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INVESTIGATION OF CHEMICAL CHANGES IN THE STRUCTURE OF THERMALLY MODIFIED WOOD♣

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ABSTRACT

Thermal modification beneficially alters several technological parameters of wood. The changes in the physical parameters are due to the significant alterations of the structure and the chemical composition of wood, which take place during the modification process. These changes are complex and some aspects are still far from being completely understood. Various industrially important hardwoods and softwoods have been treated in an autoclave in N_2 atmosphere. The physical (density, L-value, moisture content, bending strength, MOE) and chemical (pH, hemicellulose-, total phenol- and soluble carbohydrate content) parameters have been measured and evaluated. By establishing linear correlations between physical and chemical parameters the chemical changes affecting the physical properties could be investigated and tracked. Very good correlations have been found for hardwood samples, whereas for softwoods only poor correlations have been established. Results could contribute to a better understanding of the reactions of thermal modification, and could furthermore provide a basis for wood species dependent technology optimization in the future.
Keywords: Thermally treated wood, chemical properties, mechanical properties, linear correlations

INTRODUCTION

Thermal modification beneficially changes the dimensional stability, hygroscopicity and biological durability of wood (Boonstra *et al*. 2007, Militz 2008). The modification treatment is always performed in the temperature range of 180°C to 240°C. Increasing temperatures result in the transformation of solid into volatile compounds (degradation of extractives and hemicellulose, production and transformation of radicals originating from lignin, etc.) (Burmester 1975, Hakkou *et al*. 2005, Windeisen *et al*. 2003, Windeisen and Wegener 2008). In the range from 100°C to 250/280°C a mild pyrolysis generally takes place. Above 300°C the rapid degradation of cellulose also contributes to the formation of degradation products (Pfriem 2006).

Various wood species with distinct chemical compositions also behave differently during the thermal modification process (Zaman *et al*. 2000, Militz 2002, Boonstra and Tjeerdsma 2006, Windeisen *et al*. 2007); it is, however, sometimes hard to interpret or interrelate physical changes to exact chemical transformations (Tjeerdsma and Militz 2005, Windeisen *et al*. 2009).

Selected physical properties (density, L-value, moisture content, bending strength, modulus of elasticity) and chemical properties (pH, hemicellulose-, total phenol- and soluble carbohydrate content) have been measured in samples of industrially important wood species that had been thermally modified in an autoclave in N_2 atmosphere. The modification process has been carried out in two steps as described by Giebler (1981) (Step 0: untreated; Step 2 Step 3: treatments with increasing intensity).

The aim of the present work was to find and evaluate relationships between the measured chemical parameters (wood structure) and the physical features by means of linear correlations. By establishing correlations the contribution of the changes in the chemical composition to the physical and technological properties can be investigated and revealed.

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MATERIAL AND METHODS

Material and Treatment

Beech (*Fagus sylvatica* L.), Spruce (*Picea abies* L.), Scots pine (*Pinus sylvestris* L.), and Douglas fir (*Pseudotsuga menziesii* Mirb.) wood samples of approximately equilibrium moisture content were industrially thermally treated in an autoclave under N_2 gas according to the method described by Giebler (1981). The intensity of the modification increases with the number of steps. The two steps of the modification process differ in terms of pressure, residual oxygen content, temperature, and duration. The reference samples were not modified (Step 0). After closing the autoclave vacuum was generated, which was followed by establishment of an overpressured atmosphere using N_2 gas. The initial moisture content of the wood samples was between 8 - 10 %. The mixture of nitrogen, residual oxygen, and steam, which was produced during the treatment, remained in the autoclave during the whole process. Untreated wood (Step 0) and wood samples treated in two steps (Step 2 and Step 3) were tested. Step 1 was not tested, while only the treatment classes 2 and 3 are industrially used. Names come from the factory. The physical and mechanical properties were evaluated approximately 6 months after production, and the chemical parameters after 12 months. The duration of the storage can only have an effect on the volatile organic compound content of the samples, but not on the measured chemical parameters. Table 1 summarizes the wood samples investigated.

Measurement of Physical Properties

All properties investigated have been measured under normal climate conditions at 20°C and 65% relative humidity using wood samples having dimensions of 400 x 20 x 20 mm. Density was measured according to DIN 52182 and moisture content according to DIN 52183 and is indicated in kg/m^3 and in percentage units, respectively. Bending strength and modulus of elasticity (MOE) were evaluated according to DIN 52186. The so-called lightness parameter perpendicular to the wood fibre (L-value) was evaluated by colour measurement using a Minolta Chroma-Meter CR 200 device.

Table 1: Investigated wood samples. Bu and BuP represent beech wood of different origin. Sawn wood produced in different factories (different quality), thermal treatment was the same.

Species	Symbol	Treatment steps
Hardwoods		
Beech	Bu	0, 2,3
Beech	BuP	0, 2,3
Softwoods		
Douglas fir	Dk	0, 2, 3
Pine	Fk	0, 2, 3
Spruce	Fi	0, 2, 3

Approximately 200 g of each wood sample were ground and sieved. The fraction with particle sizes of 0.2 - 0.63 mm was used in all measurements.

Dry mass content

3 g of each of the wood samples were heated at 105°C until constant weight. The moisture content (%) was calculated and is based on dry wood mass.

pH

The pH of the wood samples was determined using the method of Roffael and Kossatz (1981).

Ethanol-toluene soluble extract content.

The ethanol-toluene soluble extract content was measured according to the Tappi standards T 264 cm - 97 and T 204 cm – 97, respectively. The extractive content (%) relates to dry weight (bone dry wood).

Total phenol content
0.025 g of wood sample were extracted in 6 consecutive steps with 80% aqueous methanol using an ultrasonic bath with a temperature of 25°C. Extraction was carried out by applying 8 ml of the extraction solvent for 30 minutes during each step. The extracts were collected in a flask and filled up with pure extraction solvent (80% aqueous methanol) to a final volume of 50 ml. The total phenol content was determined spectrophotometrically according to the method of Folin-Ciocâltău (Singleton and Rossi 1965) using quercetin as standard. The results have been expressed in mmol quercetin/100 g dry wood. The measurements were carried out in triplicate.

Soluble carbohydrate content
The soluble carbohydrate content (mg glucose/g dry wood) was determined from the same extract as used for the total phenol content using the method of Dubois *et al*. (1956) with glucose as standard. The measurements were carried out in triplicate.

Hemicellulose determination
The measurement of the hemicellulose content (% based on dry wood) was carried out using the method of Polyak (1948).

RESULTS AND DISCUSSION

Differences have been noted between the different wood species as to the way they respond to heat treatment. The most important differences were reported between softwoods and hardwoods (Zaman *et al*. 2000, Militz 2002, Hill 2006). Due to these, the wood samples investigated have been grouped and discussed as softwood and hardwood species.

Physical Properties

Table 2 and 3 summarize the physical properties of the investigated hardwood and softwood samples, respectively. After treatment the equilibrium moisture content and the bending strength of the wood samples decreased. The density of the treated wood samples also decreased significantly compared to the untreated samples. It can also be recognized that the thermal treatment gave no important impact on the MOE. The bending strength decreased drastically with intensified treatment (Table 2 and 3). With increasing treatment intensity the L-value was reduced, showing that the colour of wood surface became darker. No significant relationship between the colour and the bending strength could be established. In general an increased treatment intensity decreases the bending strength. Since the intensity of the treatment correlates with the colour of the wood it also can be generally followed that the darker the wood the lower the bending strength (Junghans and Niemz 2005). The strength of wood, however, is also influenced by other parameters.

Table 2: Physical properties of the beech samples

Sample	Density [kg/m³]	Moisture content [%]	L-value	Bending strength [MPa]	Modulus of Elasticity [10⁴ MPa]
Bu0 *27*	738 ± 7	10.9	72.0 ± 0.7	132.8 ± 2.2	1.31 ± 0.029
Bu2 *35*	692 ± 10	8.7	47.9 ± 0.8	76.7 ± 7.5	1.10 ± 0.061
Bu3 *42*	656 ± 10	8.3	35.9 ± 0.5	53.8 ± 4.1	1.17 ± 0.039
BuP0 *27*	685 ± 10	10.5	40.3 ± 0.6	138.1 ± 3.3	1.39 ± 0.041
BuP2 *35*	629 ± 7	7.1	18.5 ± 0.6	101.1 ± 5.4	1.35 ± 0.025
BuP3 *29*	676 ± 5	5.5	11.2 ± 0.4	54.5 ± 3.1	1.29 ± 0.025

The index behind the name indicates the number of samples tested.
The confidence interval is indicated at the p = 0.05 level.

Table 3: Physical properties of the softwood samples

Sample	Density [kg/m³]	Moisture content [%]	L-value	Bending strength [MPa]	Modulus of Elasticity 10^4 [MPa]
Fi0 45	462 ± 15	11.6	61.2 ± 1.6	90.6 ± 4.8	1.22 ± 0.065
Fi1 45	481 ± 24	9.3	29.3 ± 0.7	63.7 ± 4.6	1.31 ± 0.073
Fk0 39	552 ± 17	11.9	79.7 ± 0.6	87.4 ± 3.8	1.21 ± 0.071
Fk1 30	543 ± 16	9.7	56.4 ± 1.0	68.0 ± 7.5	1.25 ± 0.094
Fk2 81	516 ± 9	7.2	47.2 ± 1.3	57.2 ± 5.0	1.25 ± 0.040
Dk0 52	484 ± 13	13.4	72.9 ± 3.0	75.6 ± 3.8	1.13 ± 0.044
Dk1 20	477 ± 16	7.5	57.8 ± 1.0	73.1 ±3.8	1.21 ± 0.054
Dk2 96	466 ± 10	6.8	47.5 ± 0.5	67.5 ± 2.3	1.21 ± 0.039

The index behind the name indicates the number of samples tested.
The confidence interval is indicated at the p = 0.05 level.

Chemical Analyses

The chemical composition of different wood species also shows great variability: the lignin of softwoods is mainly built-up from coniferyl- and p-coumaryl-alcohol, while in the case of hardwoods, the major components are coniferyl- and sinapyl-alcohol (Németh 1997). The hemicellulose fraction of hardwoods is mainly composed of glucuronxylans which are thermally and hydrolytically more instable than glucomannans which are dominant in the hemicelluloses of softwood. A fraction of the hydroxyl groups in hemicelluloses is acetylated (Pfriem 2006). Considering cellulose, there are no significant differences mentioned between various wood species regarding structure and composition (Németh 1997). The largest differences between the chemical composition of different wood species are in the type and content of extractives.

Regarding the measured data for hardwoods it can be observed that the pH decreases (Table 4) with Step 1 treated samples of beech compared to Step 0. There is a remarkable change in the total phenol content in all of the samples. The concentration of the phenolic compounds consistently increases from Step 0 to Step 2. A good correlation can be established between the total phenol concentration and the ethanol-toluene soluble extractive content (R^2=0.955, Fig. 1). The correlation proves that phenolic compounds with low molar mass and good solubility in the ethanol-toluene mixture are formed during the treatment. The only explanation for this is a partly decomposition of lignin, which stretches over wide temperature ranges.

A decrease in the hemicellulose concentration can be observed with increased intensity of the treatment in all the hardwood samples.

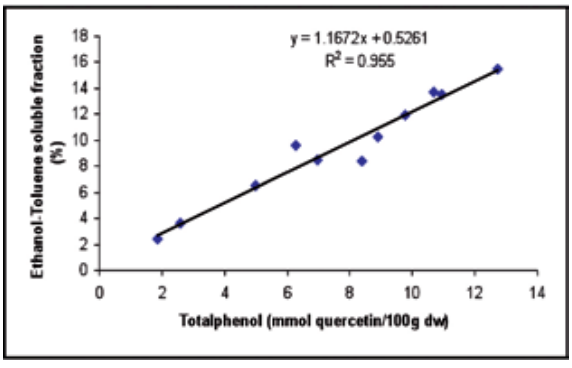

Fig. 1: Correlation between the total phenol content and the ethanol-toluene soluble fraction for various hardwood samples. Note: The diagram also includes data for other hardwood species than beech (ash, maple). For reference data, see: Hofmann *et al.* 2008.

Table 4: pH, ethanol-toluene soluble extract content, total phenol content, soluble carbohydrate content, and hemicellulose fraction of the investigated beech samples.

	Bu0	Bu2	Bu3	BuP0	BuP2	BuP3
pH	4.16	3.57	3.62	4.46	3.80	3.65
Content of ethanol-toluene solid extract [%]	3.6	8.5	11.9	2.4	9.6	13.7
Content of total phenol [mmol/100g dry wood]	2.59 ± 0.1	6.95 ± 0.25	9.78 ± 0.64	1.85 ±0.10	6.27 ±0.08	10.67 ±0.30
Content of soluble carbohydrates [mg/g dry wood]	23.2 ± 2.2	199.2 ± 27.1	65.1 ± 7.3	16.2 ± 0.9	81.2 ±12.4	61.2 ±15.8
Fraction of hemicellulose [%]	13.6	10.4	5.6	13.9	13.9	5.9

The confidence interval is indicated at the p = 0.05 level.

During the modification process, the hemicellulose content also changes in softwood samples. Comparing softwood and hardwood samples, it can additionally be concluded that during the treatment both, total phenol concentration and soluble carbohydrate content change more extensively in the hardwood samples than in the softwood samples.
The softwood samples showed also a decrease in pH values with the higher intensity of the thermal treatment. There is, however, no correlation between the total phenol concentration and the ethanol-toluene soluble extractives content (R^2=0.0755) in softwood samples as it was found for hardwood samples (Fig. 2).

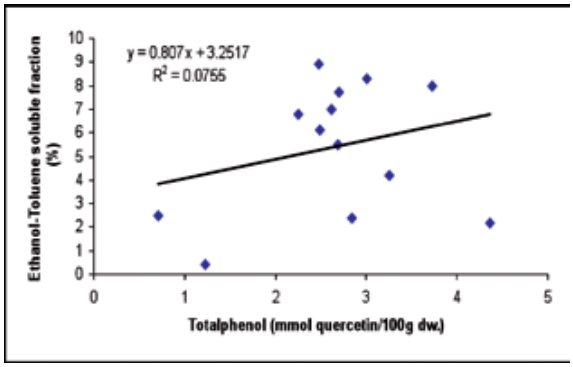

Fig. 2: Correlation between the total phenol content and the ethanol-toluene soluble fraction for various softwood samples.

Table 5: pH, ethanol-toluene soluble extract content, total phenol content, soluble carbohydrate content, and hemicellulose fraction of the investigated softwood samples.

	Dk0	Dk2	Dk32	Fk0	Fk2	Fk3	Fi0	Fi2
pH	3.64	3.63	3.52	4.10	3.92	3.59	4.15	3.56
Content of ethanol-toluene extract [%]	2.2	8.9	8.3	6.8	7	8	2.4	4.2
Content of total phenol [mmol/100g dry wood]	4.36 ±0.07	2.47 ±0.08	3.01 ±0.03	2.25 ±0.02	2.62 ±0.11	3.72 ±0.12	2.84 ±0.05	3.25 ±0.07
Content of soluble carbohydrates [mg/g dry wood]	27.99 ±2.04	118.9 ±23.3	102.6 ±15.5	15.84 ±1.96	66.46 ±1.56	55.64 ±8.37	24.89 ±2.41	45.05 ±1.84
Fraction of hemicellulose [%]	17.2	13.3	4.9	6.8	8.7	7.3	6.9	8.0

The confidence interval is indicated at the p=0.05 level.

Correlation between physical and chemical properties

The physical and technological properties of wood are altered during the thermal treatment. The change in the physical properties of wood is realized through the transformation of chemical components and through the alteration of the wood structure. Yet, which physical property is influenced by the change of which chemical parameter can only be guessed on the basis of wood chemical- and wood physical knowledge.

Possible relationships can be quantified using mathematical tools (i.e. linear correlations) in order to predict the influence of the chemical changes on various technological features. The correlation coefficient (R) alters between -1 and +1. The value of +1 indicates a positive correlation, while -1 indicates a negative correlation. A zero value means that there is neither a positive nor a negative correlation between the two parameters investigated (Inczédy 1979). Table 6 summarizes the most important correlations between the measured physical and chemical parameters. Due to the previously discussed differences between softwood and hardwood, these two groups are evaluated separately.

Table 6: Linear correlations between physical and chemical parameters for hardwood samples. Note: The calculation here also involves data for other hardwood species than beech (ash, maple). For reference data, see: Hofmann *et al*. 2008. R: correlation coefficient.

Parameter 1	Parameter 2	R
Content of ethanol-toluene extract	Content of total phenol	0.9773
Content of total phenol	Bending strength	-0.9297
Content of total phenol	Moisture content	-0.8923
Content of total phenol	Content of hemicellulose	-0.8776
Content of hemicellulose	Bending strength	0.8383
Content of total phenol	Density	-0.6574
Content of hemicellulose	Moisture content	0.6319

The bending strength decreases (R = -0.9297) for hardwood (Table 6) with increasing total phenol content. While heating up wood, the lignin is first softens (70 - 80ºC); then radicals are formed in the depolymerisation reactions (120 - 130ºC) that in turn are recombined (140 - 200ºC) to compounds that presumably have lower polarity (Windeisen and Wegener 2008). In this process the hygroscopicity of lignin decreases significantly (Pecina 1985, Tjeerdsma *et al*. 1998). During the depolymerisation reactions, simple phenolic compounds can be formed that are easily soluble in the ethanol-toluene extraction mixture, as discussed above. This could be the reason for the increasing total phenol concentrations of the wood samples. The transformation of lignin (i.e. increasing amounts on phenolic extractives in the wood) seems to be closely correlated with the decreased bending strength in the investigated hardwood species.

The transformation of lignin and the increase in the concentration of the low molar mass phenolic compounds contributes to the reduced water adsorption (lower equilibrium moisture content) in the thermally modified hardwood samples (R = -0.8923). The lower equilibrium moisture and, hence, reduced swelling and shrinkage is characteristic of the thermally modified wood (Pfriem 2006).
A reduction in the hemicelluloses content is mesured (R = -0.8776) with an increase in the total phenol concentration. During the thermal degradation of hemicelluloses the lignin-carbohydrate connections are also cleaved. The cleavage leads to the easier depolymerisation of this non-carbohydrate-bonded lignin fraction (Pfriem 2006), yielding simple phenolic compounds.

It seems that there is also a clear connection between a reduction in bending strength and hemicelluloses content (R = 0.8383), which proves the physical changes that take place during the degradation of hemicelluloses in hardwood samples. The strong degradation of hemicelluloses through thermal treatment of wood has already been proven by several researchers (Sweet and Winandy 1999, Winandy and Lebow 2001, Pfriem and Wagenführ 2007).

Compared to hardwood samples only few good correlations between physical and chemical parameters exist for softwood (Table 7). Softwoods are characterized by more thermally stable hemicelluloses that are mainly built from glucomannans; beside of this different types and quantities of extractives are given compared to hardwood. With increasing soluble sugar concentration, the MOE (R= - 0.7330) and also the bending strength (R = -0.6695) decreased. During degradation of hemicelluloses soluble carbohydrate compounds are produced in wood. The structure of the wood is altered, which probably results in the reduced MOE and the lower bending strength. The correlation between the total phenol content and the bending strength (R= - 0.5852) is also much lower than that of hardwood (R = -0.9297). Nervertheless, further investigations are needed to reveal other possible relationships between physical and chemical parameters concerning thermally modified softwoods.

Table 7: Linear correlations between physical and chemical parameters for softwood. R: correlation coefficient

Chemical parameter 1	Physical parameter 2	R
Content of soluble carbohydrates	MOE	-0.7330
Content of soluble carbohydrates	Bending strength	-0.6695
Content of total phenol	Bending strength	-0.5852
Content of total phenol	L-value	-0.5272
Content of hemicelluloses	Bending strength	0.0520

Figures 3, 4 and 5 depict typical correlation charts between physical and chemical variables for hardwood and softwood samples.

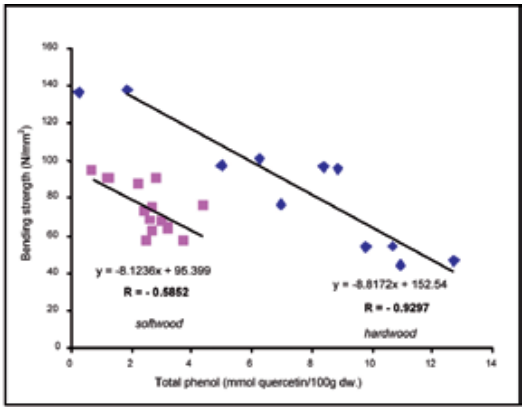

Fig. 3: Correlation between the total phenol content and the bending strength for hardwood (◆) and softwood (■) samples and the respective R values. Note: The diagram also includes data for other hardwood species than beech (ash, maple). For reference data, see: Hofmann *et al*. 2008.

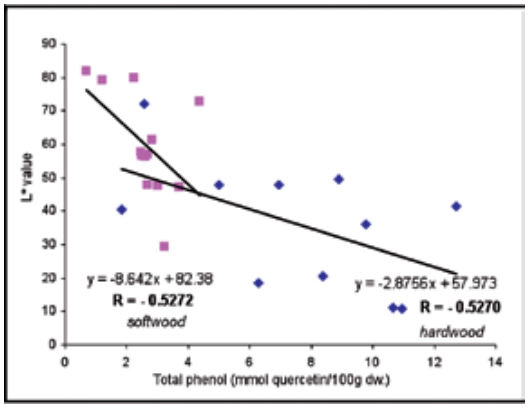

Fig. 4: Correlation between the total phenol content and the L* value for hardwood (◆) and softwood (■) samples and the respective R values. Note: The diagram also includes data for other hardwood species than beech (ash, maple). For reference data, see: Hofmann *et al*. 2008.

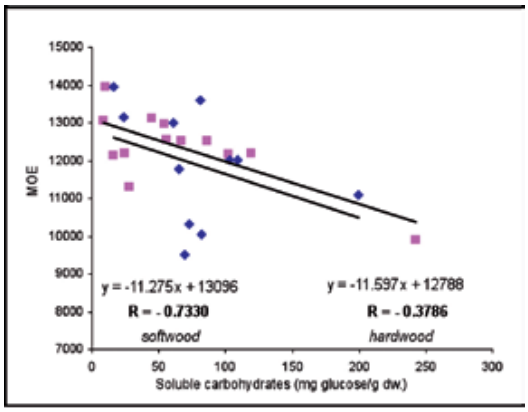

Fig. 5: Correlation between the soluble carbohydrates content and the MOE for hardwood (◆) and softwood (■) samples and the respective R values. Note: The diagram also includes data for other hardwood species than beech (ash, maple). For reference data, see: Hofmann *et al*. 2008.

CONCLUSIONS

Softwood and hardwood samples behave differently during the thermal modification process carried out in a N_2 atmosphere autoclave. The different behaviour can be assigned to the different characteristic chemical composition of each of the wood types.

Using correlations, the chemical reactions taking place during the thermal modification process can be related to the changes in the physical properties of the wood material. Very strong correlations have been found for hardwood between bending strength and total phenol content (R=-0.9297), bending strength and hemicellulose content (R=0.8383) and total phenol content and moisture content (R=-0.8923). For softwood samples poor correlations have been found, with the best between souble carbohydrate content and MOE (R=-0.7330). The results suggest that the change in the physical and technological properties are influenced differently by the change of the chemical parameters in different wood species. The presented results can serve as a basis for further research of the behaviour of different types of wood tissues during thermal treatment and could provide a basis for wood species dependent technology optimization in the future.

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