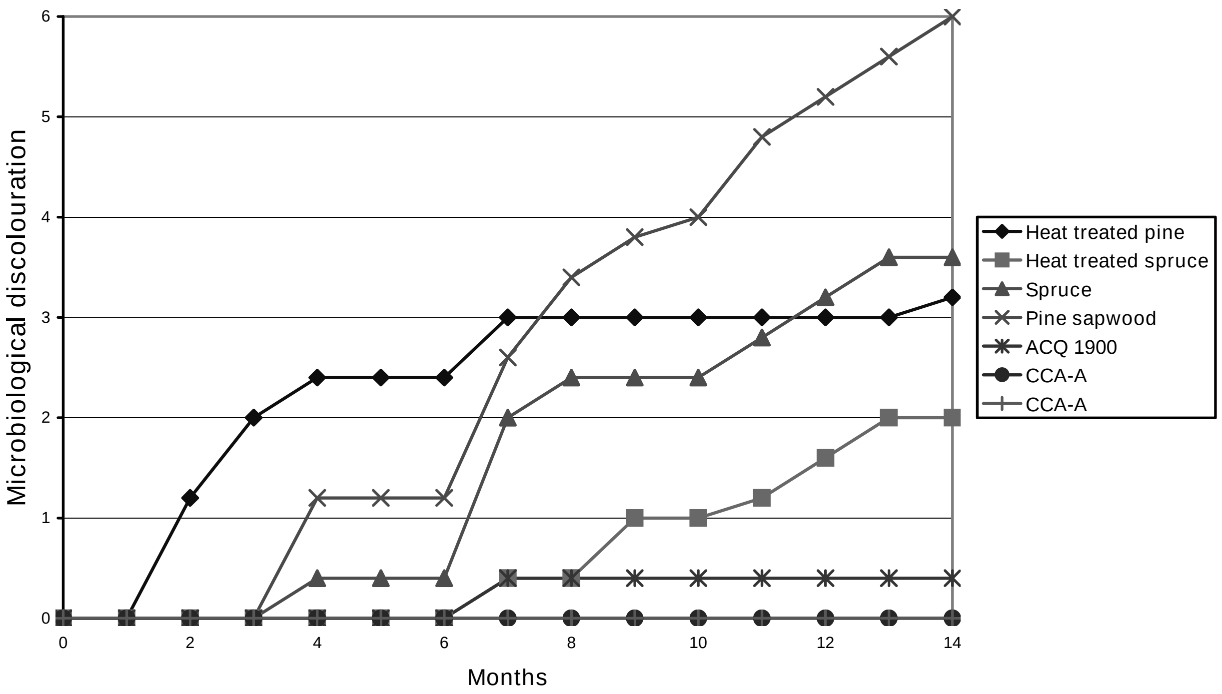


Figure 9: Microbiological discolouration of samples exposed for 14 months in the Greenhouse test hut

4 Discussion

4.1 Durability – decay fungi

In the decay tests in terrestrial microcosms, heat-treated pine and spruce show an extremely good resistance against all kinds of decaying fungi. The test is based on mass loss after exposure and the heat-treated samples had lower mass losses in all types of soils than samples treated with CCA or Kemwood ACQ 1900. The low mass loss found in this test is in agreement with other studies in the laboratory (Nordic Wood 2000), where heat-treated wood has been tested according to EN 113 and various soil-block tests. One would therefore expect good performance also in the stake test. However, after two years' testing in ground it is clear that heat-treated wood according to the specification in this trial is not suitable for end-uses in ground contact. There is a relatively high rate of failure, but a microscopical analysis carried out on these stakes shows no indication of decay. As the rating is based on the strength loss, the explanation for the high rating is probably that the strength loss as a result of the heat-treatment itself is further enhanced by the subsequent wetting in the ground and possible chemical degradation. This has to be confirmed by further studies.

No decay of heat-treated wood has yet been found in the ground proximity multiple layer field test exposed in Sweden.

The results obtained so far confirms that heat-treated wood from a durability point of view seems to be most suitable for above-ground end-uses, where strength is not a decisive property, but that more knowledge still is required to evaluate the performance of differently heat-treated wood in different end-use situations.

4.2 Durability – discolouring organisms

Evaluation of the results from the ground proximity multiple layer field test and the Greenhouse test hut has shown that heat-treated wood according to the specification tested is less susceptible to discolouring organisms than untreated wood but not as good as preservative-treated wood tested.

5 Conclusions

In this study no decay was found in the heat-treated wood after exposure in field and in laboratory. However, due to strength reduction from the treatment the heat-treated wood is suitable only for end-uses where strength is not a decisive property.

6 Acknowledgement

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