

# Effects of Thermal Modification on the Properties of Two Vietnamese Bamboo Species.

## Part II: Effects on Chemical Composition

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Bamboo is a very interesting bio resource for building materials because of its combination of strength properties and low density. However, its susceptibility to fungi and insects is problematic. Thermal modification is used in Vietnam to improve the durability and dimensional stability of bamboo. Changes in the chemical composition during this modification are the reason for such physical changes as color, equilibrium moisture content, and strength. This paper will describe the changes in the chemical composition depending on the modification conditions. Furthermore correlations between chemical composition and physical properties, which are presented in Part I, are shown.

*Keywords:* Bamboo; Thermal modification; *Dendrocalamus barbatus*; *Dendrocalamus asper*; Changes in chemical composition

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## INTRODUCTION

Thermal modification is a typical method used to increase the durability and dimensional stability of natural materials such as wood (Stefke and Teischinger 2002). This method is also a promising and environmentally friendly possibility for bamboo modification (Kamdern *et al.* 2002; Militz 2002; Leithoff and Peek 2001; Sulaiman *et al.* 2006). During thermal modification, many chemical reactions can take place, depending on the modification parameters like atmosphere, temperature, and duration (Fengel and Wegener 1989). The resulting changes in the content and chemical composition of the main components cellulose, hemicelluloses, and lignin as well as extractives have an important influence on the physical and mechanical properties.

Some knowledge about such correlations exists for ThermoWood<sup>®</sup>. Endothermic drying is obtained between 120 °C and 150 °C and thermal decomposition occurs at temperatures above 150 °C (Patzelt *et al.* 2002). Polyoses as well as lignin and extractives, show degradation and conversion reactions with various intermediates depending on the modification parameters. The different wood components have varying degradation behavior with respect to the decomposition temperature and rate. Some degradation of hemicelluloses already takes place at 120 °C (Wienhaus 1999; Patzelt *et al.* 2002). Militz and Tjeerdsma (2001) obtained separation of acetyl groups from hemicelluloses between 165 °C and 185 °C. The formed acetic acid catalyzes the further decomposition of holocellulose (Fengel and Wegener 1989; Garotte *et al.* 1999). With

increasing temperature, the decomposition rate becomes higher. From 200 °C, degradation is very intense, and at 225 °C the hemicelluloses structure is nearly completely decomposed (Patzelt *et al.* 2002; Kaygin *et al.* 2009). Cellulose degradation is slow below 300 °C (Sandermann and Augustin 1963; Topf 1971). The formation of very stable compounds takes place by condensation and dehydration reactions within lignin between 100 °C and 200 °C (Fengel and Wegener 1989). Additionally, the splitting of ether bonds and thermo-oxidative processes take place. Several extractives, which are mainly volatile hydrocarbons, are rejected from wood below 100 °C (Fengel and Wegener 1989). Fats, waxes, and resins can also decompose at higher temperatures (Nuopponen *et al.* 2003). Bamboo components are different from wood components in chemical composition and structure. Varying behavior is expected during thermal modification. Until now, there have been published results for changes in chemical composition of bamboo caused by thermal modification only for treatment in oil at temperatures between 140 °C and 220 °C (Leithof and Peek 2001; Salim and Wahab 2008). Salim and Wahab (2008) obtained a reduction of holocellulose above 180 °C, which is mainly caused by the degradation of cellulose. Hemicelluloses are not affected by the treatment under these conditions. Content of lignin decreases slightly up to 180 °C. At higher temperatures an increase of lignin was obtained. Starch is significantly degraded at 180 °C and 220 °C.

The objective of this research was the determination of the changes in physical and chemical properties indicated by thermal modification in nitrogen atmosphere. Furthermore the correlations between these properties were of interest. The changes of physical and mechanical properties are described in part I of this publication (part I: Nguyen *et al.* 2012). In the present paper the changes in chemical composition are described. Correlations between chemical and physical properties are discussed for some important examples.

## EXPERIMENTAL

### Materials

Two species of Vietnamese bamboo were chosen for these investigations. These species were *Dendrocalamus barbatus* (*D. barbatus*) and *Dendrocalamus asper* (*D. asper*). The culms were harvested in the area of Tan Lac, province Hoa Binh, Vietnam. They were about 3 years old and 15 m high. Only the internodes of the middle part of the culms (from 5 to 10 m on high) were investigated. Each internode was cut in 5 slices. Each slice was split into 2 parts – one part was used for the modification and the other part served as an unmodified control sample.

The samples were modified for 2 h or 5 h at temperatures between 130 °C and 220 °C. All samples were dried in three steps (40 °C, 60 °C, and 80 °C) before thermal modification. The modification was carried out in a closable treatment chamber. First, the chamber (with samples) was evacuated to 200 mbar and then filled with nitrogen (inert gas). After that, the chamber was heated to 100 °C with a heating rate of 0.67 K/min. Then, the heating rate was decreased to 0.33 K/min for heating to the final temperature. At this temperature, the samples were held for 2 h or 5 h. Cooling down to room temperature took place within 7 h. The modified samples and the untreated twin samples

were stored under standard conditions (20 °C, 65 % rh) for 2 weeks before cutting into various specimens for different analyses and tests (Nguyen *et al.* 2012 Part I).

## Methods

Bamboo samples for chemical analyses were chopped and milled to particle sizes below 250 µm after thermal treatment. The dry mass of the samples was determined at 103 °C. All contents of components are referred to in the dry mass.

### *Determination of content of extractives*

Five grams of the sample were extracted with 200 mL of ethanol-toluol mixture (1:1) for 6 h in a soxhlet extractor (TAPPI T 204-em-97). The solvents were removed by rotation evaporation at 40 °C and between 150 and 50 millibars. The residual extractives were dried at 40 °C for quantitative analysis.

### *Determination of composition of extractives*

Two grams of the sample were extracted with 60 mL of different very pure solvents (hexane, ethanol, acetone, and toluene) one after another. The extraction time was 6 h for every solvent. After extraction the solutions were reduced by rotation evaporation to 5 mL. The concentrated solutions were dried with sodium sulphate and analyzed by GC/MS (Agilent HP 6890 plus; Agilent HP 5973 MSD). The measurements were carried out splitless using a dimethylpolysiloxane column.

### *Determination of content of cellulose*

One gram of the extracted sample was boiled three times under reflux in 25 mL of a nitration mixture (10 mL 65 % HNO<sub>3</sub> + 40 mL ethanol) for 1 h. After each step the nitration mixture was separated by filtration and the boiling step was repeated with fresh nitration mixture. After the third step the solid residue was washed with ethanol and hot water and then boiled under reflux in 100 mL water for 30 minutes. Finally, the resulting cellulose was separated by filtration using a glass filter with medium porosity (40 µm), washed acid free and dried at 105 °C (Kürschner and Hoffer 1931)

### *Determination of content of holocellulose*

One half gram of the extracted sample was dispersed in a solution of 60 mL water, 100 µL glacial acetic acid, and 0.5 g sodium chlorite. This mixture was shaking at 70 °C. After 1 h, 100 µL glacial acetic acid and 0.5 g sodium chlorite were added. The mixture was shook five times at the conditions mentioned above. After cooling down, the resulting holocellulose was separated by filtration using a glass filter with medium porosity (40 µm), washed acid free, and dried at 105 °C (TAPPI 149-75).

### *Determination of content of hemicelluloses*

The content of hemicelluloses was determined as the difference between holocellulose and cellulose.

*Determination of composition of hemicelluloses*

A gentle hydrolysis of the separated holocellulose was carried out for analyzing the sugars of hemicelluloses. Holocellulose was swelled in water-free trifluoroacetic acid (TFA) overnight. In the next step, the acid was diluted down to 80%. The samples

were boiled in this solution for 30 min. Then, a further dilution down to 30% TFA and a boiling of this solution for 2 h were performed. After that, the solution was removed by rotating evaporation. The hydrolyzed product was washed by adding 10 mL of water and removing the solution by rotating evaporation. This procedure had to be repeated 15 to 20 times until the samples were acid-free. The air-dried sample was dissolved in water. Afterwards, the resulting solution was analyzed by high-pressure liquid chromatography (HPLC).

*Determination of content of lignin*

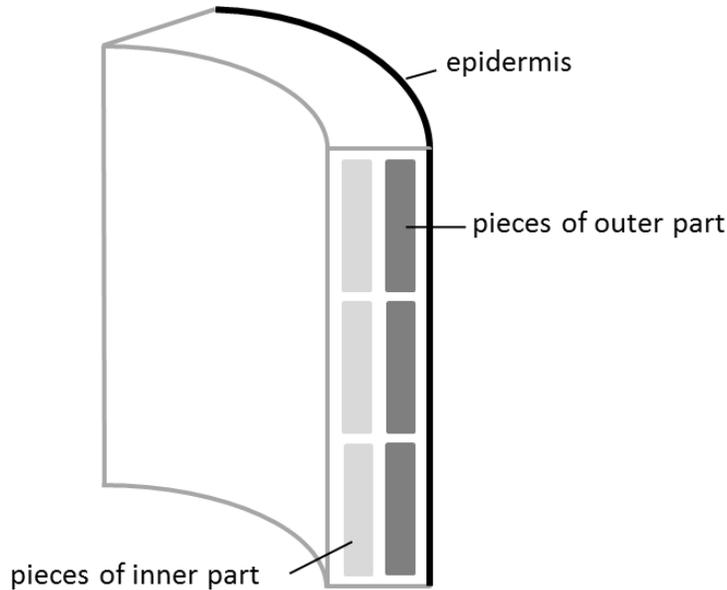
One half gram of the extracted sample was mixed with 20 mL of 72 % sulphuric acid. This mixture was stirred at room temperature for 2 h. After that the mixture was diluted with distilled water to a final volume of 650 mL. This solution was boiled under reflux for 4 h. After cooling down to room temperature the insoluble lignin was separated by filtration using a glass filter of fine porosity (16 µm). The lignin was washed with 500 mL of hot distilled water and dried at 105 °C.

*Determination of content of glucose*

Two grams of the sample were extracted with 80 % ethanol in a soxhlet extractor for 2 h. The ethanol was then removed from the solution by rotation evaporation. The resulting aqueous solution was filled in a volumetric flask (50 or 100 mL). This solution was filtrated using a syringe filter (pore size 0.45 µm). The determination of cellulose content was carried out enzymatically using a glucose kit of R-Biopharm. The extinction of the NADPH was measured at 340 nm (Urospec 2000, Biotech).

*Determination of content of starch*

Three grams of the sample were extracted with 200 mL distilled water in a soxhlet extractor for 4 h. After that the water was removed by rotation evaporation at 60 °C and 100 mbars. The residual starch was hydrolysed by boiling under reflux in 24 mL 52% perchloric acid for 2 h. Then sodium hydroxide was added until the pH was 8. This solution was filtrated using a syringe filter (pore size 0.45 µm). The determination of cellulose content was carried out enzymatically using a glucose kit of R-Biopharm. The extinction of the NADPH was measured at 340 nm (Urospec 2000, Biotech).



**Fig. 1.** Preparation of test pieces for measurement of shock resistance

#### *Determination of shock resistance*

Test pieces for measurements of shock resistance were prepared from the outer and inner side of an internode without the epidermis and the inner wall of the culm (Fig. 1). Pieces had a size of 15 mm length, 10 mm width, and 1.5 mm height. Shock resistance measurements were carried out by Dynstat Dys-e (Feinmechanik Kögel, Germany).

#### *Determination of fracture morphology*

Fracture morphology was investigated on fractures after measurements of shock resistance. The fractures were assessed by Scanning Electron Microscopy (SEM) using a JOEL T330 A on the institute of Forest Utilization (TU Dresden, Germany).

## RESULTS AND DISCUSSION

The chemical composition of a natural bamboo species depends on different parameters such as the growing conditions, age of culms, culms height, and harvesting time (Liese 1985). The content of cellulose and lignin was found to be similar for both species. Differences were found for hemicelluloses, extractives, and ash content. Whereas *D. barbatus* shows slightly higher hemicelluloses and extractive contents, *D. asper* has higher ash content.

The contents of each component were changed during thermal treatment, but in various scales. The contents of the different components depending on modification conditions are listed in Tables 1 and 2.

**Table 1.** Contents of Chemical Components of *D. asper*

Thermal treatment	Extractives	Holo-cellulose	Cellulose	Hemi-cellulose	Lignin	Glucose	Starch
Untreated	6.83	71.3	49.41	21.89	24.94	5.12	1.17
2 h 130 °C	6.01	69.33	49.81	19.52	26.13	3.64	1.99
5 h 130 °C	5.28	67.12	49.12	18.00	29.34	3.69	1.93
5 h 160 °C	2.37	65.08	48.51	16.57	32.01	0.62	1.45
2 h 180 °C	3.02	65.43	47.67	17.76	32.99	0	0.64
5 h 180 °C	2.97	63.79	47.01	16.78	34.89	0	0.8

**Table 2.** Contents of Chemical Components of *D. barbatus*

Thermal treatment	Extractives	Holo cellulose	Cellulose	Hemi-cellulose	Lignin	Glucose	Starch
Untreated	7.09	74.81	50.7	24.11	25.19	2.76	1.13
2 h 130 °C	6.49	73.98	49.79	24.19	26.12	1.95	1.21
5 h 130 °C	4.44	72.34	49.05	23.29	25.97	1.12	1.35
5 h 160 °C	3.94	68.45	47.97	20.48	31.89	0.26	1.06
2 h 180 °C	4.17	64.54	46.78	17.76	34.43	0	0.76
5 h 180 °C	3.68	61.97	46.13	15.84	37.22	0	1.27

The general mass loss of the samples up to 180 °C was 4%, which is relatively low. It depends on the temperature and on the culm zone (Part I, Nguyen *et al.* 2012).

Extractives underwent drastic changes such as a remarkable color change of their solutions. The color changed from yellow to orange with increasing temperature for both species. The change in color of *D. barbatus* was more intense than *D. asper*. The modification temperature as well as the modification duration had an influence on the type and degree of the changes. Upon progressive heating, at first there will be a release of volatile organic compounds. So extractives of samples, which were treated at 130 °C and 160 °C contain lower contents of mono terpenes and carbonyl compounds of essential oils. Furthermore the content of mono and disaccharides is lower than in the untreated sample. This indicates a decomposition of these saccharides already at this temperature. The highest mass loss can be obtained at 160 °C. Besides the above mentioned effects, a reduction of aromatic compounds was obtained at 160 °C. Especially the C9-units of lignin show the lowest content and variety of all investigated samples. The mass loss at 180 °C is a little lower because of some linking or condensation reactions. Extractives of these samples contain many furan derivatives, which are formed by the degradation of hemicelluloses and cellulose. In addition, content of aromatic compounds increases and the length of the side chains become shorter. These samples show the highest variety of C9-units of lignin. In contrast to the untreated samples, these

are compounds with short side chains. That means a significant degradation of lignin occurs. Cyclic ketones of mostly chinoide structure are observed at 180 °C. The mass loss of *D. barbatus* was generally lower than for *D. asper*. The higher mass loss for *D. barbatus* is noteworthy at modification conditions of 5 h at 130 °C.

The changes in contents of extractives should have no important influence on the physical and mechanical properties of bamboo. Investigations of color change during thermal treatment showed a significant color difference at 180 °C (Nguyen *et al.* 2012 Part I). This fact correlates with the highest content of chromophoric compounds within the extractives at 180 °C.

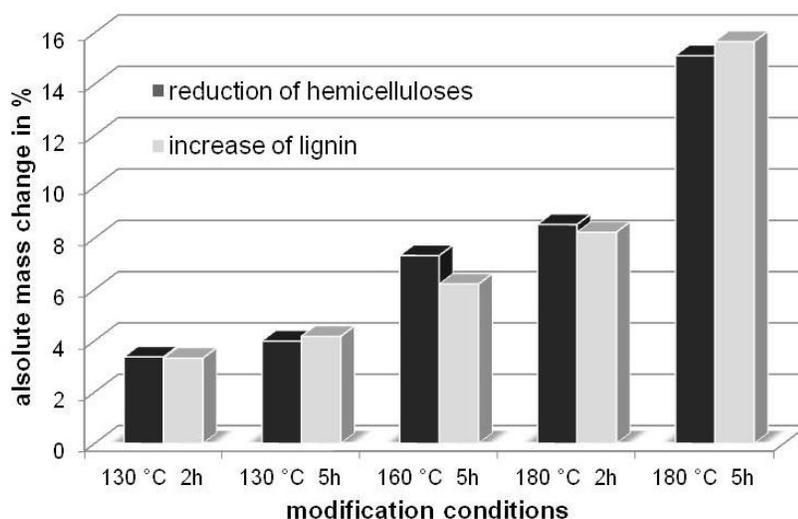
The content of holocellulose showed a continuous decrease during thermal modification. The influence of modification time and duration seemed to be similar. Degradation of holocellulose was stronger for *D. barbatus* than for *D. asper*. Bamboo hemicelluloses contain about 7% acetyl groups (Liese 1985). These groups were separated from the chains by thermal exposure and formed acetic acid. This compound has a catalytic effect on the decomposition of the glycosidic bonds of holocellulose. Holocellulose includes cellulose and hemicelluloses, which have different thermal stabilities.

Cellulose as the main component exhibited a higher thermal stability than hemicelluloses because of its crystallinity. The treated samples showed a continuous decrease during modification. But only a maximum loss of 5% of cellulose was observed up to 180 °C. This was attributed to a shortening of cellulose chains. Roffael and Schaller (1971) studied the degree of polymerization (DP) of cellulose depending on thermal treatment. They found a reduction of DP to a level between 600 and 800 after treatment.

Contrary to the results of Salim and Wahab (2008) a strong reduction of hemicelluloses occurred by thermal modification under nitrogen. Already at 130 °C, the degradation of hemicelluloses took place. A modification of 5 h at 180 °C resulted in a reduction of hemicelluloses of about 33 %. The influence of the modification duration increased with increasing temperature.

The extracted holocellulose of some samples of *D. barbatus* was hydrolyzed gently and the resulting sugars were analyzed by HPLC. Besides glucose, three monomer sugars were found: xylose, arabinose, and galactose. The ratio of sugars in untreated samples was about 18.5 : 1 : 0.6. This ratio shifted to higher xylose content with increasing temperature. This means that arabinose and galactose, which are located in the side chains as substitutes was reduced at first. At 220 °C, galactose was completely removed and most of the other two sugars were removed as well. This fact is in accordance with the results of Patzelt *et al.* (2002) for thermally modified wood.

However, this determined mass loss is not the real mass loss of the sample. The reactions result in different reactive molecules like furfural or radicals (Fengel and Wegener 1989; Nicholas 1985; Walker 2006), which can react with the unsaturated components of hemicelluloses as well as the phenolic components of lignin (Bulian 2009; Scholz 2008).



**Fig. 2.** Comparison between reduction of hemicelluloses and increase of lignin of *D. asper*

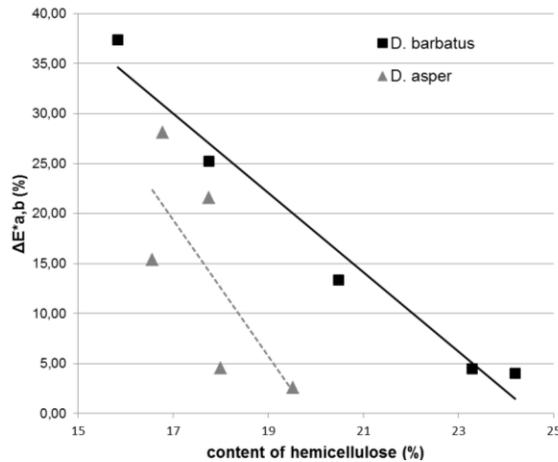
For lignin as the third component, an increase of Klason lignin was found. A comparison between the absolute increase in mass of lignin and the absolute mass loss of hemicelluloses of *D. asper* is illustrated in Fig. 2. As shown, the changes of these two components were similar.

By means of Klason lignin analysis, all components not soluble in concentrated sulphuric acid were determined. Bamboo lignin has a high content of unsaturated aromatic acids, as is typical for grass. These components can react with various decomposition products of hemicelluloses. In addition, linking of furfural or its derivatives with phenolic components of the lignin can take place. Furthermore, polymerization of decomposition products of hemicelluloses is possible. The products of such reactions should be not soluble in sulphuric acid, so they were also detected by the Klason lignin method.

The content of glucose is different for the two species. *D. asper* contains significant more glucose than *D. barbatus*. But there is a strong degradation already at 130 °C for both. Glucose is completely degraded at 180 °C. These results are in accordance with the investigations of the extractives.

The content of starch showed a slight increase at 130 °C in comparison to the untreated samples. This is caused by the degradation of cellulose into short chains, which were also extracted by hot water. A further increase of temperature gave rise to a decreasing content of starch up to 180 °C (2 h). Longer modification duration at this temperature (5 h) resulted in a more or less strong increase of starch content. Starch contains amylose and amylopectin. The water insoluble amylose undergoes a chain shortening and forms water soluble dextrans at higher temperatures. This effect was more intense for *D. barbatus*. Furthermore, the degradation of cellulose into shorter chains under these conditions was also detected.

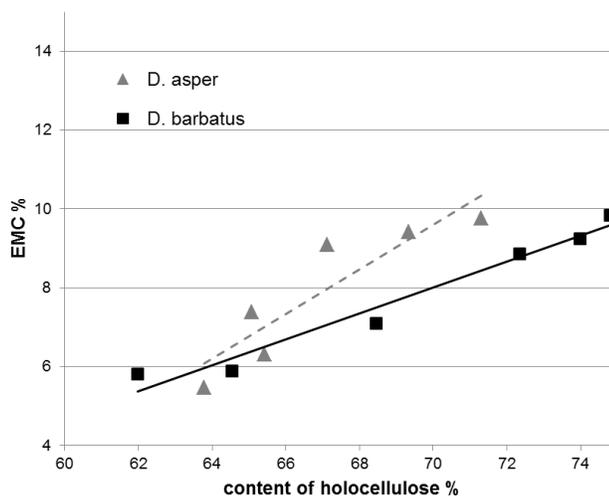
The main components, holocellulose and lignin, have an important influence on the physical and mechanical properties of plants. In Part I of this paper (Nguyen *et al.* 2012) the results of the investigations of the changes of color and equilibrium moisture content (EMC) of these samples are described.



**Fig. 3.** Correlation between content of hemicelluloses and change of color

A strong change of color of the epidermis from yellow-green to dark brown with increasing temperature was found. Besides this effect, the inhomogeneous color of the untreated samples became more homogeneous with increasing temperature. The decomposition of hemicelluloses and the chemical changes of extractives are discussed in the literature as main reasons for such changes (Sunquist 2004). The investigations showed a good correlation between the content of hemicelluloses and the change of color (color difference  $\Delta E^*_{ab}$  – calculated from the lightness  $L^*$ , the green-red coordinate  $a^*$ , and the blue-yellow coordinate  $b^*$  (Nguyen *et al.* 2012, part I). In Fig. 3 the change of color (DE) is shown to depend on the content of holocellulose for *D. barbatus*. At high contents of hemicelluloses (130 °C) the change of color is similar for both species, but with decreasing content of hemicelluloses, *D. barbatus* showed a stronger change of color.

Furthermore, the hydrophilic character will be weakened by a reduction of hydroxyl groups during decomposition of holocelluloses. A decrease of the equilibrium moisture content with increasing temperature was detected for these samples by Nguyen *et al.* (2012, Part I). A very good correlation between the EMC and the content of holocelluloses could be found (Fig. 4).



**Fig. 4.** Correlation between content of hemicelluloses and equilibrium moisture content

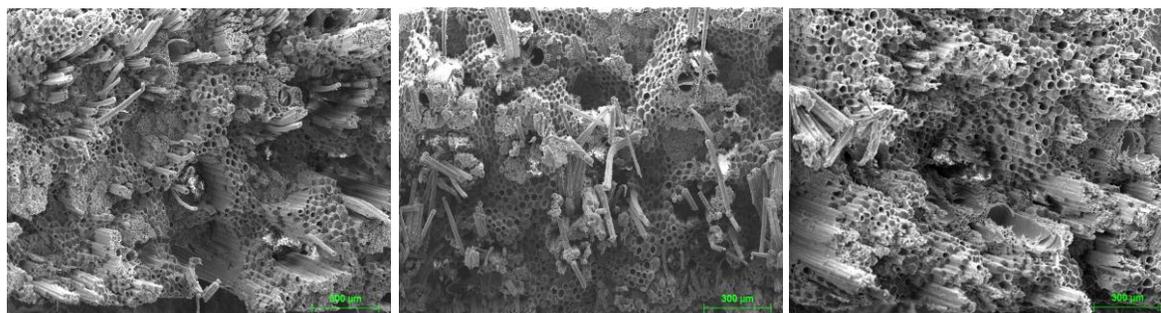
Strength is another important property that is influenced by thermal modification. The shock resistance of these samples was determined, and the fractures were investigated by Scanning Electron Microscopy (SEM). Samples of the inner and outer part of the culm were used for these experiments. The chemical determination of content of cellulose and hemicellulose was carried out after the mechanical and anatomical characterization.

The shock resistance for the non-modified and modified samples is listed in Table 3. A strong decrease of the shock resistance with increasing modification temperature was found. Untreated samples show a high variance of data within the different pieces of one sample. This variance became smaller with increasing temperature. Pieces of the outer part had mainly a slightly higher shock resistance than the inner part, because of their higher fiber content.

**Table 3.** Shock Resistance of Unmodified and Modified Samples

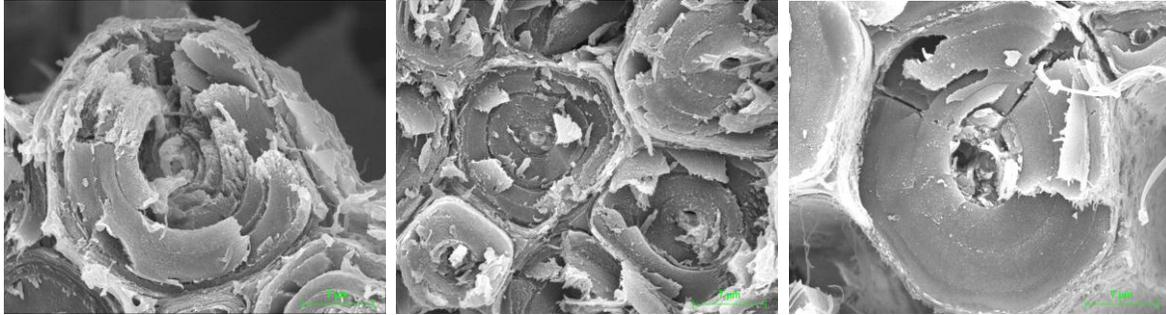
Modification	Culm	Part of culm	Investigated pieces	Shock resistance (average) J/cm <sup>2</sup>	Variance
no	1	inner	10	2.786	1.966 – 3.875
		outer	11	3.274	2.021 – 4.231
no	2	inner	12	2.529	1.633 – 3.449
		outer	12	2.696	2.007 – 4.601
5 h 130 °C	1	inner	7	1.655	1.338 – 1.975
		outer	4	2.733	2.395 – 2.916
5 h 160 °C	1	inner	2	1.082	1.034 – 1.130
		outer	12	0.695	0.623 – 0.833
5 h 180 °C	1	inner	7	0.940	0.626 – 1.296
		outer	9	1.594	0.914 – 2.373
5 h 220 °C	2	inner	12	0.791	0.510 – 0.904
		outer	11	0.672	0.436 – 1.216

Untreated samples showed typically long fractures (Fig. 5). There were fiber bundles with similar fracture planes, but the fracture planes of parenchyma cells and fibers were very different. With increasing temperature the fiber fractures became shorter. Some individual long fibers were found at 160 °C, but there were few fiber bundles. Similar fracture planes of parenchyma cells and fibers resulted after modification at 220 °C.

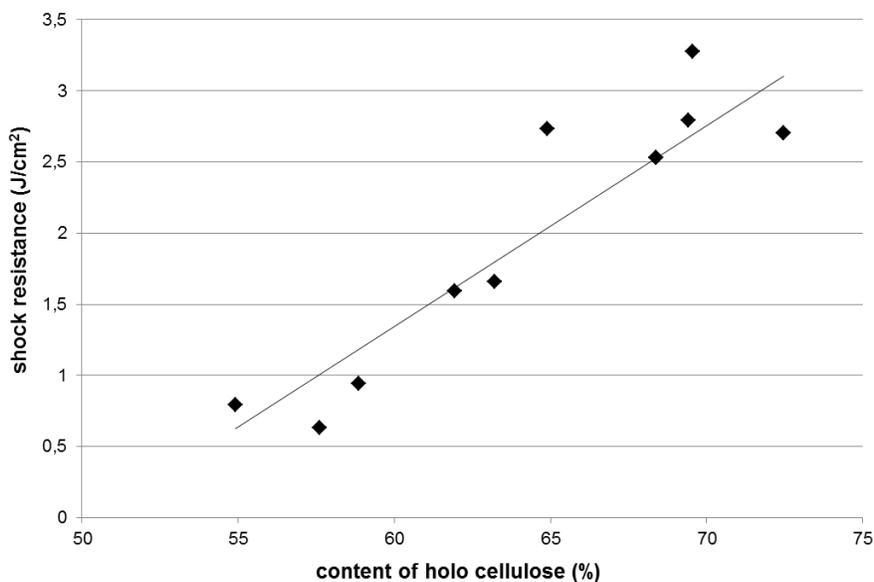


**Fig. 5.** Fracture behavior of *D. barbatus* (outer part) depending on modification temperature

Single fibers of untreated samples showed structured, partially fibrous fracture with different levels of the single lamella. The fracturing became more planar with increasing temperature. This indicates a shortening of the cellulose chains, thus reducing the microfibrillar structure. At 220 °C some tangential cavities were found (Fig. 6).



**Fig. 6.** Fracture behavior of fibers of *D. barbatus* depending on modification temperature



**Fig. 7.** Fracture behavior of fibers of *D. barbatus* depending on modification temperature

Bamboo becomes more brittle with increasing temperature. The samples normally did not break completely in the course of the shock resistance investigations, they simply were bent. Only samples modified at 220 °C showed a complete fracture.

A good correlation between shock resistance and content of holocellulose was found (Fig. 7). Larger deviations were obtained at high holocellulose content for samples with high variances within pieces of one sample.

Both parts of the holocellulose could influence the strength properties. Shortening of cellulose chains means a decrease of the degree of polymerization. This results in lower strength. But loss in strength is also caused by the degradation of hemicelluloses.

By the strong degradation of hemicelluloses, their original function, the linking of cellulose and lignin, is lost.

## CONCLUSIONS

1. The thermal modification of bamboo generates many changes in the chemical composition.
2. The extent of these changes depends on the modification temperature and duration. Treatments at 130 °C cause only slight changes. Significant changes occur by modification above 180 °C. The influence of modification temperature is significantly higher than that of the duration of modification.
3. The most important change is the severe decomposition of hemicelluloses. A mass loss up to 70 % could be observed because of the high content of xylan. Some very reactive components are formed during decomposition. Polymerization reactions and linking of these compounds to lignin results in an increase of the apparent lignin content.
4. The cellulose content decreases slightly by a shortening of cellulose chains.
5. Extractives show a mass loss, which is mainly caused by the evaporation of volatile compounds and water from hydroxyl groups. Condensation reactions and reactions with decomposition products of hemicelluloses induce the increase and darker colour of the extractives of samples modified at higher temperatures. Decomposition products of cellulose, hemicelluloses, and lignin were found within the extractives.
6. Changes in chemical compositions influence the physical and mechanical properties. Good correlations between color, EMC, and the content of hemicelluloses were found.
7. Correlations between shock resistance and content of holocellulose confirm the strong dependence of strength properties from the chemical composition. The increasing brittleness of the modified bamboo with stronger modification conditions manifests itself as a progressive increase in the amount of shorter fractures.

## ACKNOWLEDGMENTS

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