

Heat treatment of wood by the PLATO-process

Holger Militz^{1,2} och Bôke Tjeerdsma¹

^{1,2}SHR Timber Research
Wageningen, The Netherlands

²Universität Göttingen, Institut für Holzbiologie und Holztechnologie
Göttingen, Germany



Heat treatment of wood by the PLATO-Process

Prof. Dr. Holger Militz¹ and Ir. Bôke Tjeerdsma²

¹ and ² SHR Timber Research, Wageningen, The Netherlands

¹ Institute of Wood Biology and Wood Technology, University Göttingen, Germany

General

Recent efforts on thermal treatment of wood have led 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 m³.

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 (superatmospheric 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 liberated 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 C α and O4 and believed to cause some aldehyde production from lignin units C γ , all occurring in the first reaction step. Lignin autocondensation through the cleaved, positively charged benzylic C α 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 C α . 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.

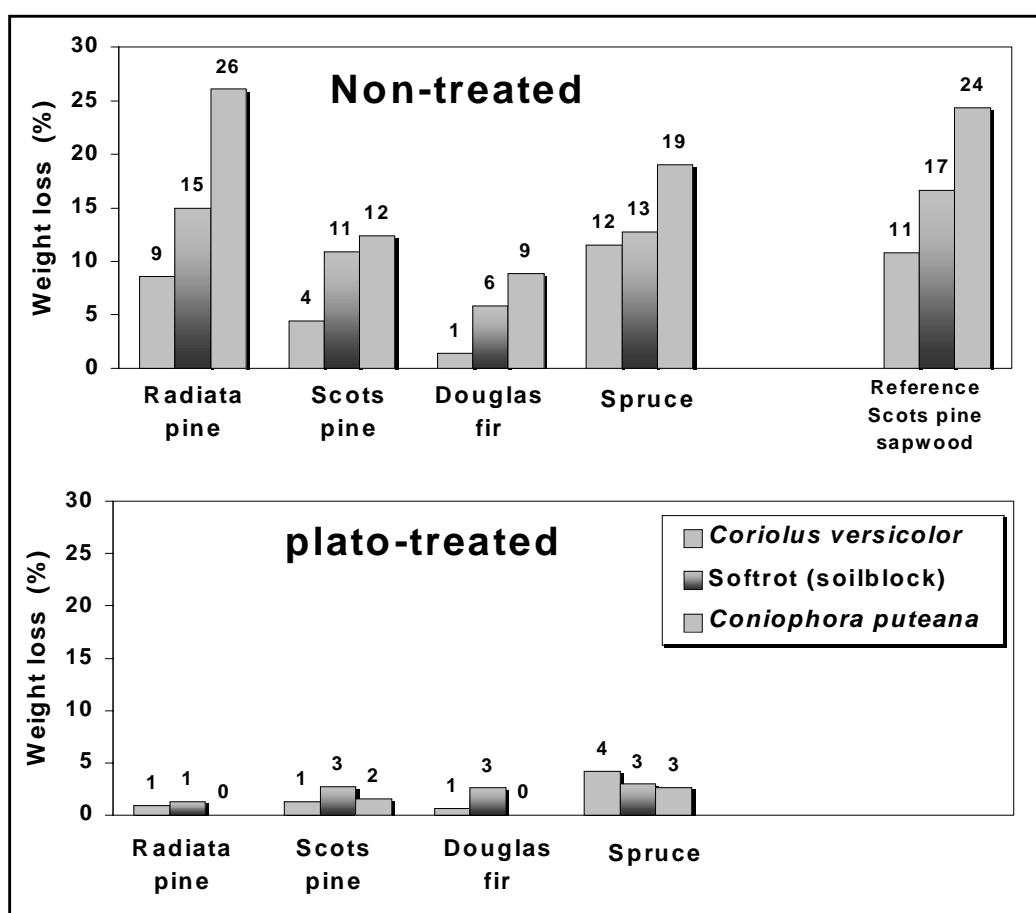


Figure 1. Weight loss of PLATO-treated and non-treated wood. Weight losses determined in the miniblock biotest (Bravery 1979) and in the soilblock test after 16 weeks of incubation.

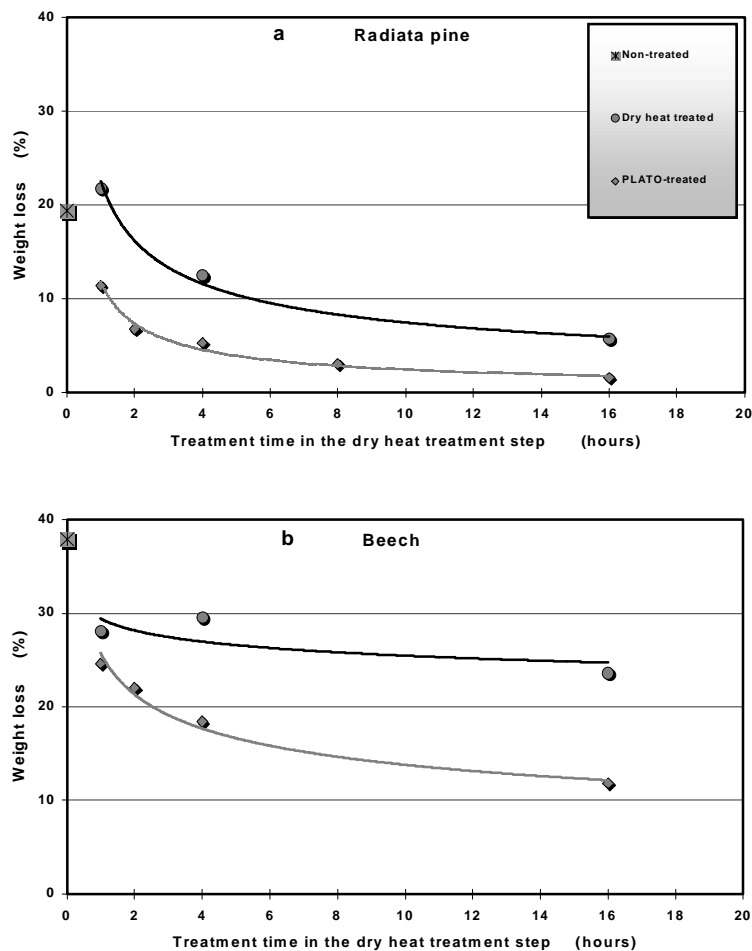


Figure 2. Weight losses of dry heat treated and PLATO-treated radiata pine and beech in a soil block test. Weight losses recorded after 16 weeks of incubation as a function of the treatment time in the dry heat-treatment step.

Strength properties

The modulus of rupture of several wood species, non-treated and heat-treated, is shown in Figure 3. 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; Giebel 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 3) is partially explained by the unforced increase of the density of this wood species after treatment.

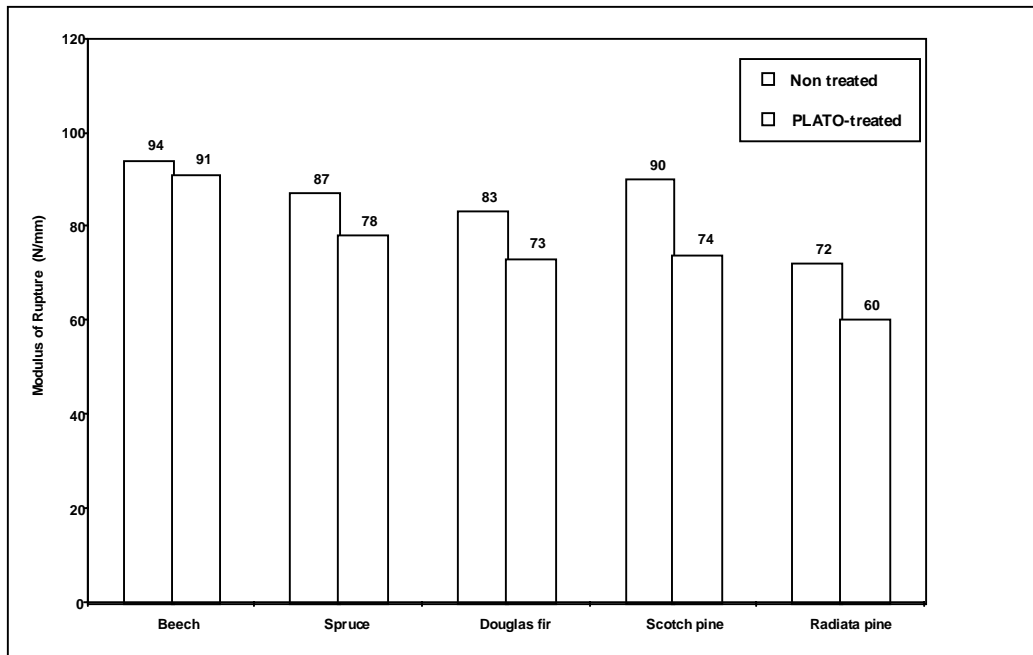


Figure 3 Modulus of rapture of some wood species before and after heat-treatment on whole plank scale.

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 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 figure 4 the adsorption and desorption curves of heat-treated and non-treated Scotch pine and Beech are shown.

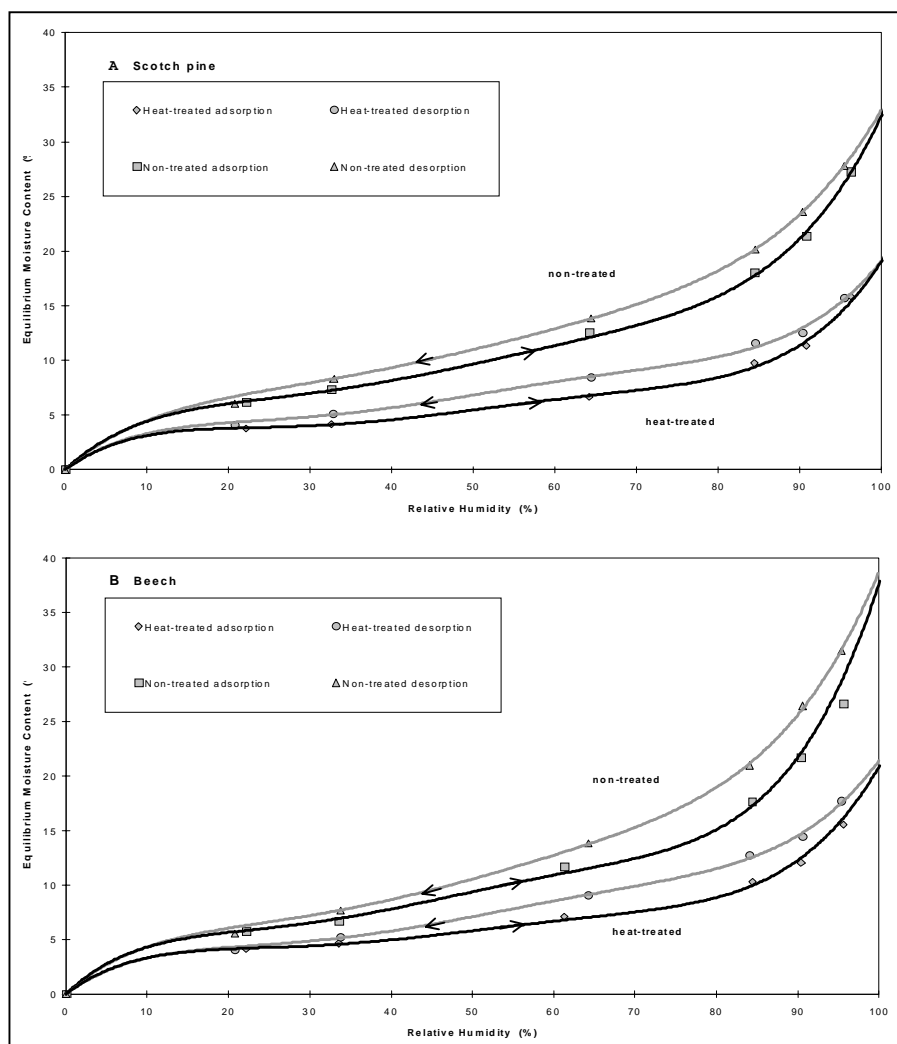


Figure 4 The adsorption and desorption curves of heat-treated and non-treated Scotch pine and Beech.

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

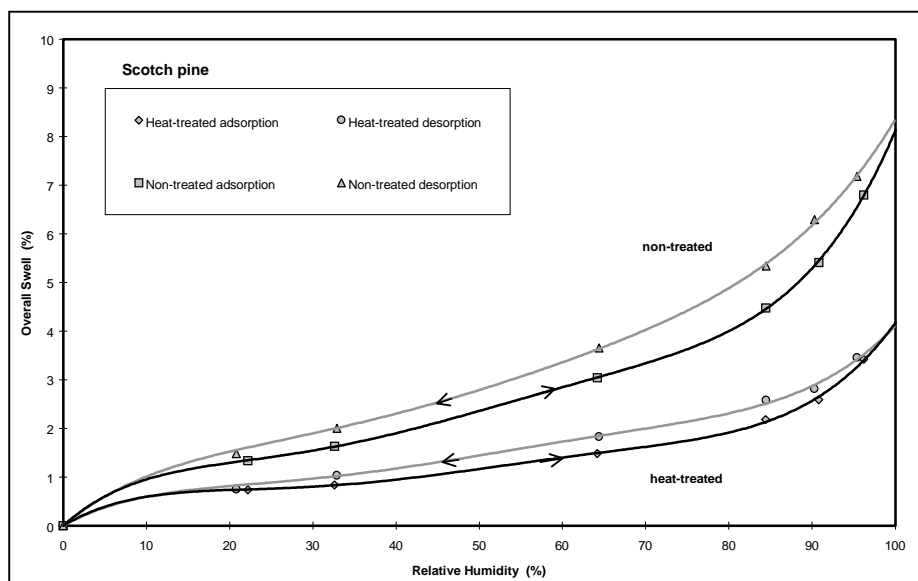


Figure 5 Dimensional stability of non-treated and heat-treated Scots Pine. Absorption: swell from oven dry to conditioned. Desorption: shrinkage from condition to oven dry. Temperature in the hydrothermolysis: 165 °C. Temperature in the curing 180 °C.

In Figure 5 the percentage of swell at different relative humidities is shown of non-treated and heat-treated Scots 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.

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

	Radial ASE %	Tangential ASE %
<i>Wood species</i>		
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- undWerkstoff 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 Zusammensetzung 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 ¹³C 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 Fachtag. 2-5.11.1997. Grazer Congress. Graz, Austria.