

FTIR-ATR SPECTROSCOPIC ANALYSIS OF CHANGES IN FIBER PROPERTIES DURING INSULATING FIBERBOARD MANUFACTURE OF BEECH WOOD

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Abstract. Fourier transform infrared–attenuated total reflectance (FTIR-ATR) spectroscopy was applied to trace changes in chemical fiber properties during the production process of insulating fiber mats. In combination with cluster analysis, FTIR spectra were used to interpret the homogeneity of the products. Beech wood (*Fagus sylvatica* L.) was used as a novel sustainable material for fiberboard production. The insulating fiberboards were either processed without binder or with potato pulp or potato starch as renewable binders and dried in a dryer or a microwave. FTIR spectral analyses revealed chemical modifications at the O-H association band of carbohydrates that distinguished the two different drying methods. Additions of plant-based renewable binders diminished the absorbance of the resulting products at characteristic wavenumbers in the IR. These decreases were closely correlated with the amount of added binder and thus have the potential to quantify binder additions to the fiberboards. Cluster analysis grouped FTIR spectra of samples from different production steps or processes correctly and therefore is an effective and simple technique for quality control of insulating fiberboards from renewable resources.

Keywords: Binders, cluster analysis, FTIR-ATR spectroscopy, beech, potato pulp, potato starch, hardwood, low-density wood fiberboard, renewable resources.

INTRODUCTION

Innovative thermal insulators based on renewable resources are still niche products (Sedlbauer 2004). To date, some insulation materials based on plant products such as wood and palm fibers,

bark, rice straw and husks have been tested (Mishra et al 1986; Richter 1993; Scheiding 2000, 2002; Han-Seung et al 2002; Al-Sulaiman 2003; Naundorf et al 2004). However, overall, research is limited for the chemical, mechanical, and technological characteristics of these insulants (Sedlbauer 2004).

Wood fibers are strong, lightweight, nonabrasive, abundant, and cost-effective (Clemons

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2002). Once processed to insulating wood fiberboards, they are suitable for internal and external use as thermal and acoustic insulation on floors, walls, and ceilings (Moser et al 2003). Compared with conventional insulants, mostly based on polystyrene and polyurethane produced of mineral oil (Kleinhempel 2005), low-density fiberboards of wood fibers have advantages of exceptional thermal insulation and permit vapor diffusion (Sedlbauer 2004).

Insulating wood fiberboards can be produced in wet or dry processes (Moser et al 2003; Chapman 2006). The dry process requires an admixture of adhesives (Moser et al 2003). In the wet process, adhesives may be added, but depending on fiber length, they may not be essential because lignin may flow and fill gaps; in addition, crosslinking has been suggested to occur as a result of the plasticization of lignin and formation of hydrogen bonds (Makas 2006). To avoid the use of adhesive formulations, petroleum-based products may be substituted by binders from renewable resources such as potato pulp or potato starch. Potato pulp is a byproduct of industrial starch production and consists of pectin (17%) and other cell wall components as well as residual starch (Mayer and Hillebrandt 1997; Mayer 1998). These bio-based binders afford the degradability of the final products (Smok 1999; Müller 2005). To date, there is no information of how these binders influence the chemistry of the wood fibers for fiberboard.

One of the most important steps in wood processing is drying (Leiker and Adamska 2004). The most common industrial method for drying fiber mats is in belt dryers in the range of 160°–220°C (Kutschera and Winter 2006). However, drying by microwave energy is much faster without deteriorating the quality of the dried products (Hansson and Antti 2003).

To date, spruce and pine are the main resources for derived timber products (Wagenführ 2000). Because the conventional resources for wood panels are becoming limited, there is a need to introduce materials from lower-quality tree spe-

cies into these production processes. Novel wood products are currently being developed using European beech (*Fagus sylvatica* L.). Beech cultivation is increasing in Germany, because this species plays a major role in all nature-oriented silvicultural concepts as the potentially dominant species in most forests of middle Europe (Ellenberg 1986). Thus, it is expected that this wood resource will increase in the near future. To generate value-added products from low-quality wood, the use of beech fibers for novel commodities is currently being investigated. Beech has not been used in wood-processing industries before because of its high density (Wagenführ 2000) and also because of the risk of allergies and cancer from potentially hazardous dusts generated during particle and fiber production (Kloeser et al 2008). However, wet production processes may circumvent the latter problem because dust formation is avoided.

The aim of this work was to investigate changes in chemical properties of beech fibers during processing of insulating wood fiberboards. To characterize the influence of fiber processing on the chemical composition of the fibers, Fourier transform infrared–attenuated total reflectance (FTIR-ATR) spectroscopy was applied. FTIR spectroscopy has been used for determination of molecular structures, identification of compounds in biological samples, and investigation of complex polymers (Arndt et al 1999; Kacuráková and Wilson 2001) and therefore is a powerful tool for wood analysis (Fengel and Wegener 2003). In combination with ATR, FTIR analysis permits reproducible qualitative and quantitative analysis of samples with little or no sample preparation (Bukowski and Monti 2007; Naumann et al 2008). We used FTIR-ATR to trace the changes in fiber chemistry during the production process of insulating fiber mats of beech wood. The goals of this study were to identify chemical alterations caused by the addition of different binders or drying methods to beech fibers and to determine if chemical changes during processing of fiberboards could

be used to classify the spectra into clusters according to their spectral heterogeneity.

MATERIAL AND METHODS

Wood Materials

Ten 56-yr-old beech trees were harvested in 2006 in the city forest of Schmallenberg (North Rhine-Westphalia, 51°14'29" N, 8°23'50" E), compartment 238a. The forest stand, established by planting and representing a mixture of *Fagus sylvatica* (40%), *Abies grandis* (50%), and *Pseudotsuga menziesii* (10%), has west to northwest orientation at an altitude of 600 m and is located on a steep slope. The soil is brown earth with limited nutrient supply. At harvest, the mean height and breast height diameter of beech were 16 m and 18 cm, respectively.

Production of Beech Fibers and Insulating Fiberboards

The stems of beech were debarked manually with a drawing knife and then cleaved. The wood pieces were processed into wood chips using a drum chipper (Klöckner Trommelhacker KTH 120 × 400 H2WT; Klöckner Wood Technology GmbH, Hirtscheid, Germany). For pulping, the chips were weighed and put into a pressurized laboratory refiner (Laboratory Refiner Type 12; Andritz AG, Graz, Austria). Without further pretreatment, the chips were pulped for 5 min at 150°C and a pressure of 480 kPa, yielding thermomechanical pulp. Afterward, the fibers were dried in a gas flash-tube dryer at 125°C for 3 s to a target moisture of 8%. Ten samples of fibers were taken randomly from different locations of the fiber packages. The fibers were milled to a fine powder (MM 2000; Retsch, Haan, Germany) for 5 min at 50 rpm. The speed was raised slowly during the next 5 min to 90 rpm. The entire milling process took 10 min for each sample.

Different types of low-density fiberboards were produced in the wet process. One type was completely free of any binder (control); the other types were treated with increasing amounts of

binder containing 10, 20, and 30% potato starch (Avebe Kartoffelstärke nativ; Prignitz/Wendland GmbH, Dallmin, Germany) or potato pulp (KF 200 dehydralisiert; Emsland-Stärke GmbH, Emlichheim, Germany). Potato pulp contained 30% (w/w) starch. The shortness of beech fibers necessitates an admixture of binders to improve the bonds among the fibers and to increase the strength values of the fiber mat.

A suspension of 500 g fibers in 15 L of water was produced. Depending on the type of board, 1 L of water was mixed with 50, 100, or 150 g [10–30% (w/w)] potato pulp or 50, 100, or 150 g [10–30% (w/w)] potato starch and admixed to the water–fiber suspension. The suspension was stirred for 1 h to soak the fibers. Afterward, the suspension was poured on a sieve in a self-constructed casting mold (length: 50 cm, width: 30 cm, height: 60 cm). The effluent was discarded, leading to pancaking of the fibers, generating the fiber mat. The mat was dehydrated in a cold press for 2 min at 10 MPa. Subsequently, mats with different binder percentages, a target thickness of 20 mm, and a moisture content of 100–120% were dried either for 7 min by microwave (MWDA 6 × 1.1 kW; Fricke und Mallah Microwave Technology GmbH, Peine, Germany) at 6600 W or for 2 h in a dryer (UNE 800; Memmert GmbH & Co. KG, Schwabach, Germany) at 170°C. The board densities (Table 1) resulted from the particular type of binder, the binder percentage, and the moisture content of the mats before drying. For each binder percentage and particular type of drying, two boards were taken of which ten specimens were analyzed.

Table 1. Raw densities of the insulating wood fiberboards of beech in dependency on the type and amount of binder.

Type of binder	Binder (%)	Raw density of the fiberboard (kg/m ³)
Potato starch	10	181
Potato starch	20	198
Potato starch	30	213
Potato pulp	10	173
Potato pulp	20	196
Potato pulp	30	226
No binder	0	163

FTIR-ATR Spectroscopy and Cluster Analysis

FTIR-ATR spectra were recorded in the wavenumber range from 4500–600 cm^{-1} with an Equinox 55 spectrometer (Bruker Optics, Ettlingen, Germany) including a deuterium triglycinesulfate detector and an attached ATR-unit (DuraSamplIR, SensIR Europe, Warrington, UK). A resolution of 4 cm^{-1} and 32 scans/sample was used. Specimens of all fiberboards as described previously (length: 50 mm, width: 30 mm, thickness: 20 mm) were investigated. If not indicated otherwise, ten individual samples were analyzed per experimental variable, yielding 100 spectra. Because of the high spectral homogeneity of potato starch and potato pulp, three FTIR-ATR measurements were performed on each. Cluster analysis was used to investigate intrinsic relationships between sample properties. According to the spectral heterogeneity, spectra were classified into clusters or classes in dendrograms. The first derivative and vector

normalization (nine smoothing points) were used as a pretreatment method for the spectra, applying a standard method for creating the distance matrix with the OPUS 6.5 software (Bruker Optics, Ettlingen, Germany). For calculating the spectral distances, Ward's algorithm was applied. The analyzed wavenumber range was 1800–600 cm^{-1} .

RESULTS

FTIR Spectra to Determine the Influence of Fiber Processing

To investigate the influence of fiber processing on fiber properties, FTIR spectra of fibers, pure binders, and low-density fiberboards produced without binder or with pulp or starch as binders were analyzed in the fingerprint region from 1800–850 cm^{-1} (Fig 1). Major spectral changes (bands 1 to 16 in Fig 1) were assigned to chemical bonds using published data (Table 2). Fiberboards free of binders had the highest absor-

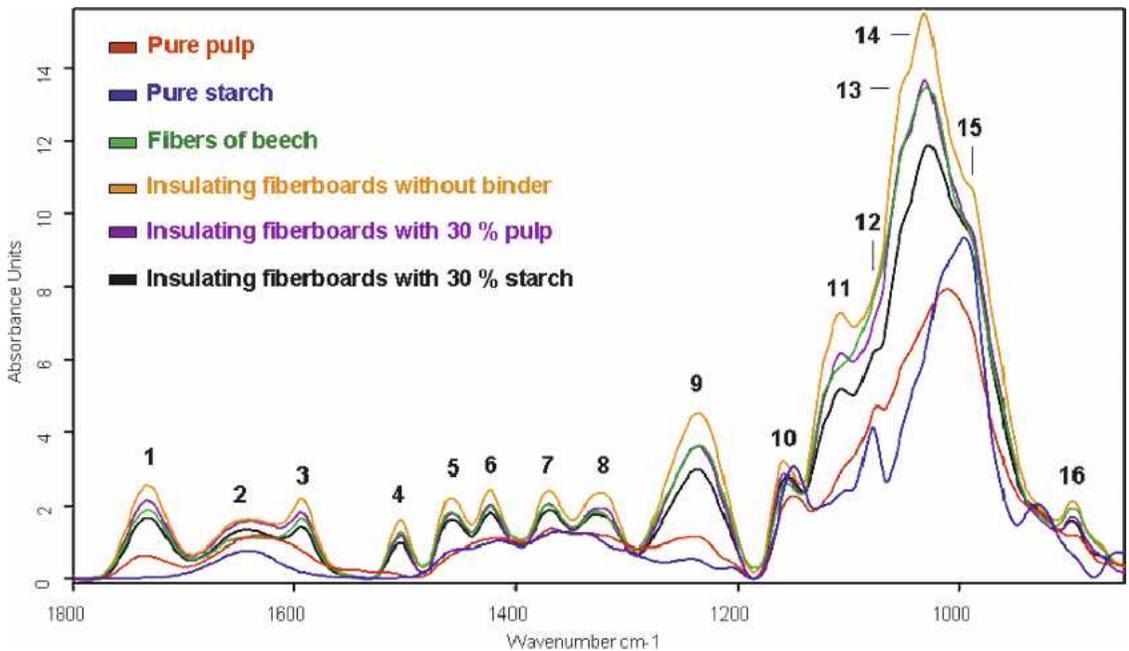


Figure 1. Mean Fourier transform infrared–attenuated total reflectance spectra of beech fibers, pulp, and starch as binder and different types of insulating fiberboards dried by microwave. For each spectrum, ten individual samples were averaged. The spectra were baseline corrected and vector normalized; the bands numbers have been assigned to the compounds indicated in Table 2.

Table 2. Wavenumber characteristics.^a

Wavenumber (cm ⁻¹)	Assignments	Band numbers
1732	C = O stretch in unconjugated ketones, carbonyls, and in ester groups (frequently of carbohydrate origin); C = O in xylan acetates (hemicelluloses)	1
1649	Absorbed O-H and conjugated C-O	2
1593	Aromatic skeletal vibration plus C = O stretch	3
1505	Aromatic skeletal vibration plus C = O stretch	4
1460	C-H- deformation; asymmetric in -CH ₃ - and -CH ₂ -	5
1424	Aromatic skeletal vibration combined with C-H in plane deformation	6
1372	C-H deformation in cellulose and hemicelluloses	7
1328	Syringyl ring plus Guaiacyl ring condensed	8
1235	Syringyl ring and C = stretch in lignin	9
1157	C-O-C vibration in cellulose and hemicelluloses	10
1109	O-H association band in cellulose and hemicelluloses	11
1076	C-H, C-O deformation	12
1056	C-O stretching in cellulose and hemicelluloses	13
1031	Aromatic C-H in plane deformation, guaiacyl type and C-O deformation; primary alcohol	14
998	C-O stretching in cellulose and hemicelluloses	15
897	C-H deformation in cellulose	16

^a After Usmanov et al 1972; Faix 1991; Faix 1992; Pandey and Theagarjan 1997; Fengel and Wegener 2003; Pandey and Pitman 2003. Band numbers in the table refer to the numbers assigned to the bands in Fig 1.

bance of all materials investigated across the whole fingerprint region, especially in the cellulose and hemicelluloses regions (Fig 1: peaks 10, 11, and 13–16). Spectra of fibers and fiberboards treated with potato pulp were overlapping over large areas, and the lowest absorbance was generally found for spectra of starch-treated fiberboards (Fig 1).

Fibers showed lower absorbance than binder-free fiberboards (Fig 1). The spectral differences indicated alterations of C=O, C-H, C-O, and

O-H bonds for the entire fingerprint region. Furthermore, all fiberboards showed a distinct band at position 11, whereas fibers had a spectral shoulder at this wavenumber (Fig 1). This band was most likely from the drying process, which caused a modification to O-H in cellulose and hemicelluloses, mostly likely indicating increased hydrogen bonding in boards compared with loose fibers.

Fiberboards treated with potato pulp showed absorbance units in bands 5–9, 13, and 15 similar to those of fibers (Fig 1), whereas spectral deviations were detected in the other bands: increases in bands 1–3, 10, 11, and 14 and decreases in bands 4, 12, and 16. Fiberboards treated with potato starch displayed higher absorbance units at bands 2 and 10 than those of fibers (Fig 1). All other bands showed lower absorbance across the whole fingerprint region. These repressions were particularly pronounced at band positions 9 and 14 compared with boards treated with pulp or those free of binders (Fig 1).

In the spectra of pulp- and starch-treated boards, a spectral shoulder at position 12 was more pronounced than in fibers (Fig 1). Both binders also displayed a significant band at this position. Thus, the shoulder at position 12 in the spectra of fiberboards can be ascribed to presence of the binders. This was the only band that increased in absorbance in the spectra of binder-containing compared with binder-free fiberboards.

Comparing the spectra of potato pulp and potato starch, several differences were apparent (Fig 1). Pulp, which contains cell debris with cell fragments, pectin and starch, and so on, showed a more complex spectrum than pure starch. Starch, which contains amylose and amylopectin polymers based on α -D-glucose, has two prominent peaks at bands 12 and 15, whereas pulp shows an additional broad peak at band 1, which is indicative of xylan acetates. Because band 12 is characteristic for pulp and starch but not for fibers, which consist of approximately 40% cellulose (β -D-glucose units), changes in band 12 may represent differences between α and β -glucose polymers. This is further supported by

the lack of this band in crystalline cellulose (Müller et al 2007).

Effect of Increasing Binder Concentrations on Spectral Properties of Fiberboards

It was not expected that spectra of fiberboards treated with pulp or starch would show significant decreases in absorbance compared with those of fiberboards free of binders. This might have been caused by chemical modification or simply by “dilution” of the original absorbance from the addition of agents with little or no absorbance at the wavenumbers in question. In the latter case, we would expect a linear correlation between the decrease in absorbance and the increase in binder. To test this hypothesis, we produced fiberboards with increasing binder amounts from 0 to 30% (see Table 1).

The FTIR spectra of fiberboards containing increasing binder concentrations showed decreasing absorbance units at most spectral positions (data not shown). To analyze these relationships, band 1 at 1732 cm^{-1} , typically for carbohydrates and hemicelluloses, was selected because starch did not show any absorbance and pulp very little at this wavenumber. The decrease of absorbance at this position is significantly correlated with an increase of the binder yielding correlation coefficients of $R = 0.991$ for pulp and $R = 0.984$ for starch (Fig 2). However, the decrease is approximately two times less pronounced for pulp than for starch (Fig 2), which is probably caused by the fact that pulp contains xylans, which are absent in starch. Overall, this analysis indicates that the loss in absorbance was caused by a “dilution” effect of the binder and can be used to quantify binder additions.

Characterization of Different Production Processes Using FTIR and Cluster Analysis

Fiberboards with different binders. To ensure high-quality products, uniformity of both production processes and the resulting products is required. We reasoned that it should be possible to separate raw materials and products based on

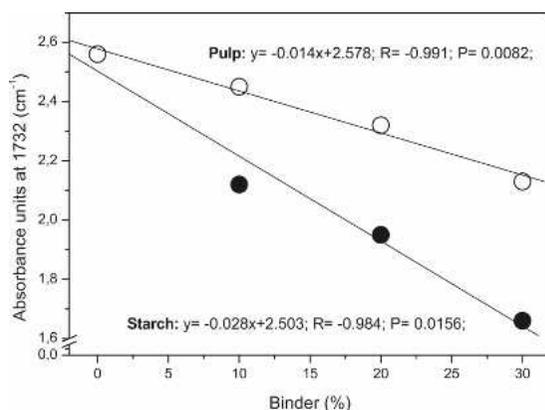


Figure 2. Correlation of absorbance units and percentage of pulp (open circles) or starch binder (closed circles) in the insulating fiberboard. The absorbance of band 1 (1732 cm^{-1} , C = O stretch in unconjugated ketones, carbonyls, and in ester groups (frequently of carbohydrate origin, C = O in xylan acetates) was plotted with the amount binder added for fiberboard production. The points were plotted by linear regression analysis.

the spectral differences. If the production processes led to uniform products, they should be separable into distinct groups. To test these assumptions, the FTIR spectra obtained for individual samples were subjected to cluster analysis. By this means, the heterogeneity of the spectra of fibers and boards treated with different types of binder or without binder was investigated yielding a dendrogram (Fig 3). In the dendrogram, the different materials clearly formed distinct groups indicating that the heterogeneity within a certain class of materials was much smaller than between different materials. Two main subclusters were obtained separating the spectra of fibers from those of fiberboards (Fig 3). The spectral heterogeneity between fiberboards and fibers was 2.1, which was twice as high as the heterogeneity within the different fiberboard products. Within the subcluster for fiberboards, two second-order subclusters were obtained, separating boards treated with starch from those free of any binders or treated with pulp (Fig 3). The subclusters of the fiberboards showed heterogeneity of 0.9, more than four times as high as that of pure fibers. The heterogeneity between boards free of binders and boards treated with pulp was approximately 0.2,

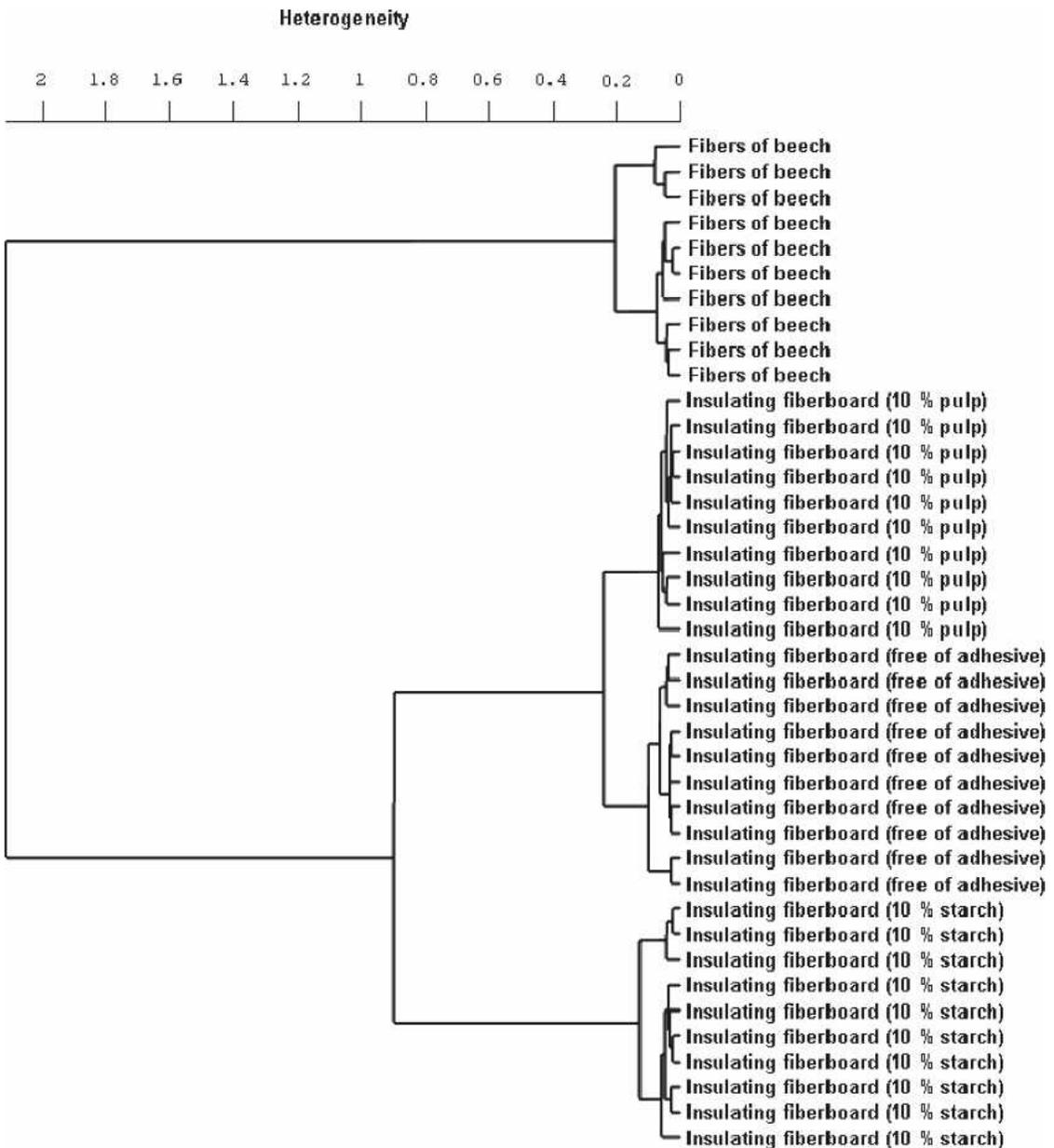


Figure 3. Cluster analysis for individual spectra of beech fibers, insulating fiberboards treated with 10% pulp or 10% starch and fiberboards free of any binder. All boards have been dried by microwave. Cluster analysis was performed with the spectral data in the wavenumber range from 1800–600 cm^{-1} .

twice as high as that of fiberboards treated with starch. Boards with increasing binder content were analyzed and form clear groups according to binder content and binder type (data not shown).

Fiberboards dried by microwave or air dryer. In addition to microwave drying, fiberboards were also dried conventionally in a drying oven. We measured a maximum temperature of 170°C in the dryer and intermittently 220°C in the fiber

mats dried by microwave, suggesting differences in thermal modification of the fibers in the boards. Bands 11, 14, and 16, typical for O-H, C-H, and C-O in cellulose and hemicelluloses, respectively, decreased very subtly in the boards dried by microwave compared with boards dried in the dryer (data not shown). Although the spectra of fiberboards dried by the two methods were quite similar, cluster analysis was able to successfully distinguish materials from the two drying processes (Fig 4). We received two main clusters separating fibers and fiberboards without any binder. The latter cluster was split into two further subclusters, which contained spectra resulting from the particular drying technique. Fibers and fiberboards displayed a high spectral heterogeneity of 2.0, more than 10 times higher than the heterogeneity within the fibers or within the fiberboards. Still, the heterogeneity among the fiberboards was sufficiently high to separate their spectra resulting from the particular drying technique. The differences caused by the drying method between fiberboards were also maintained in boards treated with binders (data not shown).

DISCUSSION

To our knowledge, this is the first study in which changes in the chemical fingerprint of beech fibers have been analyzed during processing through to insulating wood fiberboards. We showed that FTIR-ATR spectroscopy combined with cluster analysis has the potential to characterize spectral deviations between fibers and novel insulating fiberboards and can be used to identify the influence binders from renewable resources have on the chemical composition of the resulting insulators.

We detected a decrease of the absorbance units in the spectra of the fiberboards with increasing binder concentrations (Fig 2). Initially, we assumed that a possible reason might have been the modification and decomposition of the holocellulose fraction during drying of the boards. However, analysis of a series of fiberboards with increasing binder content clearly revealed strict

correlations between the amount of binder and the decrease in absorbance. Therefore, we conclude that the fiberboard composition is diluted by the particular binder. A consequence of the observed strict correlations is that this type of spectral analysis can be used to quantify the amount of binder added to the product.

FTIR spectroscopy also enabled us to identify modifications of the chemical constituents during processing. Because hemicelluloses can be degraded and acetylated at temperatures below 180°C (Garrote *et al* 2001; Sivonen *et al* 2002), deviations in the spectra of fibers and fiberboards might have been caused by such modifications of the holocellulose fraction during the drying process (Fig 1). These deviations were independent of the binder or its particular concentration in the board. Furthermore, we determined a significant increase in band 11 for the treated fiberboards, typical for O-H association in cellulose and hemicelluloses. Because this band appeared in the spectra of all fiberboards but not in the fibers, the deviation was most likely caused by the drying process. The high temperatures in the dryer and the microwave might have led to a modification of cellulose and hemicelluloses, most likely causing increased hydrogen bonding and thereby leading to higher absorbance units at this wavenumber.

Using FTIR spectroscopy, we identified a clear spectral influence of pulp and starch binders on band 12 (Fig 1). This band was more prominent in the spectra of fiberboards treated with binders and in the spectra of the binders themselves than in fibers and was most likely the result of α -glucose units because it is not present in cellulose FTIR spectra (Müller *et al* 2007).

The differences in the mean spectra identified by direct interpretation were apparently sufficiently high and consistently present in individual samples to enable grouping of the different products by cluster analysis. Spectra of all fiberboards were distinguishable from those of the fibers (Fig 3). Fiberboards treated with 10% pulp were more similar to boards free of binders than to boards treated with starch (Fig 3). This

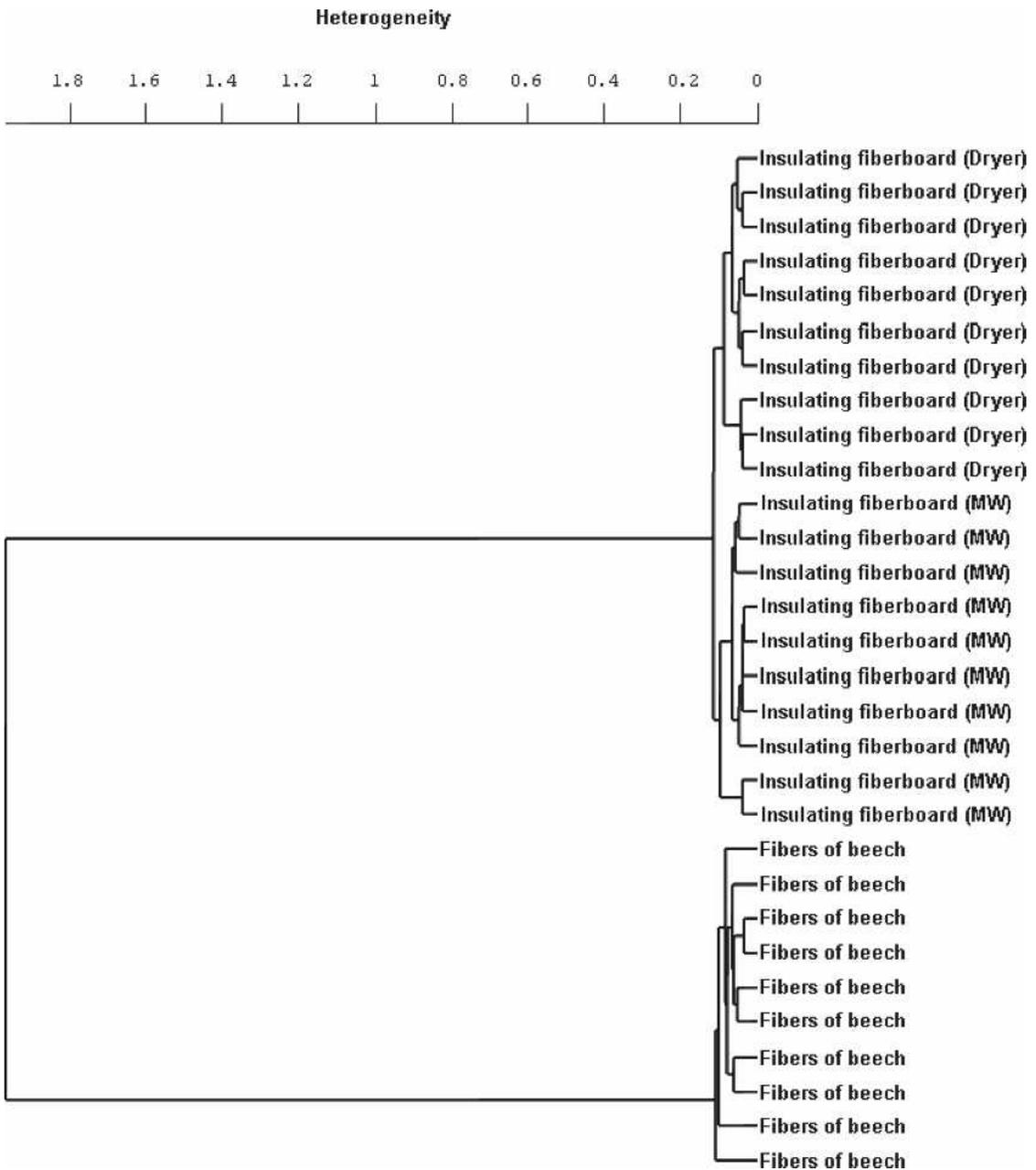


Figure 4. Cluster analysis for individual spectra of beech fibers and insulating fiberboards dried by microwave (MW) or in the dryer, respectively. Cluster analysis was performed with the spectral data in the wavenumber range from 1800–600 cm^{-1} .

was most likely the result of the high spectral similarity, visible in bands 5–9, 13, and 15 (Fig 1). However, it must be noted that cluster analysis does not provide information on chemical

basis of the differences. Still, it is a very useful method to supervise and control production processes because the homogeneity is an inherent feature of product quality that can be easily assessed.

The conventional technique for drying fiber mats is by air circulation (Kutschera and Winter 2006). However, drying by microwave energy is much faster (Hansson and Antti 2003). Therefore, we dried fiber mats conventionally or by microwave and used cluster analysis to investigate influence of the particular drying technique on the chemical composition of the fibers (Fig 4). The spectra of fibers were separated from spectra of fiberboards, illustrating modification of the chemical components during drying. These were most pronounced at band 11, which indicates changes in O-H associations in cellulose and hemicelluloses (Table 2). The drying technique resulted in further subclustering of fiberboard spectra (Fig 4). It is likely that different temperatures during drying and different methods of drying caused different modifications of the chemical compounds. A dryer warms up the outside area of the sample with the highest temperatures prevailing on the surface (Fischer *et al* 2007). Microwave drying is based on the so-called volumetric warming, which heats up the sample itself and leads to the highest temperatures in the center (Fischer *et al* 2007). The increased heating rate during microwave drying can cause microstructural changes (Rhee 2002). Kaasová *et al* (2002) reported that microwave energy and temperature of treatment resulted in amylose degradation. As a result of such effects, a significant modification of the chemical constituents might have taken place, enabling us to separate both drying techniques with cluster analysis.

CONCLUSIONS

We showed that FTIR spectroscopy can be used to detect changes in beech fiber properties during processing through to insulating wood fiberboards. Drying causes chemical modifications through increased hydrogen bonding, which can be used to distinguish the drying method used. Our results also show that additions of plant-based renewable binders can be quantified by

specific decreases in absorbance of the fiberboard spectra. Because cluster analysis is a method to group samples according to similarity, it is suitable for quality control of products. Here, we have shown that cluster analysis of FTIR-ATR spectra can be applied to distinguish fibers and fiberboards from different production processes. These methods can therefore be used to support the optimization of production processes for innovative insulating wood fiberboards or other organic compounds.

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