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COST ACTION E22
Environmental optimisation of wood protection

REVIEW ON HEAT TREATMENTS OF WOOD

Edited by
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Proceedings of Special Seminar
held in Antibes, France
on 9 February 2001

Preface

This report provides reviews on each of the four different major European heat treatment processes and properties of wood thermally modified after these processes:

the Finnish THERMO WOOD

the Dutch PLATO WOOD

the French RETIFICATION

the German OIL HEAT TREATMENT

All four modification treatments have in common that solid wood is subjected to temperatures close to or above 200°C for several hours in an atmosphere with low oxygen content. By this thermal modification some mechanical properties are reduced but the dimensional stability and the biological durability of wood is increased without adding outside chemicals / biocides to the wood. Therefore thermally modified wood is discussed as a new material for several applications.

The review papers on the state of the art of heat treatment in the European Union were presented by their respective authors at the COST E22 WG3 Special Seminar on Heat Treatments held at Antibes on 9 February 2001.

The NETWORK THERMAL MODIFICATION in WG3 of COST Action E22 is very grateful to Dr. Gérard Deroubaix and to his colleagues who assisted with organising and hosting the workshop. Also the support of the Scientific Secretary, Mr Günter Siegel of the COST Secretariat is greatly acknowledged.

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Contributions

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PRODUCTION AND CLASSIFICATION OFHEAT TREATED WOOD IN FINLAND

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INTRODUCTION

In Finland heat treatment of wood started in early 90´s when the first treatment plant was built to Mänttä. During the first ten years the interest against this new material has grown and today there are eight so called traditional heat treatment plants in Finland. The research work started nearly at the same time. Today producers co-operate at the research projects, especially which aim to quality control and classification of heat treated wood.

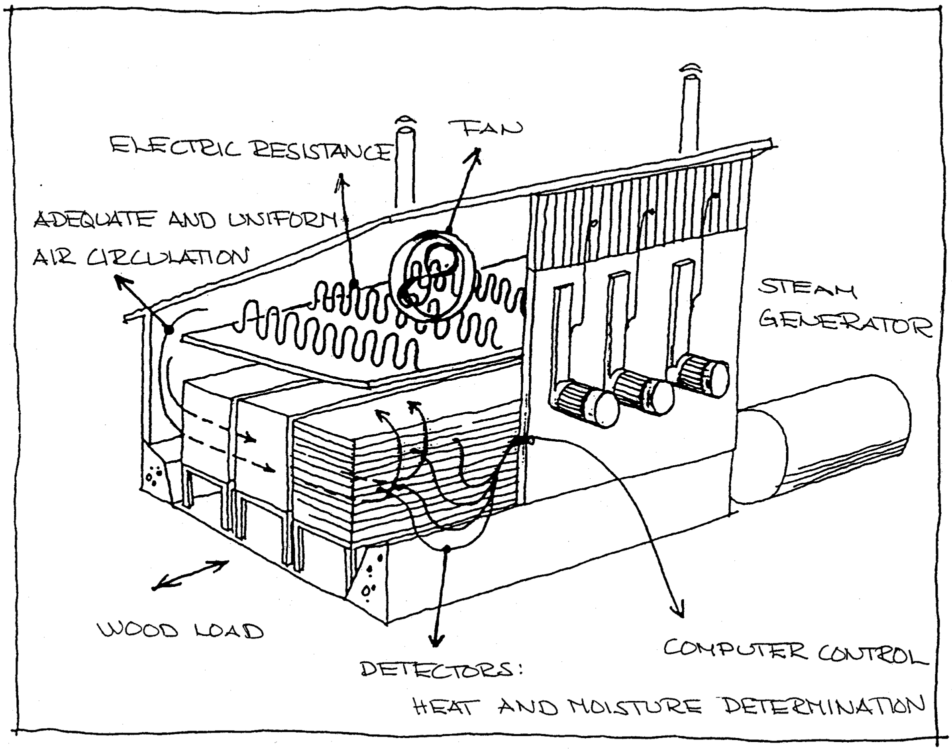
HEAT TREATMENT PLANTS AND EQUIPMENT

Today in Finland there are eight heat treatment plants in Finland and one quite big plant is under construction. The capacity of these eight plants is little under 50 000 m^3/year (year 2000) and the production is around 35 000 m^3/year (year 2000). The treatment plants are so called traditional heat treatment plants. That means that chemicals or pressure are not used. Only heat and water vapour.

The principle of a heat treatment plant is shown in picture 1. In this picture the heat is produced with electric resistance. In industrial scale heat is usually produced with thin oil. In Finland there is already one heat treatment plant which uses bark, sawdust and plane dust to produce heat.

The fan makes the air circulation adequate (10 m/s) and the wood load must be lathed so that the air circulation is equal. The steam generator produces the steam needed. Water steam is needed to prevent the wood from burning (air content must be under 3..5 %) and it also affects the quality of heat treated wood.

There are detectors around the chamber to measure the heat and moisture content of air and wood load and the heat treatment plant is controlled by a computer. Computer control adjusts the heat treatment process according to the initial data and data gathered from treatment process. Because of the automation the process can be recorded and rechecked later.



Picture 1. *Principle of a heat treatment plant*

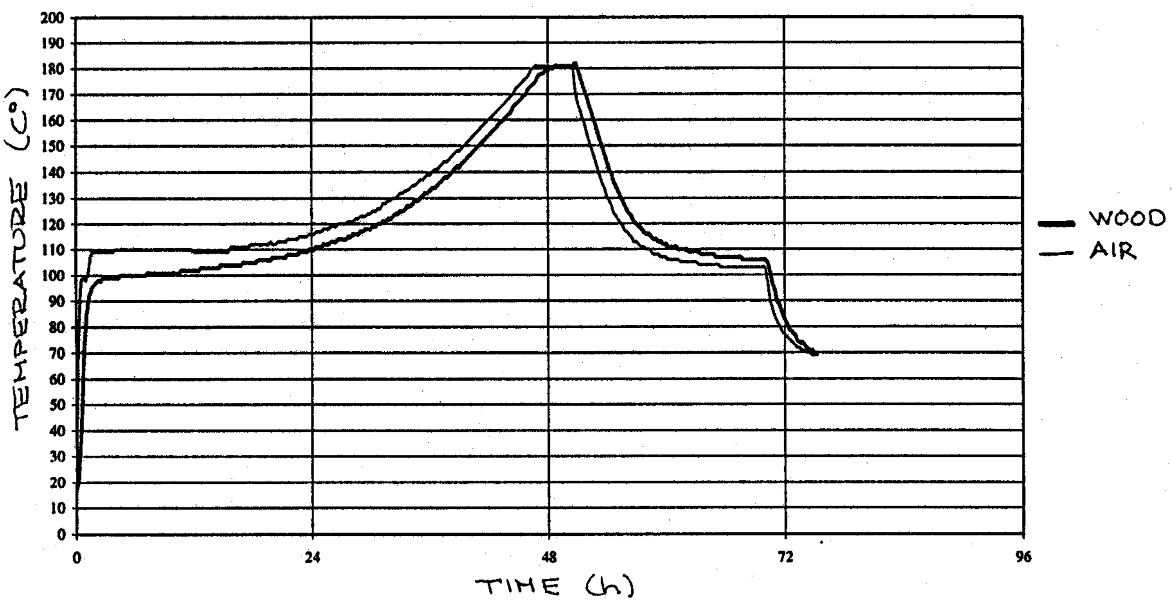
HEAT TREATMENT PROCESS

The process used for the Finnish treatment method can be divided into three different steps:

1) temperature rise period (preliminary warm up (→ 100°C) + kiln dry at hot temperatures if needed (100 - 150°C) + temperature rise period (150°C →)), up to 48 hours

2) actual heat treatment (constant temperature of between 150-240°C), 0,5…4 hours

3) cooling + stabilising, up to 24 hours



Picture 2 *Example of a heat treatment process.*

During the temperature rise period the temperature of the oven is raised to the temperature at which the actual heat treatment occurs. If the moisture content of the material is too high (> 10 %) before heat treatment a lot of splitting and colour differences may result. The kiln dry period can be integrated to the temperature rise period. The temperature rise period can take up to 48 hours.

The temperatures used for the actual heat treatment period range from 150 °C to 240 °C and during the actual treatment the temperature of the oven is kept constant. In heat treatment both the time and the temperature affect on the quality of the heat treated timber. The actual heat treatment takes from 0,5 hours to 4 hours.

During cooling and stabilising the temperature decreases to normal. Cooling and stabilising takes about 24 hours. During all these periods it is important that the temperature differential between the wood and the air is not too large. If the temperature difference is large the quality of treated wood is not good. It is also important that there is water vapour in the oven during whole treatment. The water vapour affects the quality of heat treated timber and it also acts as a protective atmosphere to prevent the material from burning.

MATERIAL

In Finland the most common wood species used for heat treatment are pine (Pinus Silvestris), spruce (Picea Abies), birch (Betula Verrucosa/Pubescens) and European aspen (Populus Tremula) although other species are also treated. The heat treatment process is different for each wood species and the final result is different because of the different chemical compositions and cellular structures. Usually softwoods are treated more strongly and hardwoods are treated more lightly. This is because of the different usage of the heat treated species.

The quality of wood which is going to be heat treated must be good. For example the knots are problem if they are dry and they drop out or crack. Also decayed wood may cause colour differences after treatment.

The final result is also affected by how the log is sawn. The conventional simple cut may result especially for softwoods a lot of peeling off and peeling of annual rings. In this case the annual rings are nearly horizontal to the surface. If the wood pieces are sawn so that the annual rings are at least in 45° angle to the surface the deformations will be smaller, the hardness of the surface will be stronger and the ”general looks” after heat treatment is better.

Usually softwoods are treated more strongly and are used in constructions which need moisture protection, for example in outdoor constructions. Hardwoods are treated more lightly and usually the most important property is the colour or good surface quality. Heat treated hardwoods are used indoors, for example in kitchen furniture, panelling and parquets.

Pine

Pine is a good material for heat treatments. Usually pine is used in outdoor constructions and because of this it is also treated quite hard. After treatment the knots of pine are mainly solid. The small and dry knots in butt logs may loosen when treated. The fresh knots in top logs stay solid and only in big knots there is some cracking. Especially for small dimensions there may happen some torsion because of big knots.
The problem in treating pine is that the resin comes out of the wood. It causes problems with heat treatment equipment if they are not cleaned between fillings. Also planing is difficult because of resin. But the good thing is that when the resin comes out many new uses can be found for heat treated pine.

Spruce

Spruce is also mainly used at outdoor constructions. Spruce is not that good material for heat treatment as pine. This is because of the knots and annual rings which loosen very easily. Already at low temperatures the fresh knots crack and loosen during treatment more often than for pine. Planing makes the annual ring rise up. Also the resin is problem for spruce as it was for pine.

Birch

The aim of treating birch is not the better decay resistance but the other benefits. Birch has had an important role for Finnish carpentry industrial as such and now when it is heat treated it gives more interesting opportunities. Birch is treated usually more lightly than pine and spruce. Already the chemical construction requires lower temperatures as for softwoods. The most important properties for treated birch are usually the colour or good surface quality. Birch is used indoors, for example in kitchen furniture and parquets.

The biggest problem with birch is twisting. The big knots, curves at logs and tension wood makes it difficult to predict how the sawn timber is going to behave when treated.

Aspen

The heat treated aspen is used indoors and especially as wood material for sauna furnishing. Problem is that the colour is not always equal and a lot of splitting may result. Splitting result especially when in wood material there is decay or when there is both sapwood and heartwood in one piece a lot of internal cracking.. But when the treatment is done right and the treated material is good the colour of aspen is beautiful after treatment and for aspen characteristic strong curling is decreased essentially.

USE

The improved characteristics of heat treated timber offer the timber product industry many potential and attractive new opportunities. The most important property compared to untreated wood is that the equilibrium moisture content of the heat treated wood is reduced and as a consequence of this shrinkage and swelling of the wood is also reduced. The best way of utilising heat treated timber is to make use of these improved properties. Wood species having no commercial value as such can be heat treated and in this way a new use found for these species.

Heat treated pine and spruce are mainly used for outdoor constructions, for example garden furniture, windows, doors and wall or fence boarding. When better weather and decay resistance is desired the temperatures used for the heat treatment process must be over 200°C. At these temperatures the strength properties also decrease, a factor which has to be taken into account. Although the rot resistance improves when the timber is heat treated strongly it is not recommended to use heat treated timber in ground contact.

Heat treated birch and aspen are used indoors. The most important property of heat treated birch and aspen is dimensional stability (due to moisture content changes) but also very beautiful but selectable shade of colours varying from light brown to almost black. For indoor use the treatment temperatures are under 200 °C. Birch and aspen are used for furniture, kitchen furniture, parquets, panelling and sauna furnishing.

THE EFFECT OF HEAT TREATMENT TO WOOD

The extent of the change in timber properties during heat treatment depends on

- the maximum temperature and the maximum length of the actual heat treatment period
- the temperature gradient
- the maximum length of the entire heat treatment
- the use and amount of water vapour
- the kiln drying process before the actual heat treatment
- the wood species and its characteristic properties

Temperatures over 150°C alter the physical and chemical properties of wood permanently. Heat treatment darkens the colour of the wood. It reduces the shrinkage and swelling of the wood and improves the equilibrium moisture content of the wood. At the same time the strength properties start to weaken. Very high temperatures improve the resistance to rot and also reduces the susceptibility to fungal decay.

The improved characteristics of heat treated timber offer the timber product industry many potential and attractive new opportunities. Also wood species having no commercial value as such can be heat treated and in this way new uses can be found for these species.

CLASSIFICATION

The classification of heat treated wood is based on standard EN 335-1 (Wood and wood based products - Definition of hazard classes of biological attack - Part 1: Solid wood). In the Finnish Wood Preserving Association's project the heat treated wood was classified into three heat treatment classes which are shown in table 1. In Finland heat treated timber is not recommended for use in hazard class 4.

Table 1 Heat treatment classes

Heat treatment classes	Hazard class EN 335-1	Situation in service	Description of exposure to wetting in service	Moisture content of untreated wood	Classification of impregnated timber
1	1	Above ground covered (dry)	permanently dry	permanently under 18 %	
2	2	aboved ground, covered risk of wetting	exposed to occasional wetting	occasionally over 20 %	
3	3	above ground not covered	exposed to frequent wetting	frequently over 20 %	AB (HC3/P8) B (HC3/P5)
	4	in contact with ground or fresh water	permanently exposed to wetting in contact with ground or fresh water	permanently over 20 %	A (HC4/P8)
	5	in salt water	permanently exposed to wetting by salt water	permanently over 20 %	M (HC5/P8)

Class 1

Very slight heat treatment. Mainly colour changes. Recommended to be used as untreated wood. Usage in constructions above ground covered and in conditions where the equilibrium moisture content of untreated wood will remain permanently under 18 % during service.

Class 2

Slightly heat treated timber. To be used in constructions above ground where is a risk of accidental wetting or condensation and the equilibrium moisture content of untreated wood occasionally exceeds 20 %. For example, kitchen furniture, parquets, windows and doors. The strength properties are slightly poorer as for untreated timber.

Class 3

Strongly heat treated timber. To be used in constructions above ground in situations where the timber will be continually exposed to the weather or to other sources of wetting such as condensation during service, but where the wood will not be in contact with the ground. The timber can be expected to have a moisture content of above 20 % repeatedly. To be used in constructions where the very good dimensional stability and lower moisture content are good properties. The strength properties have decreased.

QUALITY CONTROL

Every Finnish heat treatment plant inform that they have their own internal quality control. All the plants are computer controlled and they can recheck the process after treatment. Test pieces are taken out of the wood loads and check them if there are internal cracks or external cracks, is the general looks good, etc. Buyers have usually own standards according which the control is done. In Finland there is not yet external control or marking/labelling requirements.

R&D-PROJECTS

In Finland there are four different research centres which have studied heat treated timber. These research centres have carried out research work for the producers of heat treated timber but not very much for the general public. Finnish Wood Preserving Industry Ltd began to co-ordinate the research work among the producers and research centres in December 1997 and launched a project in which the aim was to assess the quality of heat treated timber in Finland. Another aim was to produce the basic values for a classification scheme and for quality control in heat treated timber.

This project is extended with a second project *Long term durability and painting of heat treated wood*. The objective of this project is to focus classification system, to write quality control instructions and painting instructions (schedule July 2000 - July 2003)

References

Kotiranta, R. 1995. Puun lämpökäsittelylaitteiston mitoitus. Espoo. 82 s. M Sc final work. (in finnish)

Mali, J., Koskela, K., Kainulainen, K. 1999. Lämpökäsitellyn puun ominaisuudet (The properties of heat treated timber). Espoo. 68 p. Final report of a project. (in finnish)

Möller, K., Otranen, L. 1999. Puun lämpökäsittely [Heat treatment of timber]. Institute of Environmental Technology, YTI julkaisuja - publication No 4. Mikkeli. 115 p. (in finnish)

Viitaniemi, P. Jämsä, S. 1996. Puun modifiointi lämpökäsittelyllä [Modification of wood with heat treatment]. Technical Research Center of Finland, VTT julkaisuja - publicationer 814. Espoo. 57 p. (in finnish)

HEAT TREATMET OF WOOD -
BETTER DURABILITY WITHOUT CHEMICALS

Dr. Saila Jämsä and Prof. Dr. Pertti Viitaniemi
VTT Building Technology

INTRODUCTION

Heat treatment of wood has an effect on wood's chemical composition and through that on the properties of wood. The effect of heat treatment on wood's properties was already known by our forefathers when heating the edges of fence poles to increase durability. In addition to better durability the advantages of heat treated wood are reduced hygroscopicity and improved dimensional stability.

Many methods of thermal modification of wood have been reported in the literature. The first articles concerning wood heat treatment were found in literature from 1920's. After this the method has been developed in Germany /1-4/, France /5,6/, Finland /7-9/ and Netherlands /10,11/.

The reason why heat treatment method has not been commercialised earlier is mainly because the processes were complicated in large scale production due to high temperature needed to get good biological durability. The problem has been wood's burning if shielding gas is not used. There have also been problems in getting the heat effect evenly inside the wood without surface charring. Also treatment decreased wood's strength making it too brittle for many applications.

VTT has developed together with Finnish industry an industrial scale wood heat treatment process, Thermowood T^{o}W. Pilot scale production of Thermowood T^{o}W has been started by the developers of the process, Enso Timber Ltd, UPM-Kymmene Timber and Valmet UTEC Ltd in Finland. One of the first applications in full scale can be found in the latticework of the headquarters of Mac Donalds in Helsinki.

VTT PROCESS

The Thermowood **T°W** is based on heating wood at high temperatures, 180 - 250 °C, by using a water vapour as shielding gas. While heating wood at temperatures over 200 °C wood undergoes a large number of chemical changes, like degradation of wood hemicelluloses. VTT method differs from other methods that these methods often use nitrogen as shielding gas and some processes are done under pressure.

The process has been divided into three parts. The first is the rise of temperature, the second is the treatment time and the third is the decrease of temperature /12,13/. Many things have an effect to total treatment time. It will depend on kiln size, kiln loading amount, dimensions of wood species and the temperature decrease during cooling.
When raising or decreasing the temperature a special adjustment system is used in order to prevent inside cracking. The temperature rise in the kiln is regulated by the wood's inside temperature. The difference between kiln and wood temperature is dependent on the dimensions of the wood specimens.

Raw material can be green or kiln dried wood. If the process starts from green wood the wood can be dried in a very quick steam drying process developed by VTT. Quick drying is possible because we do not have to care for the colour changes and because resins will anyway flow from the wood in heat treatment process.

During the heat treatment wood degrades and the degradation products are mainly acetic and formic acid, a small amount of phenolic compounds and other aromatic compounds and wood extractives. The gases which evolve during treatment are mainly carbon monoxide and carbon dioxide and methanol. This means that the equipment has some special requirements, it must be built of acid resistant stainless steel and it needs a washing system where the degradation products are absorbed.

WOOD PROPERTIES

The properties of heat treated wood are dependent on the treatment process: treatment time and temperature. Temperature has greater influence on many properties than time. Treatment in lower temperatures for longer times does not bring corresponding properties.

All heat treatment processes used today are not similar and so the properties of heat treated wood may also vary a lot. The colour of wood changes easily and colour changes do not tell anything about how much the other properties have changed compared to untreated wood.

VTT has clarified how the properties of Finnish pine , spruce and birch are modified by heat treatment. The following results were reached /7/:

Colour change into brown or dark brown

The colour of wood changes already in a mild heat treatment. The colour is not stable for UV-light.

Reduction of equilibrium moisture content of wood by 50 %

Heat treatment slowers water uptake and wood cell wall absorbs less water because of the decrease of the amount of wood's hydroxyl groups.

Reduction of shrinking and swelling 50 - 90 %

As a consequence of the reduced number of hydroxyl groups the swelling and shrinking are lower.

Improvement of biological durability

The biological resistance in a laboratory test EN 113 showed very good durability depending on the treatment temperature and time. In order to produce wood which has good decay resistance temperature over 220 ^{o}C has to be used. The treatment time in that temperature is at least 3 hours.

The resistance in ground contact is not acceptable.

The improved biological durability is based on the chemical degradation in wood components and formation of new compounds. The essential changes in wood chemistry are not exactly known.

Decrease of wood mechanical properties by 0 - 30 %

The higher is the treatment temperature the better is wood's biological durability. But at the same time more weakened are wood's mechanical properties. A negative consequence is that the wood becomes more brittle, and bending and pulling strength decrease by 10 % to 30 %. No changes were found in compression and impact strengths and surface hardness. As also the dry knots are loosened, the use of heat-treated wood in load-bearing constructions is restricted.

Other properties:

Heat conductivity decreases by 10 - 30 %
Resin flows out of wood
Wood looses some of it's weight (5 - 15 %)

HEAT TREATED WOOD AS MATERIAL

The performance of heat-treated wood differs from that of normal wood. Several considerations have to be kept in mind when using the new material.

As the wood has become brittle, sharp blades have to be used to prevent the wood from ripping. Wood dust coming from the process is very finely divided and dry. It can irritate respiratory tracks.

Heat-treated wood absorbs slowly water and water based glues, such as PVAc. That is why longer pressing times are needed, making the glueing more difficult. Suitable glues for heat-treated wood are resorcinol-phenol, polyurethanes, and other two-component glues. In assembling lower compression pressures should be used, because the material is brittle.

The darkened colour created in the process is not durable in UV-light, unless the surface is treated with UV-resistant coating. Normal painting processes present no problems, but when electrostatic painting is used, heat-treated wood requires extra moisturising.

APPLICATIONS

It is possible to tailor the process for different end uses. There are multiple correlations between the properties like dimensional stability, decay resistance and strength, so if e.g. small reduction in strength is sought the dimensional stability will be less pronounced.

Due to it's good weather resistance, thermowood is suited for outside applications such as external cladding, window frames and garden furniture. It is also suited for end uses where it is an advantage that resin has flown out of wood and heat insulation has increased like interiors in bathrooms and saunas. Because of the dimensional stability the treated wood gives better durability for coating. If the material is to be used for furniture and furnishing the process can be modified to give the desired degree of colour change.

REFERENCES

1. Kollman, F. and Schneider, A. 1963. Über das Sorptionsverhalten wärmebehandelter Hölzer. Holz als Roh- und Werkstoff 21 (3), p. 77 - 85.

2. Schneider, A. and Rusche, H. 1973. Sorptionsverhalten von Buchen- und Fichtenholz nach Wärmeeinwirkung in Luft und im Vakuum. Holz als Roh- und Werkstoff 31, p. 313 - 319.

3. Burmester, A. 1975. Zur Dimensionsstabilisierung von Holz. Holz als Roh- und Werkstoff 33, p. 333 - 335.

4. Giebeler, E. 1983. Dimensionsstabilisierung von Holz durch eine Feuchte/Wärme/Druck-Behandlung. Holz als Roh- und Werkstoff 41, p. 87 - 94.

5. Bourgois, J. and Guyonnet, R. 1988. Characterization and analysis of torrified wood. Wood Sci. Technol. 22, p. 143 - 155.

6. Dirol, D. and Guyonnet, R. 1993. The improvment of wood durability by retification process. The International Research Group On Wood Preservation, Section 4 - Process. 24 Annual Meeting, Orlando, May 16 - 21, 1993. 11 p.

7. Viitaniemi, P. and Jämsä, S. 1996. Modification of wood by heat treatment, VTT publications 814. Espoo, 57 p (In Finnish, English abstract).

8. Viitaniemi, P. 1997. Decay-resistant wood created in a heating process. Industrial Horizons. Espoo. VTT's Communications. p. 22 - 23.

9. Viitaniemi, P. 1997: Thermowood-Modified wood for improved performance. In: Edit.: Trätek 1997: Proceedings of the 4th Eurowood Symposium ”Wood-The Ecological Material” 22-23 September 1997, Stockholm/Sweden, Trätek Rapport No. P 9709084, p. 67 - 69.

10. Boonstra, M., Tjeerdsma, B. and Groeneveld, H. 1998. Thermal Modification of Non-Durable Wood Species. 1. The Plato technology: thermal modification of wood. The International Research Group On Wood Preservation, Section 4 - Processes. 29 Annual Meeting, Maastricht, June 14 - 19, 1998. 13 p.

11. Tjeerdsma, B., Boonstra, M. and Militz, H. 1998. Thermal Modification of Non-Durable Wood Species. 2. Improved wood properties of thermal treated wood. The International Research Group On Wood Preservation, Section 4 - Processes. 29 Annual Meeting, Maastricht, June 14 - 19, 1998. 10 p.

12. Pat.US-5678324, Method for improving biodegradation resistance and dimensional stability of cellulosic products. VTT, Viitaniemi, Pertti, Jämsä, Saila, Ek, Pentti and Viitanen Hannu. Appl.545791, 13.5.1994. Publ. 24.11.1994. 12 p.

13. Pat. EP-0759137, Method for processing of wood at elevated temperatures. VTT, Viitaniemi, Pertti, Ranta-Maunus, Alpo, Jämsä, Saila and Ek, Pentti. Appl. EP95918005, 11.5.1994. Publ.11.9.1995. 12 p.

HEAT TREATMENT OF WOOD BY THE „PLATO-PROCESS“

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GENERAL

Recent efforts on thermal treatment of wood have lead to the development of several treatment processes previously or presently introduced to the European market. This has resulted in the development of processes in Finland (Viitaniemi *et al.* 1994), in France (Weiland and Guyonnet 1997) and PLATO®-wood in the Netherlands. In the Netherlands a production plant was built and started its production in summer 2000. This plant is designed to treat initially 50.000 m^3 .

PLATO-PROCESS

The PLATO-process uses different steps of treatment and combines successively a hydrothermolysis step with a dry curing step. The impact of the hydrothermolysis in the PLATO-treatment results in the occurrence of different chemical transformations. One aim of this 2-step process is the use of the presence of abundant moisture in the woody cell wall during the hydrothermolysis. This provokes an increased reactivity of the cell wall components under comparable low temperature. In order to reach a selective degree of depolymerisation of the hemicellulose during the hydrothermolysis, relative mild conditions can be applied to limit unwanted side reactions, which can influence the mechanical properties negatively (Tjeerdsma et al 1998b).

The PLATO-process (Ruyter 1989, Boonstra *et al.* 1998) principally consists of two stages with an intermediate drying operation. In the first step (hydrothermolysis) of the process, green or air dried wood, is treated at temperatures typically between 160 °C - 190 °C under increased pressure (superathmospheric pressure). A conventional wood drying process is used to dry the treated wood to a low moisture content (ca. 10%). In the second step (curing) the dry intermediate product is heated again to temperatures typically between 170 °C - 190 °C.

The process time is depending on the wood species used, the thickness, form of wood etc., and looks in general:

1. thermolysis 4-5 hours
2. drying step 3-5 days
3. curing step 14-16 hours
4. conditioning 2-3 days

Depending on wood species and thickness of the material, these times can be shorter as well. The heating medium can be steam or heated air.

CHEMICAL TRANSFORMATION PROCESS

Relative mild thermal treatments of wood according to a two step process which lead to improved dimensional stability and improved timber performance were investigated by solid phase CP-MAS 13C-NMR to understand at molecular level the reasons for the improvements reported (Tjeerdsma et al. 1998). All the occurrences described appear to be the consequence of reactions which are known in wood chemistry. These are the formation of acetic acid deliberated from the hemicelluloses, which further catalyses carbohydrates cleavage, causing a reduction of degree of polymerisation of the carbohydrates. Acid catalysed degradation results in the formation of formaldehyde, furfural and other aldehydes as well as some lignin cleavage at Cα and O4 and believed to cause some aldehyde production from lignin units Cγ, all occurring in the first reaction step. Lignin autocondensation through the cleaved, positively charged benzylic Cα to form some methylene bridges presumably starts already to occur in this first phase. The increase in the number of free reactive sites on the aromatic ring of some lignin units already occurs in this phase but continues into the next.

In the second treatment step completion of the autocondensation of lignin is believed to occur through the formation of methylene bridges connecting aromatic rings. The aromatic nuclei sites are released by demethoxylation and through the cleaved, positively charged benzylic Cα. Reactions occur of some of the aldehyde groups formed in the first step phase with lignin aromatic nuclei sites to connect aromatic rings through methylene bridges.

The extend of these reactions is mild, but nonetheless they lead to an increase in cross-linking with consequent improvement in its dimensional stability and decreased hygroscopicity of wood.

Wood specimen of Beech (*Fagus silvatica* L.) and Scots pine (*Pinus sylvestris* L.) modified by a hydrothermal treatment process were analysed by means of Fourier Transform Infra Red spectroscopy (FTIR). The chemical transformation of the cell wall material was studied and associated with improved wood qualities. The results were published by Tjeerdsma et al 1999. For this purpose FTIR spectroscopy was used since this technique has been found appropriate to determine the intensity of specific bonds and functional groups within the polymeric structure. Cleavage of acetyl groups of the hemicellulose has been found to occur in the first treatment step under moist conditions and elevated temperature. This results in the formation of carbonic acids, mainly acetic acid. Most of the acetyl groups were found to be cleaved during the treatment of wood at a high temperature, whereas only partial deacetylation was found to occur at moderate treatment temperature. The concentration of accessible hydroxyl groups was measured by acetylation and found reduced after treating at high temperature. Esterification reactions were found to occur under dry conditions at elevated temperature in the curing step, indicated by the increase of the specific ester carbonyl peak at 1740 cm^{-1} in the FTIR spectrum. The formed esters turned out to be mainly linked to the lignin complex considering that the newly formed carbonyl groups were found present in heat-treated wood, yet were found to be absent in the isolated holocellulose. Esterification contributes to a decrease of hygroscopicity of wood and consequently improvements of its dimensional stability and durability. However the role of esterification in the decrease of hygroscopicity in the examined hydrothermal treatment process is believed to be minor compared to the influence of cross-linking reactions known to occur during thermal treatment of wood.

MATERIAL PROPERTIES

Durability

Samples of several wood species were treated in a two steps process, subsequently hydrothermal and dry heat-treated, and analysed for their resistance against fungal attack. Both treated and dry heat-treated specimen were prepared and analysed, in order to study the influence of moisture during hydrothermal treatment of wood. The resistance against all of the studied types of fungi was improved considerably after the treatment. Especially the resistance against brown rot fungi was increased by the treatment. Also the resistance against white rot and soft rot was improved. The increase of the decay resistance was found dependent on the applied process conditions. The treatment was found to be more efficient compared to a one step dry heat-treatment, with respect to improving the resistance against fungal attack. The effectiveness of the treatment is improved by applying a hydrothermal step before the dry heat-treatment step. The process conditions in the curing step appeared to have the largest effect on the resistance against soft rot and brown rot decay. White rot decay was less dependent on the curing conditions and found more affected by the hydrothermolysis, suggesting the decomposition of hemicellulose in the hydrothermolysis. The higher effect on brown rot and soft rot decay is assigned to the reduced hygroscopicity of the material. Detailed information on the biological resistance of Plato wood is given in Tjeerdsma et al. 1998 and Tjeerdsma et al 2000.

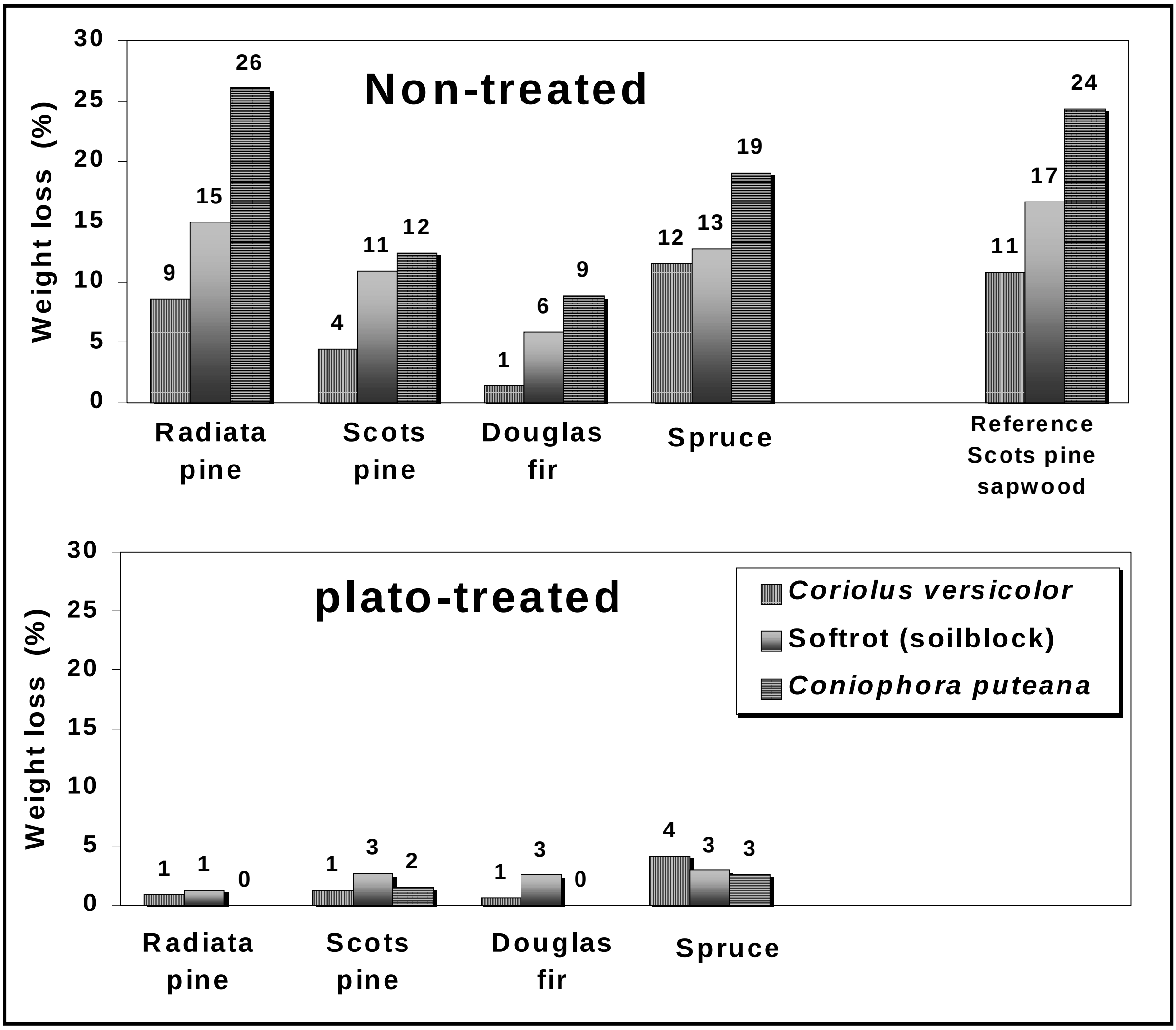


Figure 0/1. Weight loss of PLATO-treated and non-treated wood. Weight losses determined in the miniblock biotest (Bravery 1979) and in the soilblock test after 16 weeks of incubation

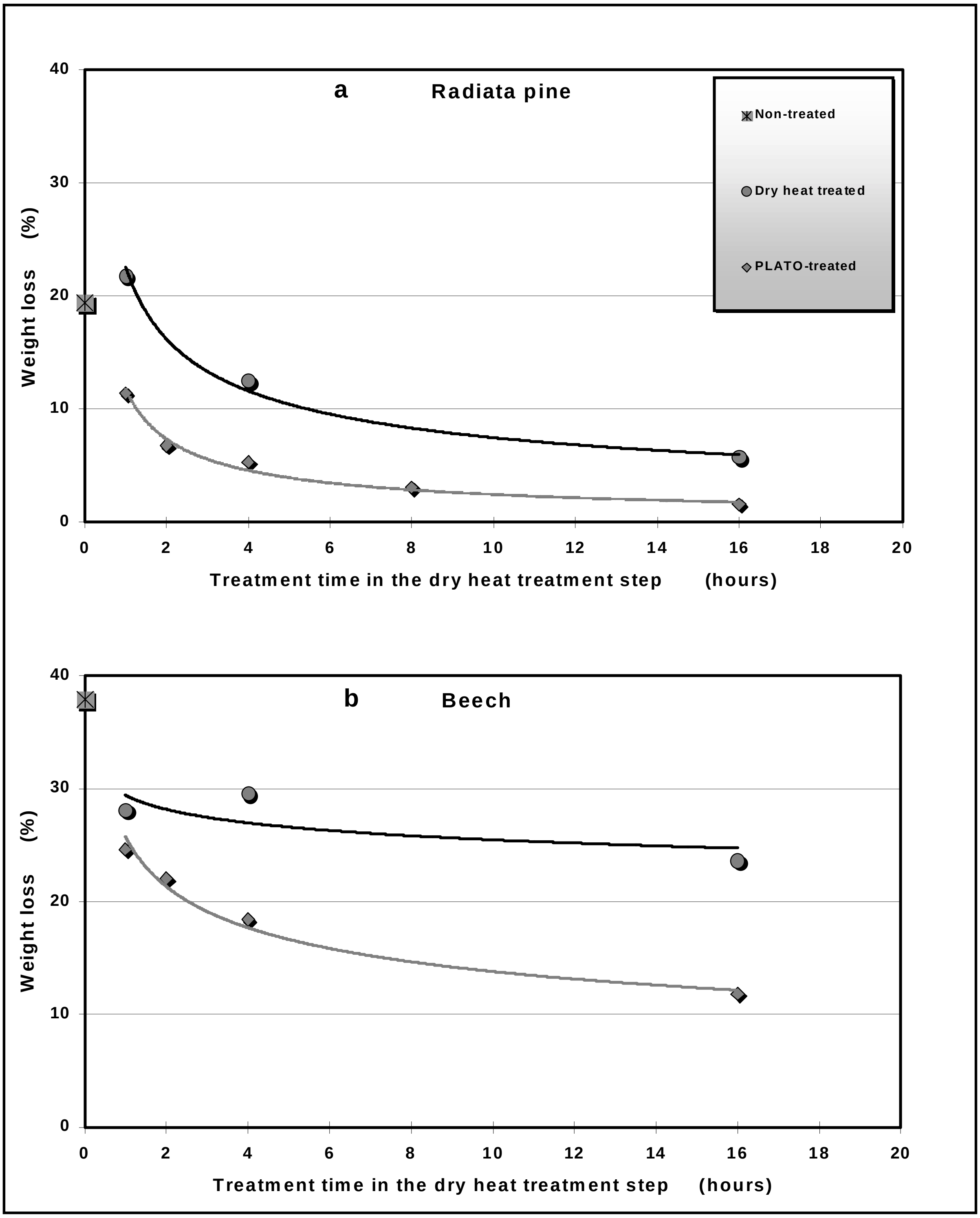


Figure 0/2. Weight losses of dry heat treated and PLATO-treated radiata pine and beech in a soil block test. Weight losses recorded after 16 weeks of incubation as a function of the treatment time in the dry heat-treatment step.

STRENGTH PROPERTIES

The modulus of rupture of several wood species, non-treated and heat-treated, is shown in Figure 1. The figure shows that an average strength loss of 5% to 18% has been found for wood heat-treated at whole plank scale (40 mm X 150 mm X 2200 mm). Earlier studies on this subject showed in general a strength loss to approximately 50% or more (Seborg *et al*. 1953; Davids and Thompson 1964; Giebeler 1983). The treated wood species comply with the required lower limit for use in joinery in the Netherlands. The low strength loss of Beech (Figure 1) is partially explained by the unforced increase of the density of this wood specie after treatment.

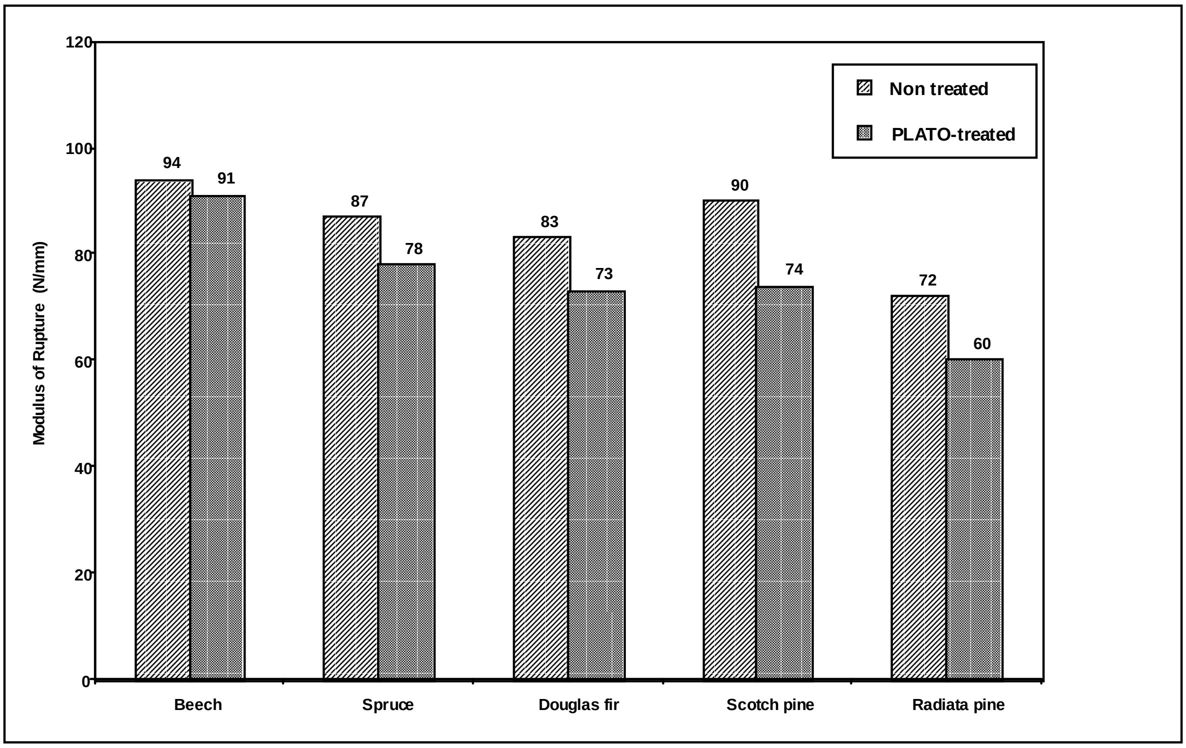
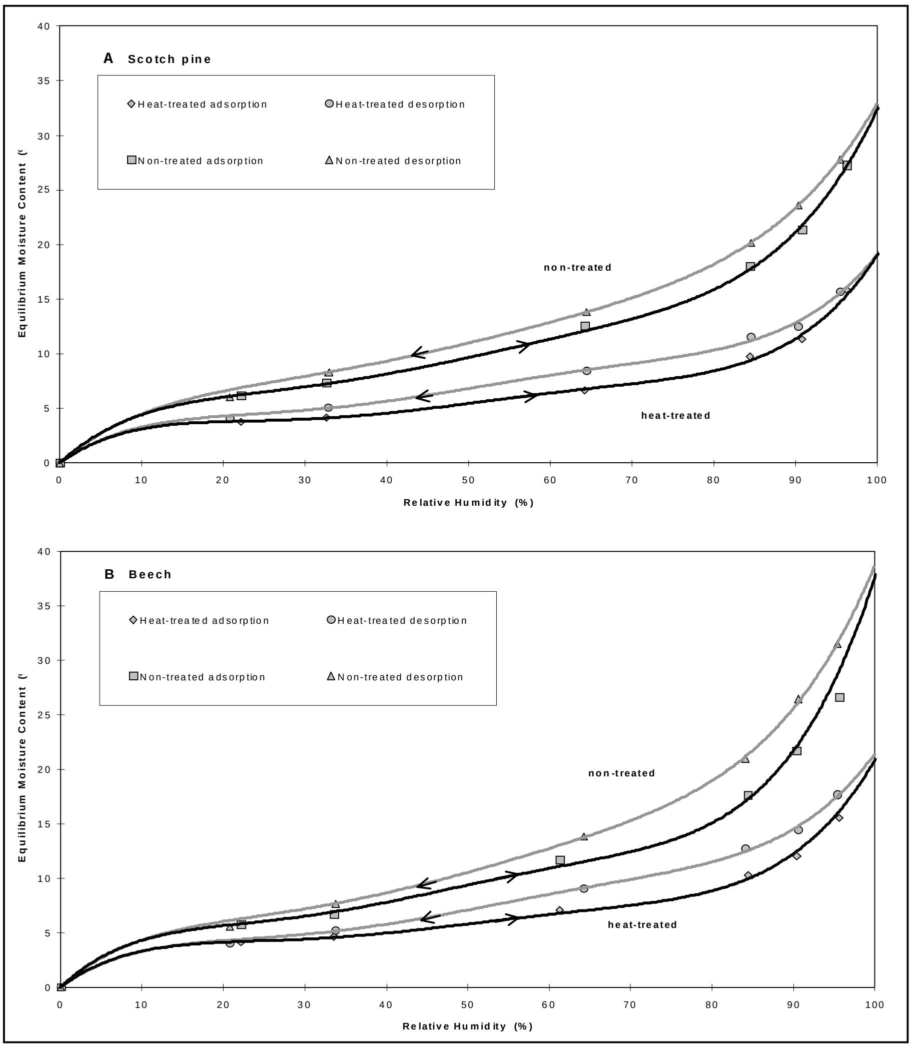


Figure 1. Modulus of rapture of some wood species before and after heat-treatment an whole black scale.

The results shown in the figure are based on wood samples free of defects and planks treated under mild conditions. During the process high tensions can occur in the wood since this treatment consists of three steps in which the wood is exposed to high temperatures and rapid evaporation of water. Some of the wood species were found difficult to treat and showed a number of defects (mainly cracks), if not treated carefully. Several softwood species are known to have a high resistance against liquid impregnation. These wood species were indeed found difficult to treat and showed a comparative higher strength loss. Altogether the strength was found to be dependent the applied process conditions and affected predominantly by the process temperature in combination with wood species.

HYGROSCOPICITY

The changed wood composition results in a lower hygroscopicity. As has been stated earlier



Figuur 2. Sorption curves of non-treated and heat-treated Scotch pine and Beech.

the hygroscopicity is the most indicative characteristic of wood with a major influence on both dimensional stability and durability. In this research the hygroscopicity is expressed in the Equilibrium Moisture Content (EMC) measured up the wood after conditioning in a specific climate. In the figure the adsorption and desorption curves of heat-treated and non-treated Scotch pine and Beech are shown.

The strong impact of the heat-treatment on the hygroscopicity is illustrated in the figure by the sorption curves of the treated wood positioned substantial lower than the sorption curves of the non-treated wood. The absolute improvement of the hygroscopicity is most pronounced of wood conditioned in humid air (R.H. > 70 %). For the relative improvement of the hygroscopicity a difference between Scotch pine and Beech has been found. The relative improvement of the hygroscopicity of Scotch pine appeared independent of the applied climate conditions and is over the whole range determined as an improvement of 40%. For Beech the improvement is approximately 30 % under dry conditions increasing to an improvement of 45 % in humid air of 96 % R.H.

Wood has the typical characteristic that it can adopt two different EMC's in one specific condition (R.H.), dependent on whether it is moisturised (adsorption) or dried (desorption) in order to reach this specific equilibrium condition. This hysteresis effect was found undiminished by the heat-treatment of wood. In all cases the hysteresis is clearly visible, showing an insignificant larger difference between adsorption and desorption for Beech. From corresponding research it is known that the hygroscopicity of heat-treated wood can be varied over a broad range varying process time and temperature in the second step of the treatment (Tjeerdsma *et al*. 1998b).

DIMENSIONAL STABILITY

In Figure 3 the percentage of swell at different relative humidities is shown of non-treated and heat-treated Scotch pine. The figure corresponds with the hygroscopicity. It can clearly be seen that the swell has been reduced substantially by the heat-treatment. The swell reduction was found independent of the relative humidity. The swell (or shrinking) reduction expressed in the ASE of the results shown range to approximately 50%. This was found to be near the maximum reachable ASE under the examined process conditions. In table 1 average ASE values found for several wood species are shown. The table illustrates that overall the ASE ranges from a minor to a substantial improvement of the dimensional stability of the wood by the treatment. In general the swell reduction in tangential direction in the wood was found higher compared to the reduction in radial direction. A decreased difference between the absolute swell in radial and tangential direction will result in less tensions in the wood when exposed to changeable climatically conditions.

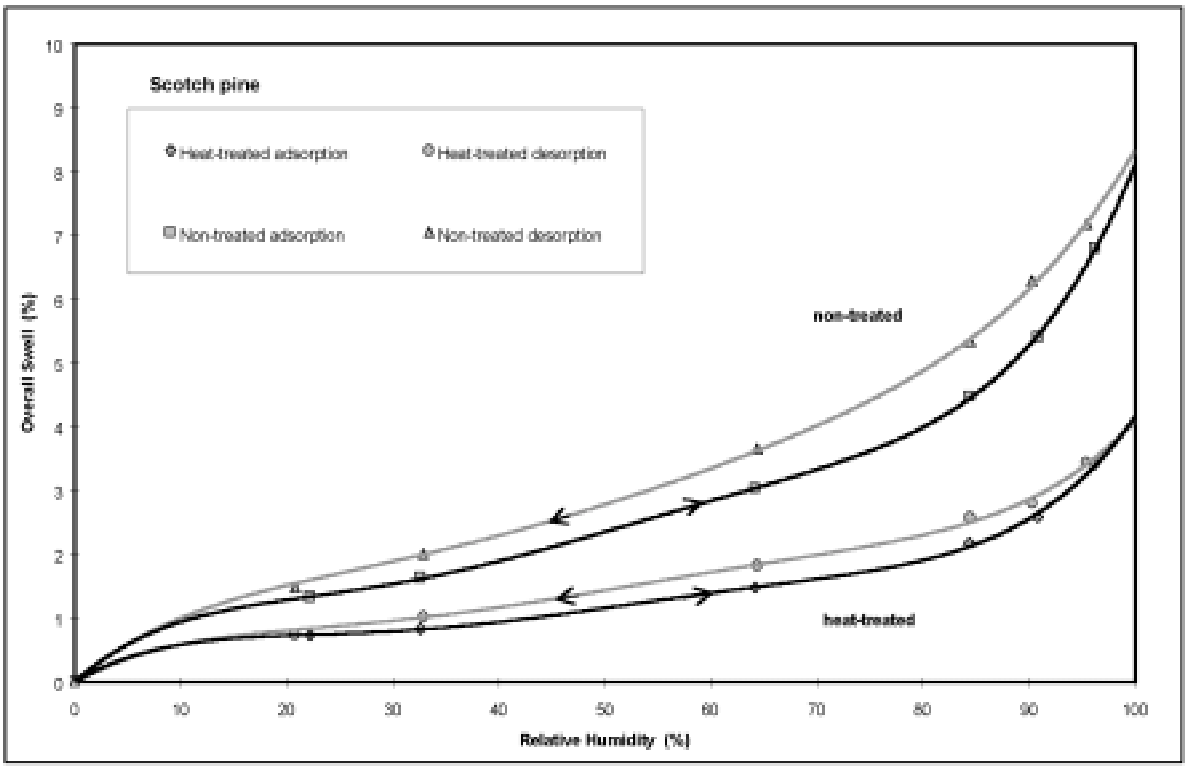


Figure 3 Dimensional stability of non-treated and heat-treated Scots pine. Adsorption: swell from oven dry to conditioned. Desporption: shrinkage from conditioned to oven dry. Temperature in the hydrothermolysis: 165°C. Temperature in curing: 180 °C

Table 1. Dimensional stability (Anti Shrinking Efficiency ASE) of several heat-treated wood species.

		Radial ASE %	Tangetial ASE %
Wood specie			
Beech		**10**	**13**
Douglas fir		**13**	**23**
Spruce	(poles)	**11**	**40**
Scotch pine		**33**	**41**
Radiata pine		**35**	**40**

Spruce has been treated as poles; 100 mm diameter X 2200 mm length.

Costs (figures given by Plato BV)

The production costs per m³ Plato wood are given by Plato bv with ca. 100 Euro. These costs are including handling costs, energy, water, depreciation of the plant etc., but excluding the costs of the timber itself. The selling costs of the product are depending on the species used and the end product.

The plant purchase costs are ca. 10 – 15 mln Euro for a plant of 75.000 m³ annual production and are depending on the infrastructural costs, logistics and facilities on the site (steam, energy etc.).

The operational costs are given with ca. 20 Euro per m³ Plato wood, including water, energy, post treatment of effluents etc.

Literature

Boonstra, M.J., B.F. Tjeerdsma and H.A.C. Groeneveld. (1998). Thermal modification of non-durable wood species. 1. The PLATO technology: thermal modification of wood. International Research Group on Wood Preservation, Document no. IRG/WP 98-40123.

Bravery, A. (1979). Miniaturised wood-block test for the rapid evaluation of preservative fungicides. Proc. Symposium (IRG), Screening techniques for potential wood preservative chemicals, Swed. Wood Pres. Inst., No 136, 57-65.

Burmester. A (1973) Einfluß eine Wärme-Druck-Behandlung halbtrockenen Holzes auf seine Formbeständigkeit, Holz als Roh- und Werkstoff 31: 237-243.

Burmester, A (1975) Zur Dimensionsstabilisierung von Holz, Holz als Roh- und Werkstoff 33: 333-335.

Burmester, A and Wille, W E (1976) Quellungsverminderung von Holz in Teilbereichen der relativen Luftfeuchtigkeit, Holz als Roh- und Werkstoff 34: 87-90.

Kollmann, F and Fengel D (1965) Änderungen der chemischen Zusamensetzung von Holz durch thermische Behandlung, Holz als Roh- und Werkstoff 23 (12) 461-468.

Kollmann, F and Schneider A (1963) Über das sorptionsverhalten wärmebehandelter Hölzer, Holz als Roh- und Werkstoff 21 (3) 77-85.

Garrote G., Dominguez H., Parajó J.C. 1999. Hydrothermal processing of lignocellulosic materials. Holz als Roh- und Werkstoff 57: 191 - 202.

Giebeler, E (1983) Dimensionsstabilisierung von Holz durch eine Feuchte/Wärme/Druck-Behandlung, Holz als Roh- und Werkstoff 41 : 87-94.

Rayner, A.D.M. and L. Boddy. 1988. Fungal decomposition of wood. John Wiley & Sons, p 587

Ruyter, H.P. 1989. European patent Appl. No. 89-203170.9.

Tjeerdsma, B.F., M. Boonstra, A. Pizzi, P. Tekely and H. Militz. 1998a, Characterisation of thermal modified wood: molecular reasons for wood performance improvement. CP-MAS ^{13}C NMR characterisation of thermal modified wood. Holz als Roh- und Werkstoff. *56*, 149-153.

Tjeerdsma, B.F., M. Boonstra and H. Militz. 1998b. Thermal Modification of Non-Durable Wood Species. 2. Improved wood properties of thermally treated wood. International Research Group on Wood Preservation, Document no. IRG/WP 98-40124.

Tjeerdsma, B.F., M. Boonstra and H. Militz. 1999. Chemical changes in hydro thermal treated wood; FTIR analysis of combined hydro thermal and dry heat-treated wood. Submitted for publication.

Tjeerdsma, B.F., M. Stevens, H. Militz 2000: Durability aspects of hydrothermal treated wood. . International Research Group on Wood Preservation, Document no. IRG/WP 00-4

Viitaniemi, P. and S. Jamsa. 1996. Puun modifiointi lampokasittelylla (Modification of wood with heat-treatment). Espoo 1996, VTT Julkaisuja - Publikationer 814.

Weiland, J.J. and R. Guyonnet. 1997 Retifiziertes Holz. 16. Verdichter Holzbau in Europa.

Motivation, Erfahrung, Entwicklung. Dreilander Holztagung. 10. Joanneum Research.

HEAT TREATMENT OF WOOD IN FRANCE – STATE OF THE ART

Dr. Michel Vernois, CTBA, Paris, France

INTRODUCTION

The poor dimensional stability of wood under variable atmosphere and the low durability of many species have originated research for stabilization treatment inducing the limitation of moisture absoption of the lignocellulosic material.

One of the process under study for the last decade in France and in Europe consists in submitting wood to heat treatment ranging from approximately 180°C to 250°C depending on the type of species and the physico-mechanical characteristics to reach.

The main objective is to reduce the hydrophilic behavior of wood by the tridimensional modification of the chemical structure of some of its components through heat treatment in controlled atmosphere as a soft pyrolysis reaction.

The way the pyrolysis is conducted and the selection of the various parameters involved in the process have an influence upon the characteristics of the final product.

Operating conditions are essential, and such parameters as atmosphere- temperature - processing time - rate of heating - species - weight and dimension of the pieces - original moisture of the wood should be taken into account for they can strongly affect the final properties. The aim is to reach the optimum balance between the improvement of the moisture resistance and the decrease of the mechanical characteristics depending on usage.

It has been observed that when submitted to heat treatment at high temperature, the kinetics of humidity absorption is noticeably modified resulting in a major reduction of the volume retraction and a lowering till a certain extent of some mechanical properties depending on the treatment applied.

Improved durability results from the combination of two factors induced by thermal treatment:

- A noticeable reduction of moisture absorption. Rot fungi need a minimum of 20% of humidity to develop.
- Elimination of some of the nutrients required by wood rotting fungi.

It should be noted that heat treatment induces chemical modifications in the wood which darken original color over the whole material.

Mainly two processes are in use at the present time in France.

The first one called Retification (Retified wood) has been developped by Ecole des Mines de Saint-Etienne and operating licences and patents have been acquired by the Company NOW (New Option Wood) (Also known as RETITECH).

The process consists in starting from wood previously dried around 12 % in humidity and to heat slowly in a specific chamber up to 210 – 240°C in a nitrogen atmosphere with less than 2 % in oxygen. The Industrial oven has been developed by the Company Four et Brûleurs REY, near Saint-Etienne.

Three Industrial Units are already in operation with a capacity of 3 500 m^3/year for each corresponding to a heat chamber of 8 m^3.

One more plant is already ordered and should be in operation new April 2001. A few others should be implemented next year in France.

The second Process is named "Le Bois Perdure" and the oven has been developed by the Company BCI-MBS.

Instead of starting from dry wood such a process allows to use fresh wood. The first step of the process consists in an artificial drying in the oven. Then the wood is heated up to 230°C under steam atmosphere (steam generated from the water of the wood).

TEMPERATURE USED DURING THE PROCESS

In both cases there is a compromise between durability and mechanical properties-higher the temperature, better the durability and lower some mechanical properties as strength to rupture. The treated wood at 230 – 240°C is much more durable but can loose up to 40 % in Modulus of rupture and is more brittle.

At 210°C, the material, depending upon the species, can be less brittle with mechanical characteristics close to the original values but the durability could be improved only slightly.
It means that the heat treatment shall be adjusted in terms of rate of heating, duration of treatment and maximum temperature to reach according to the application on usage.

The processes are very sensitive to slight changes in temperature which shall be controlled with accuracy. For example, in the Retification Process it has been observed and published recently that 230°C corresponds to a define modification of the lignin leading probably to crosslinking. Under such a temperature the treated wood does not show the same behavior, in terms of durability, that at a temperature above 230°C.

HEATING MEDIUM

The Retified wood is processed under inert nitrogen atmosphere, with the residual content of oxygen lower than two percents. The "Bois perdure" is processed under saturated water vapor atmosphere.

COSTS

Costs are very dependent upon the production level. The reprocessing of byproducts generated in some cases (for example: in the Retification process) should be taken into account in the cost as some of these byproducts constitute a certain pollution.

It is generally assumed that the cost of the Retification process is in the order magnitude of 150 – 160 EURO per treated cubic meter.

In the case of "Obis perjure" the supplier of the oven mentions costs of 100 EURO per treated cubic meter.

However, the yield is not the same for these two processes. We can easily understand that due to a better control of the raw material at the inlet of the oven and due to the fact that the wood is maintained under pressure during the process by a special device, the final yield is much higher in the Retification process than in the "Bois Perdure".

Plant – purchase cost

For the Retification process, with and oven of 8 cubic meters in capacity, which means an annual capacity of 3 500 cubic meters the total investment is in the range of 750 000 EURO. The "Bois Perdure" process seems to be less costly of 500 000 EURO.

Plane operation cost

The main operation costs are as follows:

- investments
- energy consumption
- nitrogen consumption (for the Retification process)
- maintenance
- treatment of effluents
- cost related to licence fees (patents)
- labor costs.

Retification process uses electrical energy

"Bois Perdure" uses gas energy. It should be noticed that, in that case, the VOC gases are reinjected in the burner to minimize air pollution as well as to improve the global energy consumption.

DOCUMENTED PROPERTIES

All properties are very dependent upon the wood species, the type of process, the final temperature reached.

However, in all cases, the material turns brownish in color, higher the temperature reached, darker the final product.

Smell

Wood treated at high temperature has always a strong smell just after treatment. After few days, such a smell decreases in intensity but could remain for several months.

Mechanical properties

As mentioned above, mechanical properties are very dependent upon the control of the process, the final temperature, the wood species etc.

The parameters to take into account are numerous and very sensitive to slight modification. In any case, the material becomes more brittle. At 230°C, quite often, a decrease of MOR in the range of 30 to 40 % can be measured with a very brittle behavior. (Catastrophic failure, without creep).

According to previous testing, mechanical properties after heat treatment are not strongly affected for poplar as they are for other species like pine trees.
This means that the density is not the only parameter involved.

Paintability

Surface tension of the wood is drastically affected after heat treatment.
Any kind of painting and finishing usually used for untreated wood cannot be used. However, it is possible to find some formulation and paints adequate on a surface of heat treated wood. If needed surface tension can be adjusted by additives. The main problem can arise from exudation of the resin from the resinous species.

Gluability

Proper glues have to be applied with heat treated wood. Research projects are presently carried out in CTBA Bordeaux on that topic.

Weathering properties

Wood treated at high temperature turns grey in colour after exposition to sun and UV, for few weeks. It is generally assumed that such grey colour is more homogenous than for untreated wood. Cracking, due to dimensional motion is reduced in comparison with natural wood.

Hygroscopicity

Wood treated at high temperature has less hygroscopicity than natural wood. It stabilises around 4 – 5 % in humidity instead of 10 to 12 %. This low hygroscopicity is of importance on biological durability (rot, stains, mould).

However, the material presents a certain porosity and when dipped in water it can absorbe more than 20 % of water. But when dried again the water can take out quite easily. Such behaviour is of importance for building materials.

Dimensional stability and cracking

It is known that heat treatment at a temperature above 200°C reduces by factor two dimensional movements. However, dimensional stability is largely dependent upon the process, the final temperature, the wood species.

Wood species

Species of high density are more difficult to process than low-density species. With species of high density (mostly hardwood) heat treatment has a tendency to induce cracking lowering drastically mechanical properties. Poplar seems to be interesting to process giving good results in terms of physical properties and durability. A large study on maritime pine has been carried out in France and the main results will be commented.

Durability

Durability is very dependent upon several factors:

- wood species
- type of process
- control accuracy of the process
- process time

maximum temperature reached and duration of time at such temperature.

Should we refer to natural durability or durability induced by chemical additives?

CTBA decided to refer to natural durability in terms of testing, standardisation, and requirements.

Industrial production

If plants have a theoretical capacity of 3 500 m3/year most of them are producing at around 50 % of their capacity. Total present industrial production in France is in the order of magnitude of 8 000 m3/year.

How many plants are in commercial use?

Six units are already in operation. Two others are expected and already announced for 2001.

Quality control and quality assurance

Each production unit has its own quality control. There is no quality assurance up to now and CTBA has been asked to insure such quality assurance in a future.

Production control of the plant

The raw material is checked according to internal specifications (dimension – moisture content) for each process. Each step of the process is recorded (heating time, percentage of oxygen, heat plateau, maximum temperature etc).

External control

Up to now there is no external control. However, such an external control could be part of quality assurance in the future.

Marking/labelling requirement

There is no marking and labelling today. There is a real need for such a labelling taking place after full quality assurance.

Quality testing after leaving the plant

In any case, we will have to find a non-destructive testing after heat treatment to insure that the treatment has been carried out properly according to the specifications required for the final usage. A simple test has to be found for industrial use. Few labs in France are already working on that topic.

R&D PROJECTS

Most of these projects are related to a specific process (for example: Retification). Hereunder are the present R&D topics:

- heat treatment of new wood species
- understanding of heat transfer in the material
- understanding of physico-chemical modification of lignin at high temperature.
- machinability of heat treated wood.

OIL HEAT TREATMENT OF WOOD IN GERMANY – STATE OF THE ART

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INTRODUCTION

After the introduction this paper will follow the structure and questions given by the organisers. By this the maximum comparability with the other heat-treatment processes shall be provided for the reader.

Thermal wood improvement processes have been developed and optimised in various countries for a considerable time. Stamm *et al.* (1946) reported on the first systematic attempts to increase resistance to wood-destroying fungi in a hot metal bath. Buro (1954, 1955) studied the heat treatment of wood in different gaseous atmospheres and in molten baths. Other aspects of the thermal treatment of wood were pursued in subsequent years. Interest often focused on the drying characteristics (Schneider 1973) and the chemical changes of heat-treated wood (Sandermann and Augustin 1963a; Kollmann and Fengel 1965; Topf 1971; Tjeerdsma *et al.*1998a) as well as increased dimensional stability (Kollmann and Schneider 1963) and changes in strength (Schneider 1971, Rusche 1973). Burmester (1973) found improved wood characteristics on applying a thermal pressure treatment. This process was further developed by Giebeler (1983). There have been continuing attempts to improve wood by thermal treatment for some years, especially in Finland, France and some other European countries (e.g. Dirol and Guyonnet 1993; Viitanen *et al.*1994;Troya and Navarette 1994; Boonstra *et al.* 1998; Tjeerdsma *et al.* 1998a; EC project BRE-CT-5006, 1998). The various wood improvement processes are documented in patent specifications (e.g. EP0018446, 1982; EP0612595, 1994; EP0623433, 1994; EP0622163, 1994; EP0759137, 1995; US5678324, 1997). In most of the publications on the heat treatment of wood, reference is made to improved dimensional stability and increased resistance to fungi, though also to negative changes in the wood's characteristics. The high temperatures during treatment increase the brittleness and the formation of cracks, in particular. Spotted surfaces due to exudation of rosin and low UV resistance of the heat-related brown hue also prove to be problematic during practical use of the wood. More recent investigations point to a lower resistance to fungi of heat-treated wood in contact with soil than suggested by earlier findings (Jämsä and Viitaniemi 1998; Rapp *et al.* 2000). The aim of the procedural approach presented here, which is based on the heating of wood in hot oil, is to improve some of these critical characteristics.

Heat treatments usually take place in an inert gas atmosphere at temperatures between 180 and 260°C (Leithoff and Peek 1998). The boiling points of many natural oils and resins are higher than the temperature required for the heat treatment of wood. This opens up the option of the thermal treatment of wood in a hot oil bath. Improvements in various wood characteristics can be expected from the application of oil-heat treatment as compared with heat treatment in a gaseous atmosphere, due to the behaviour of oils in conjunction with the effect of heat.

SHORT INFORMATION ABOUT THE EQUIPMENT AND PROCESS

The principle design of the plant

The principle design of the plant can be seen from Fig. 1.

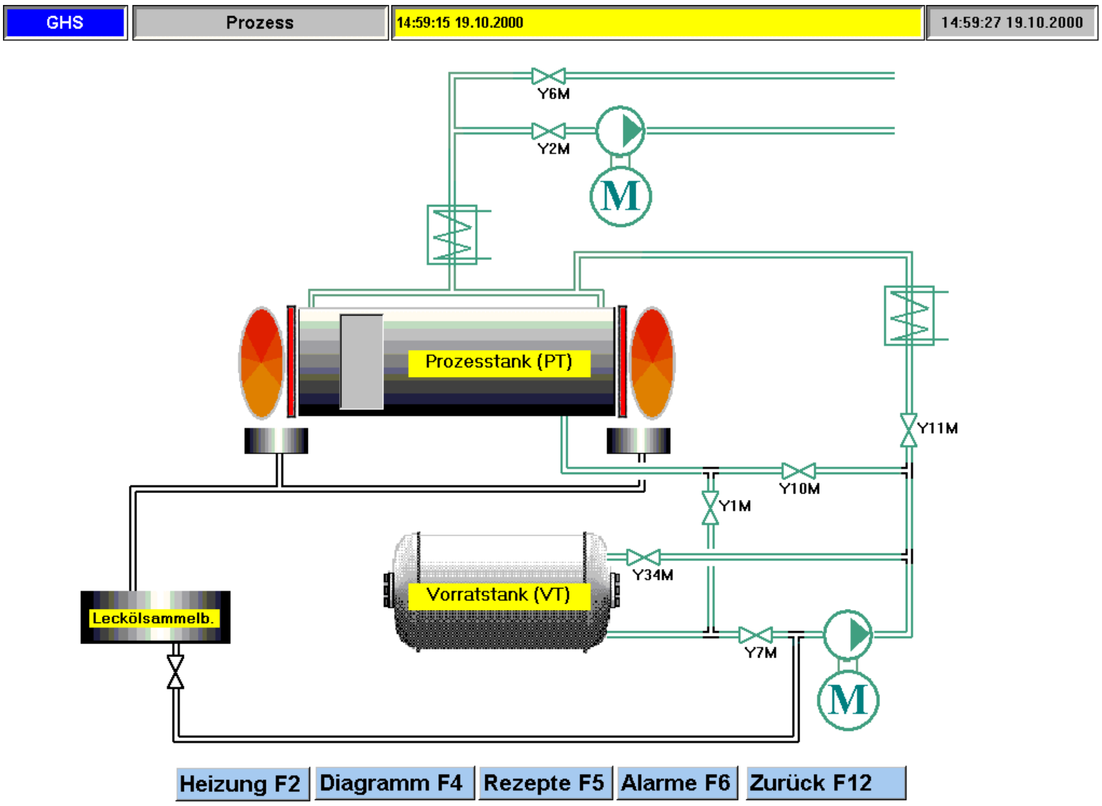

Fig. 1 Principle design of the plant. Diagram: designed by MENZ HOLZ Germany.

The process is performed in a closed process vessel (PT). After loading the process vessel (PT) with wood, hot oil is pumped from the stock vessel (VT) into the process vessel (PT) where the hot oil is kept at high temperatures circulating around the wood. Before unloading the process vessel (PT) the hot oil is pumped back into the stock vessel (VT).

Temperatures used during the process

For different degrees of upgrading, different temperatures are used.

To obtain maximum durability and minimum oil consumption the process is operated at 220°C. To obtain maximum durability and maximum strength temperatures between 180°C and 200°C are used plus a controlled oil uptake.

Process times

It proved to be necessary to keep the desired process temperature (for example 220°C) for 2-4 hours in the middle of the wooden pieces to be treated. Additional time for heating up and cooling down is necessary, depending on the dimension of the wood. Fig. 2 gives an example of the a heating up phase for logs with a cross section of 90 mm by 90 mm. Typical process duration for a whole treatment cycle (including heating up and cooling down) for logs with a cross section of 100 mm x 100 mm and length of 4 meters is 18 hours.

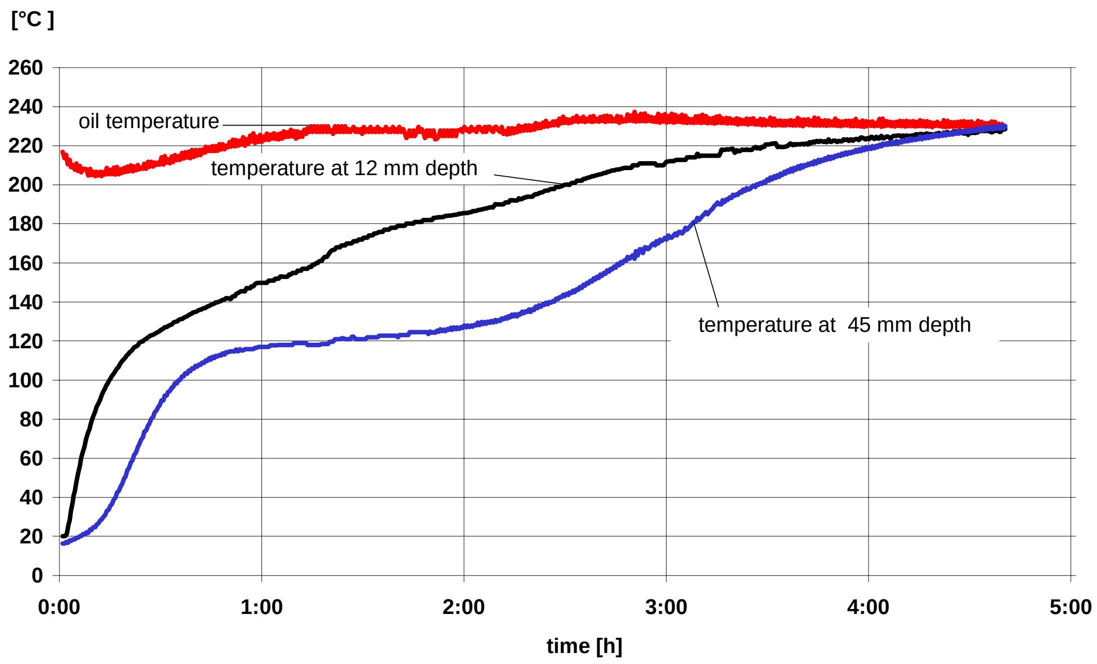

Fig. 2. Course of temperature in the oil and wood during the phase of heating up. *Picea abies* L. Karst., with a cross section: 90 x 90 mm².

Heating medium

The heating medium is crude vegetable oil. For example rape seed, linseed oil or sunflower oil. The oil serves for both,

fast and equal transfer of heat to the wood, providing the same heat-conditions all over the whole vessel

perfect separation of oxygen from wood

Natural plant oils lend themselves to the oil-heat improvement of wood from an environmental point of view and because of their physical and chemical properties. As renewable raw materials they are CO_2 neutral. The use of other plant oils, such as rape seed oil, sunflower oil, soybean oil or even tall oils or tall oil derivatives in addition to drying oils such as linseed oil, is also conceivable. Linseed oil proved to be unproblematic though the smell that develops during the heat treatment may be a drawback. The smoke point and the tendency to polymerisation are also important for the drying of the oil in the wood and for the stability of the respective oil batch. The ability of the oil to withstand heating to a minimum temperature of 230°C is a prerequisite. The consistency and colour of the oil change during heat treatment. The oil becomes thicker because volatile components evaporate, the products arising from decomposition of the wood accumulate in the oil and change its composition. This obviously leads to improved setting of the oils.

COSTS

Plant – purchase cost

The investment for a capacity of 8500 m³/a is 450.000 €.

Based on a 10 years period of use, 5.2 €/m³ are the calculated depreciation.

Plant – operation cost

The operation costs for treatment of spruce are 60 to 90 €/m³, depending on the desired oil loading.

Cost per treated cubic meter timber

The costs for 1 m³ of oil-heat-treated spruce are 265 to 295 €/m³ based on costs for untreated timber of 200 €/m³.

DOCUMENTED PROPERTIES

Before determining the properties, the material was treated as follows:
Fresh, untreated pine (*Pinus sylvestris* L.) and spruce (*Picea abies* L. Karst.) were used for the oil-heat treatment. The specimens with a wood moisture content of 6% were heated at three temperatures (180°C, 200°C and 220°C) unpressurised and with exclusion of oxygen in an oil bath of refined linseed oil. On the oil reaching the desired temperature, the wood specimens were immersed in it for 4.5 hours. Virtually no oil was absorbed during the actual heat treatment. To achieve the desired oil loading, the specimens cooled off in the oil bath for 15 minutes. Reference samples were also treated for 4.5 hours at corresponding temperatures in a drying chamber in an air atmosphere.

Rot

The oil-heat treated specimens and specimens treated in a hot air atmosphere were tested for resistance to *Coniophora puteana* in accordance with DIN EN 113 (1996). This was done using spruce as well as pine sapwood because the oil loading was
different, even though they were subjected to the same treatment, due to the different impregnability of the two types of wood. Treated specimens and untreated controls were incubated in a Kolle flask for 19 weeks. The percentage loss of mass was determined in relation to the pure wood mass after treatment.
Untreated spruce controls showed 48% loss of mass and pine controls 40% loss of mass. The resistance of heat-treated spruce and pine to the brown rot fungus *C. puteana* was improved with increasing temperatures. Treatment of wood in hot air did not prevent an attack of *C. puteana*. An average mass loss of 11% was determined for Scots pine, 5.5 % for spruce (Tab. 2).

Table 1: Mass loss after 19 weeks exposition of heat treated specimens acc.to DIN EN 113 (Fungus: *Coniophora puteana*)

	oil-heat-treatment				air-heat-treatment			
	pine sapwood		spruce		pine sapwood		spruce	
treatment	[g]	[%]	[g]	[%]	[g]	[%]	[g]	[%]
180°C	1,1	13,0	1,2	15,0	2,3	25,0	2,5	31,2
200°C	0,1	1,9	1,1	13,1	1,0	15,8	2,2	26,7
220°C	0,1	2,0	0,0	0,0	0,9	11,0	0,4	5,5

A noticeably lower loss of mass was determined for oil-heat treated specimens than for air-heat treated specimens. A loss of mass of less than 2% was found in the case of pine sapwood, when oil-heat treatment was applied at 200°C. With spruce, on the other hand, a decisive increase in resistance was only obtained at 220°C.

Stains and mould

Weathering tests in the field have shown, that superficial treatments are necessary to prevent the nice brownish coloured wood from bleaching and staining.

Insects

Insect tests have not been performed with oil-heat-treated wood in Germany.

Termites

Termite tests have not been performed with oil-heat-treated wood in Germany

Marine borers

Marine tests with heat-treated wood and oil heat treated wood are currently running. Preliminary results after 1 year do indicate that both, heat-treated wood and oil heat-treated wood are not resistant against marine borers.

MOE, MOR, Impact bending strength, brittleness

The MOE and MOR was determined in a three point bending test with medium force applied on 150x10x10 mm^3 treated and untreated wooden slats parallel to the grain on a universal testing machine. Tests of the impact bending strength provide information on the dynamic stability of wood specimens. They were performed using a Louis Schopper pendulum impact machine. The changes in the impact strength due to oil-heat or air-heat treatment were calculated in relation to untreated specimens of the same type of wood.
The highest MOE of more than 11,000 N/mm^2 were achieved at 200°C in the case of oil-heat treated specimens (Fig. 3). There was no reduction in the values for the MOE of elasticity of untreated coniferous wood with either heat treatment process.

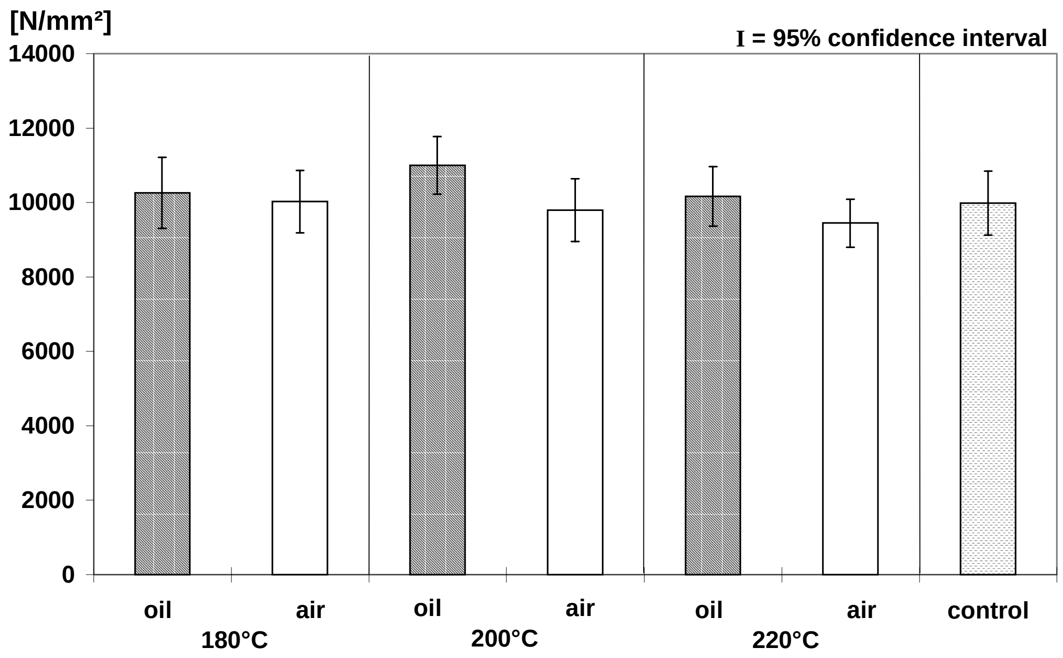

Fig. 3. Modulus of elasticity of *Pinus sylvestris* L. parallel to the fibres (150x10x10 mm³) after treatment; n=15

The MOR of wood which was oil-heat-treated at 220°C was about 70% of the value of untreated controls. The impact bending strength is the most critical value for all kinds of heat treatments. It declines considerably and the wood becomes brittle. Oil-heat-treated wood achieved a 51% and air heat treated wood only 37 % of the impact bending strength of untreated controls as the treatment temperature increased (Fig. 4).

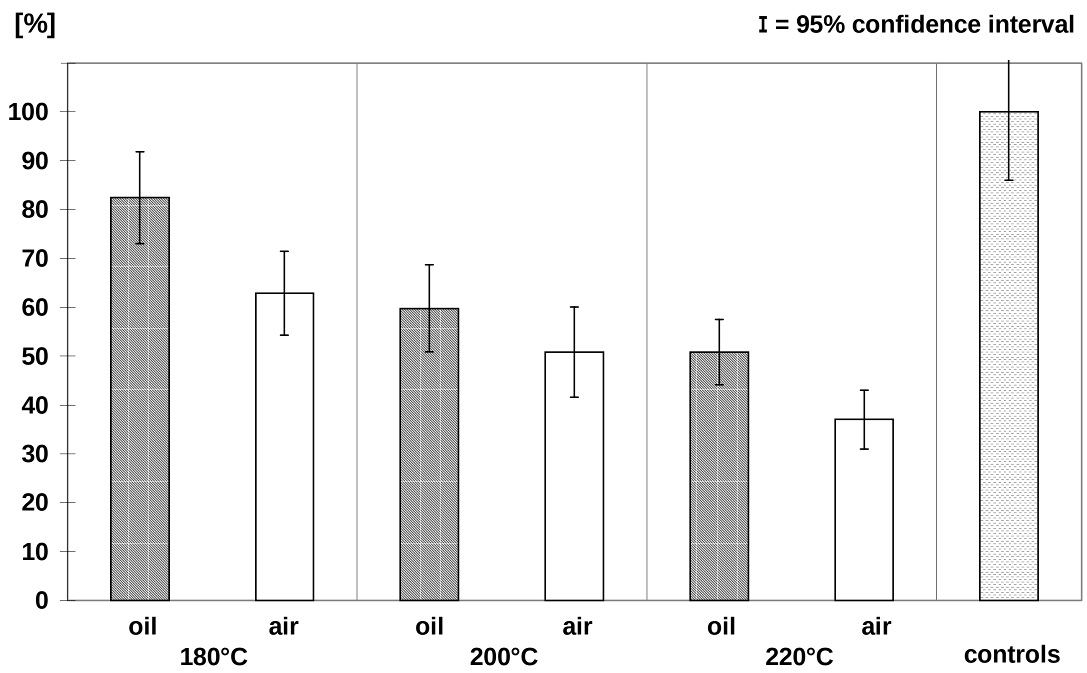

Fig. 4. Changes of the impact bending strength at *Pinus sylvestris* L. (150x10x10 mm³) after treatment compared to untreated controls; n=15

Smell

Like all heat treated woods also oil-heat-treated wood has that initially typical smoky smell. This could lead to limitations in use in internal areas, though this smell evaporates after some time. In any case this should hardly be a problem outdoors.

Colour and surface properties

At the end of the treatment cycle the oil remaining on the surface of the wood was absorbed by the wood very quickly during the cooling down of the specimens so that a dry wood surface appeared a few minutes after treatment. The surfaces were light brown in colour at lower treatment temperatures and dark brown at higher treatment temperatures. Unlike the air-heat treated specimens, no spotted discoloration due to the uneven distribution of exudated rosin was found on oil-heat treated specimens.

Paintability

The paintability of oil-heat-treated wood for acrylic water based paints as well as for alkyd solvent based systems proved to be good during two years of weathering. Surprisingly after two years the adhesion of the paints and varnishes on the oil-heat-treated wood was even better than on gas-heat-treated wood.

Gluability

Initial tests have been made with the following results: After planing of oil-heat-treated spruce, gluing was no problem. However for oil-heat-treated pine with higher oil uptake, only modified glues lead to good results.

Weathering properties

For heat treated softwoods its typical initial brownish colour is not UV-stable without surface treatments. After a half year of weathering in the field the colour of oil-heat-treated spruce came close to that of weathered untreated larch heartwood.

Hygroscopicity

If the oil-heat-treatment is performed for 4 hours at 220°C then the moisture content at fibre saturation was 14% whereas the moisture content of untreated controls was 29% under the same conditions.

Dimensional stability and cracking

Specimens for the examination of swell and shrink improvement (ASE) were exposed to a temperature of 20°C and a relative humidity of 35%, 65% and 85% after the oil-heat treatment. The dimensions of the specimens subjected to the above climatic conditions were determined after their masses becoming constant. The ASE was calculated from the ratio of the percentage volumetric change of the treated specimens in relation to the volumetric change of untreated wood specimens.

The improvement in the ASE of specimens that were treated at 220°C was similar for both types of treatment, at about 40%. The degree of improvement in this case depended on the relative humidity. When humidity was increased, the ASE became lower, with less difference in the specimens treated at higher temperatures than in those treated at lower temperatures (Fig. 5 to Fig. 7).

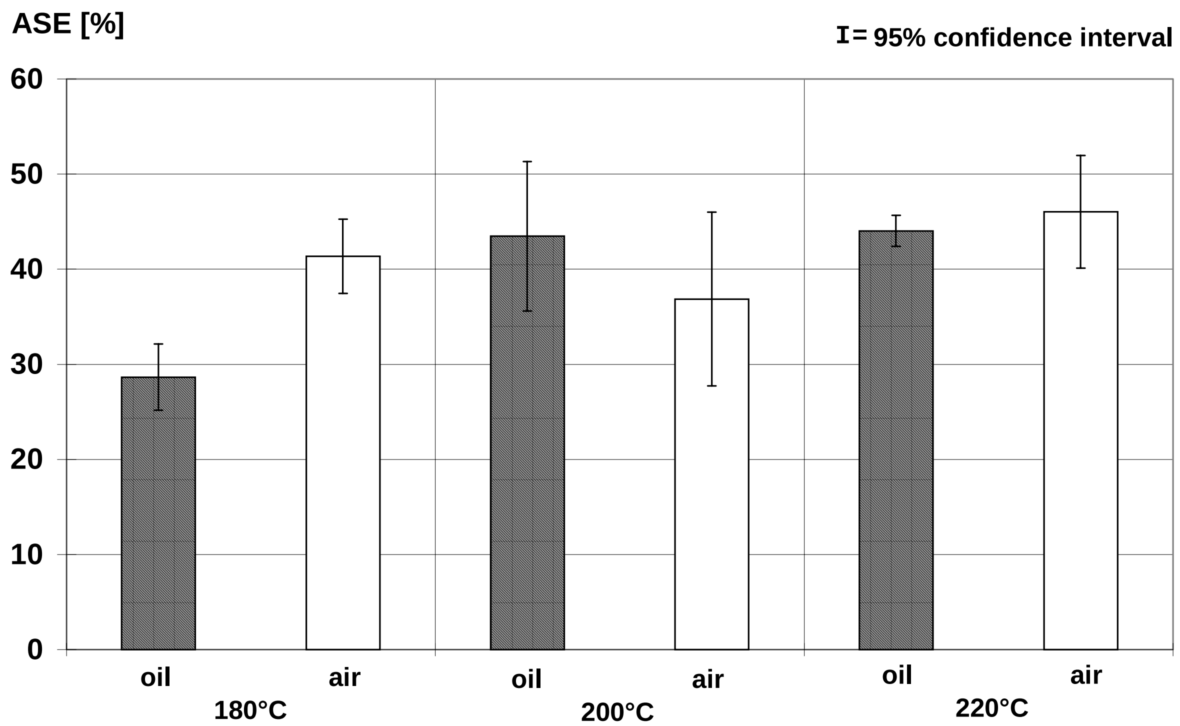

Fig. 5. ASE between 0 and 35% relative humidity of treated *Pinus sylvestris* L. (10x20x20 mm³), n=4

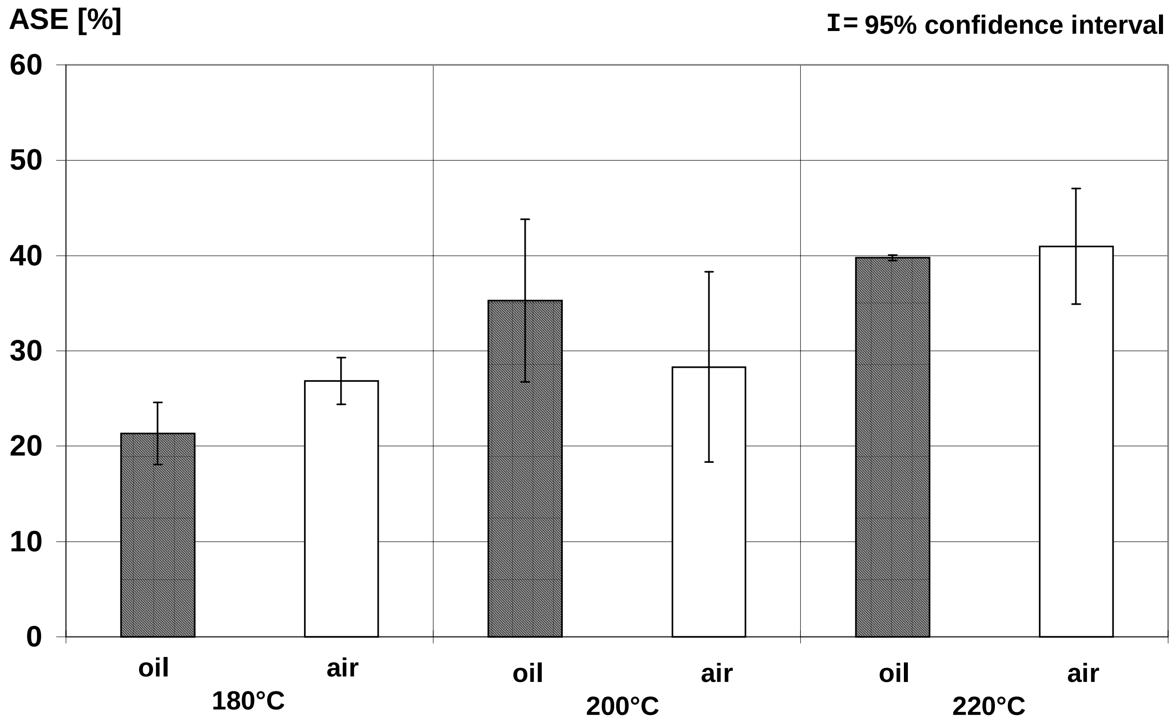

Fig. 6. ASE between 0 and 65% relative humidity of treated *Pinus sylvestris* L. (10x20x20 mm³), n=4

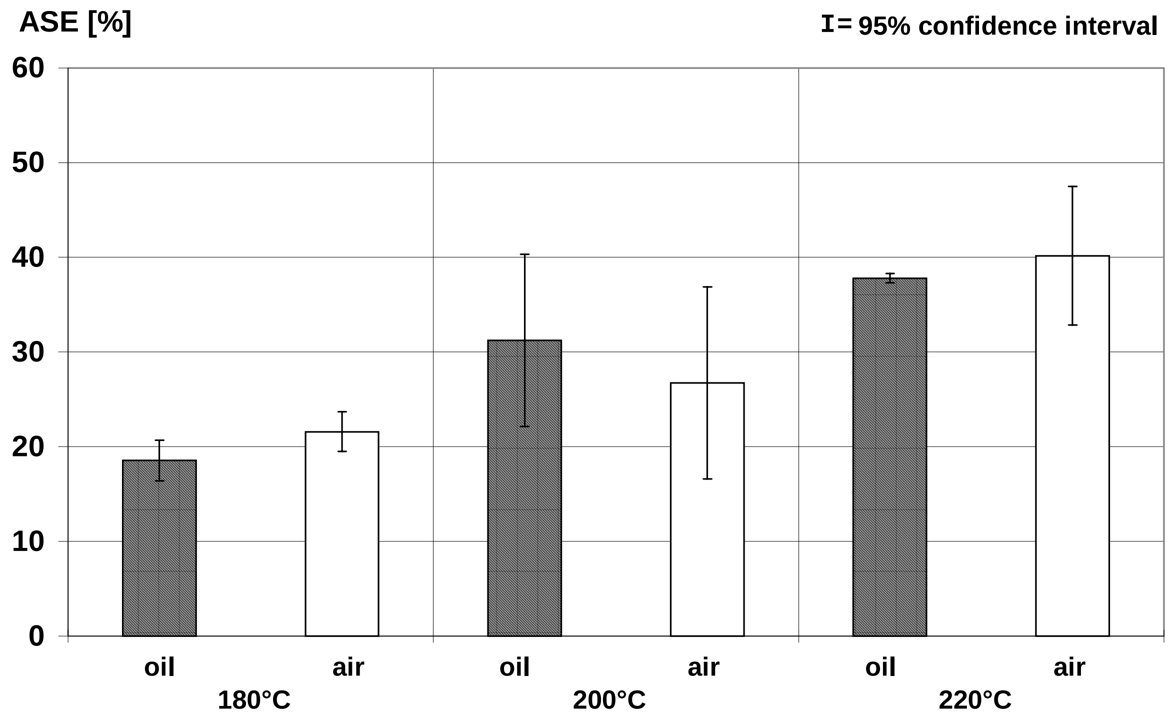

Fig. 7. ASE between 0 and 85% relative humidity of treated *Pinus sylvestris* L. (10x20x20 mm³), n=4

In Anglo-Saxon literature the term ASE (anti-swell efficiency or anti-shrink efficiency), coined by Stamm (1964) to describe the reduced swell or shrink of treated wood compared to untreated controls, has become established. In this case the swell or shrink characteristic of wood is determined under laboratory conditions with appropriate climatic conditions. However, it is not yet possible to make a statement on dimensional stability under open land conditions on the basis of these values because experience shows that the behaviour of samples subject to the multi-faceted stresses of open land is different from their behaviour under laboratory conditions. But since the ASE is easy to determine, it is often ascertained and therefore offers a good means for comparison of the various wood improvement processes. It is expected that the dimensional stability of oil-heat-treated samples will prove to be better in open land than that of wood that has undergone conventional heat-treatment in an oxygen-free gas atmosphere, due to the additional water-repellent effect of the oil component. Among other things this should have a beneficial effect on the stability of surface treatments, and on reduced crack formation during weathering. On the whole, the ASE values are consistent with the findings of Seborg *et al*. (1953) and Tjeerdsma *et al*. (1998b).

WOOD SPECIES

Suitable wood species

Unlike wood impregnation processes in which protective substances are introduced into the wood, even larger-dimensioned wood of types that are difficult to impregnate can be protected right into the inner areas of the log by oil-heat improvement. In Germany this applies in particular to spruce, which is available in large quantities at favourable prices.
The whole chain from basic research, standard testing to process optimisation was done for spruce (*Picea abies*) and pine (*Pinus sylvestris*).

Adoption of process parameters for different wood species

The use of the refractory spruce and the permeable pine sapwood allows to vary the oil uptake within a wide range. The process can be run with spruce with minimum oil uptake (20 to 60 kg/m³ depending on the dimension) when no pressure is applied. If high durability and high strength properties are desired, then pine can be used at lower temperatures but with higher oil uptakes (see Table 1). By application of pressure in the process the desired uptake can easily be adjusted.

Influence of wood species on durability

The combination of vegetable oils and heat treatment led to greater improvement in the resistance of wood to *Coniophora puteana* than heat treatment in air. Besides the pure thermal modification at high temperatures the oil uptake contributes to the durability (see Table 1). It is conceivable to use oil-heat treated wood with a higher oil-loading under more sever conditions in European hazard class 3. In European hazard class 4, high oil loading extends the service life of oil-heat-treated wood, compared to heat treated wood with no oil uptake.

Influence of wood species on technical properties

Regarding technical properties, the investigated species, spruce and pine, did not reveal particular differences of the oil-heat-treated wood.

Recommended species for the treatment

There are no species excluded from oil-heat-treatments. However the most experience exists for the treatment of Norway spruce and Scots pine. For most commodities in European hazard class 3 Norway spruce is suitable.

SUITABLE COMMODITIES

- claddings
- pergolas
- exterior joinery
- garden furniture
- decks
- fencing
- noise barriers
- wood in soil contact (treated at high temperatures and high oil loadings)

INDUSTRIAL PRODUCTION

Plants in commercial use

There is currently one plant in commercial use in Germany. The plant (Fig. 8) is operated since August 2000 by MENZ HOLZ in Reulbach, 30 km east of the city of Fulda. MENZ is intending to cover the German marked segment of oil-heat-treated wood for garden furniture and wood for gardening. The company is interested to find partners for licensed production of oil-heat-treatment wood for different market segments in all European countries.

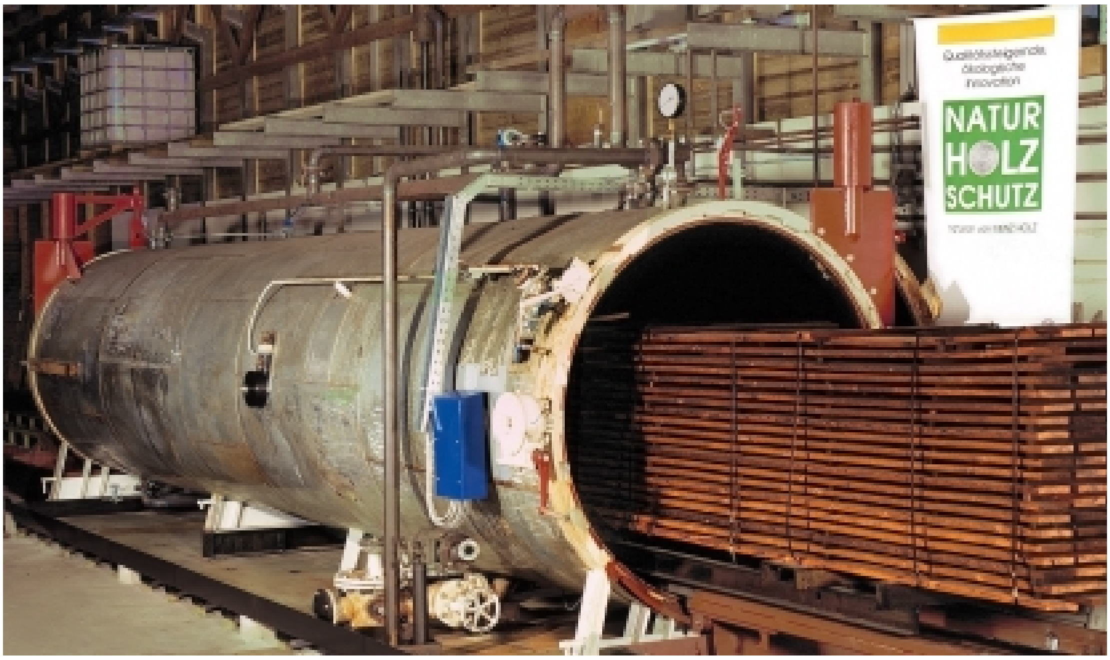

Fig. 8: Process vessel for the oil-heat-treatment in Reulbach. (Foto: MENZ HOLZ, Germany).

Produced volume per annum

The existing vessel has a capacity of 2900 m³/a. The future vessels planned by MENZ have typical capacities of 8500 m³/a.

QUALITY CONTROL AND QUALITY ASSURANCE

Production control at the plant (internal control)

The process is computer controlled. Data of the following values are controlled and logged:

- time
- oil consumption (≈ oil uptake)
- temperature inside the wood
- oil temperature
- pressure

Each batch is documented by the full set of computer process data in a diagram.
From each batch samples are drawn for determination of the DIV = durability indicator value and the SIV = strength indicator value.

The methods and procedures for determination of the DIV and SIV of heat treated wood are developed by BFH (Federal Research Centre of Forestry and Forst Products, Hamburg, Germany) in the frame of an ongoing research project for quality control of heat treated wood.

External control

MENZ is intending to assure quality of oil-heat-treated wood by external quality control.

Marking/labelling requirement

MENZ is intending to set up a marking/labelling system for the different qualities of oil-heat-treated wood.

Possibility of quality testing after leaving the plant

The DIV and SIV are meant for quality assessment of material after leaving the plant, since they are based on analysis of the treated material and therefore independent of the batch documentation of the process.

INFORMATION ABOUT ONGOING RESEARCH AND DEVELOPMENT PROJECTS

- BFH is currently developing a quality control system for heat treated wood on the basis of DIV and SIV.
- BFH is currently running comparative assessment of properties of heat treated wood produced after 4 different processes in different countries.
- BFH has together with Swedish University of Agricultural Sciences Uppsala (SLU) and Chalmers University ongoing field tests to assess the long term performance of heat treated wood.

Recent results of research projects of BFH are published in:

Sailer, M.; Rapp, A. O.; Leithoff, H. 2000: Improved resistance of Scots pine and spruce by application of an oil-heat treatment. IRG/WP/00-40172, 16p.

Sailer, M.; Rapp, A. O.; Leithoff, H.; Peek, R.-D. 2000: Vergütung von Holz durch Anwendung einer Öl-Hitzebehandlung. Holz Roh-Werkstoff 58: 15-22.

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LITERATUR

Boonstra MJ, Tjeerdsma BF, Groeneveld HAC (1998) Thermal modification of non-durable wood species. IRG/WP 98-40123, 13 p

Burmester A (1973) Einfluß einer Wärme-Druck-Behandlung halbtrockenen Holzes auf seine Formbeständigkeit. Holz Roh- Werkstoff 31: 237-243

Buro A (1954) Die Wirkung von Hitzebehandlung auf die Pilzresistenz von Kiefern- und Buchenholz. Holz Roh- Werkstoff 12: 297- 304

Buro A (1955) Untersuchungen über die Veränderungen der Pilzresistenz von Hölzern durch Hitzebehandlung in Metallschmelzen. Holzforschung 9: 177-181

DIN EN 113 (1996) Prüfverfahren zur Bestimmung der vorbeugenden Wirksamkeit gegen holzzerstörende Basidiomyceten: Bestimmung der Grenze der Wirksamkeit.

Dirol D, Guyonnet R (1993) The improvement of wood durability by retification process. IRG/WP 93-40015, 11 p

EC Bericht BRE-CT-5006 (1998) Upgrading of non durable wood species by appropriate pyrolysis thermal treatment. EC-Industrial & Materials Technologies Programme (Brite-EuRam III), 17 p

Giebeler E (1983) Dimensionsstabilisierung von Holz durch eine Feuchte/Wärme/Druck-Behandlung. Holz Roh- Werkstoff 41: 87-94

Jämsä S, Viitaniemi P (1998) Heat treatment of wood. Better durability without chemicals. Nordiske Trebeskyttelsesdager: 47-51

Kamdem D P, Pizzi A, Guyonnet R, Jermannaud A (1999) Durability of Heat-Treated Wood. IRG/WP 99-40145, 15 p

Kollmann F, Schneider A (1963) Über das Sorptionsverhalten wärmebehandelter Hölzer. Holz Roh- Werkstoff 21: 77-85

Kollmann F, Fengel D (1965) Änderungen der chemischen Zusammensetzung von Holz durch thermische Behandlung. Holz Roh- Werkstoff 23: 461-468

Leithoff H, Peek R-D (1998) Hitzebehandlung - eine Alternative zum chemischen Holzschutz. Tagungsband zur 21. Holzschutz-Tagung der DGfH in Rosenheim: 97-108

Patents:

EP0018446 1982: Verfahren zur Vergütung von Holz, 5 p

EP0612595 1994: Process for upgrading low-quality wood. 6 p

EP0623433 1994: Process for upgrading low-quality wood 6 p

EP0622163 1994: Process for upgrading low-quality wood 6 p

EP0759137 1995: Method for processing of wood at elevated temperatures. 12 p

US5678324 1997: Method for improving biodegradation resistance and dimensional stability of cellulosic products. 12 p

Rapp, A. O., Sailer, M., Westin, M. (2000) Innovative Holzvergütung – neue Einsatzbereiche für Holz. In: Proceedings of the Dreiländer-Holztagung, Luzern, Switzerland

Rusche H (1973) Festigkeitseigenschaften von trockenem Holz nach thermischer Behandlung. Holz Roh- Werkstoff 31: 273-281

Sandermann W, Augustin H (1963a) Chemische Untersuchungen über die thermische Zersetzung von Holz. Holz Roh- Werkstoff 21: 256-265

Sandermann W, Augustin H (1963b) Untersuchungen mit Hilfe der Differential-Thermo-Analyse. Holz Roh- Werkstoff 21: 305-315

Schneider A (1971) Untersuchungen über den Einfluss von Wärmebehandlung im Temperaturbereich von 100°C bis 200°C auf Elastizitätsmodul, Druckfestigkeit und Bruchschlagarbeit von Kiefern-Splint und Buchenholz. Holz Roh- Werkstoff 29: 431-440

Schneider A (1973) Zur Konvektionstrocknung von Schnittholz bei extrem hohen Temperaturen. Holz Roh- Werkstoff 31: 198-206

Seborg RM, Tarkow H, Stamm AJ (1953) Effect of heat upon dimensional stabilisation of wood. Journal of Forest Products Research Society 3: 59-67

Stamm AJ (1964) Wood and cellulose science. New York: Ronald Press

Stamm AJ, Burr HK, Kline AA (1946) Heat stabilized wood (staybwood). Rep. Nr. R. 1621. Madison: Forest Prod. Lab

Tjeerdsma BF, Boonstra M, Militz H (1998a) Thermal modification of non-durable wood species II. IRG/WP 98-40124, 10 p

Tjeerdsma BF, Boonstra M, Pizzi P, Tekely P, Militz H (1998b) Characterisation of thermally modified wood: molecular reasons for wood performance improvement. Holz Roh- Werkstoff 56: 149-153

Topf P (1971) Versuche zur Frage der Selbstentzündung, des Gewichtsverlustes, des Brennwertes und der Elementaranalysen. Holz Roh- Werkstoff 29: 295-300

Troya MT, De Navarrete AM (1994) Study of the degradation of retified wood through ultrasonic and gravimetric techniques. IRG/WP 94-40030, 6 p

Viitanen HA, Jämsä S, Paajanen LM, Nurmi AJ, Viitaniemi P (1994) The effect of heat treatment on the properties of spruce. IRG/WP 94-40032, 4 p

Viitaniemi P, Jämsä S (1998) Thermowood ToW. Decay-resistant wood created in a heating process. Vortrag bei der 29 IRG-Tagung in Maastricht, 4 p

SHORT SUMMARY OF DISCUSSION ON HEAT TREATMENTS

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During the intense and lively discussion the following subjects were touched.

- Quality control: It was stated that the quality control is not yet under control and a general standard for all heat treatment processes is needed. The difference between internal quality control during the treatment process and the quality control for the customer was addressed. For the quality control the durability performance is only one parameter, as all heat-treated products aim also on the improvement of other wood properties as well. The discussion showed that the development of a quality control system for the customer is at an early stage and a lot of work has to be done in this field.

- Work place exposure problems: Fields of interests that were named during the discussion were VOC's, PAH's, effluents and dust. It was pointed out, that the dust of untreated Oak and Beech is already stated as carcinogenic. For all different treatments it was stated identical, that dust generation can be a problem due to the higher amount of dust during machining. It seems also that the dust particles are of smaller size than untreated wood. There are no studies known that deal with the possible problems with regard to a different work place exposure for the work with heat treated wood and research work is needed here.

- Free radicals: During the discussion it was shortly addressed that there seems to be differences in the occurrence of free radicals between different treatment processes. Whereas in the French and the Dutch process the free radicals in the wood are decreasing very fast, free radicals were found in the heat treated wood from the Finnish process after 7 years.

- Blunting of tools: It was stated identical for all discussed heat treatments that no problems with the blunting of tools occur.

Furthermore the group was informed that Stora Enso is building a heat treatment plant in Finland with an annual capacity of 20.000 m^3.

This report provides reviews on each of the four different major European heat treatment processes and properties of wood thermally modified after these processes:

the Finnish THERMO WOOD

the Dutch PLATO WOOD

the French RETIFICATION

the German OIL HEAT TREATMENT

All four modification treatments have in common that solid wood is subjected to temperatures close to or above 200°C for several hours in an atmosphere with low oxygen content. By this thermal modification some mechanical properties are reduced but the dimensional stability and the biological durability of wood is increased without adding outside chemicals / biocides to the wood. Therefore thermally modified wood is discussed as a new material for several applications.