

Nondestructive Evaluation of Thermally Modified Ash (*Fraxinus excelsior* L.) by Near Infrared Spectroscopy (NIRS)

Helmut Bächle¹, Bernhard Zimmer¹, Elisabeth Windeisen² and Gerd Wegener²

¹Salzburg University of Applied Sciences, Markt 136A, 5431 Kuchl, Austria
[email:helmut.baechle@fh-salzburg.ac.at]

²Holzforschung München, Technische Universität München, Winzererstr. 45, D-80797 Munich, Germany

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ABSTRACT

Ash (*Fraxinus excelsior* L.) modified by thermal treatment in an industrial scale plant was analysed by FT-NIR spectroscopy. Mechanical and physical properties were determined. For quantitative analysis Partial-Least-Squares-Regression (PLSR) was used. The samples were divided into a calibration and a validation set. Calibration models were developed for density, bending modulus of elasticity (MOE), modulus of rupture (MOR) and moisture content. r^2 -values and prediction errors range from very good to moderate depending on spectral treatment, acquisition mode and the actual property being modelled.

INTRODUCTION

Improvement of wood properties like dimensional stability and durability are essential for thermally modified timber (TMT) to be used as a building material. However, major drawbacks like the loss of stiffness and density limit the range of potential application. Further a high variability in the properties of the product can be observed due to variability in wood quality and a production process which is influenced by different parameters (Hanger *et al.* 2002). However, customers demand constant quality and therefore the product itself needs to be evaluated to meet the expectations and it is still time consuming and costly to characterise wood properties like modulus of elasticity (MOE), modulus of rupture (MOR) and density. The use of near infrared spectroscopy (NIRS) for the evaluation of chemical composition and properties of lignocellulosic material has increased since its first application in the mid 20th century (Osborne *et al.* 1993). Meanwhile NIRS has found its way into wood analysis and it is considered to be a promising technique for quality control. Overviews of applications are given by So *et al.* (2003) and Tsuchikawa (2007). NIRS has also been considered a tool for evaluating thermally modified wood (Esteves and Pereira 2008). Since NIR spectra consist of mainly overlapping bands of C-H- and O-H- overtones and combination bands, multivariate statistics has to be applied for interpretation. This study uses partial least square regression (PLSR) to evaluate MOE, MOR, oven-dry density and the moisture content of ash samples thermally modified in an industrial scale kiln.

EXPERIMENTAL

Sample preparation

Matched ash boards were thermally modified in an industrial scale plant. The treatment temperature ranged from 180 °C to 220 °C at constant time. Subsequently bending test samples (20*20*360 mm³) according to DIN 52186 were cut and stored at 20 °C/65 % r.h. for about 2 months.

FT-NIR-spectroscopy

FT-NIR-spectra were obtained from the radial and tangential face of bending test samples by a Bruker MPA spectrophotometer equipped with an integrating sphere using a resolution of 8 cm⁻¹ (32 Scans). Seven single spectra per sample were averaged. Vector normalization, standard normal variate (SNV), Savitzky Golay 1st (13 points) and 2nd derivative (15 points) were performed to eliminate baseline shifts or scattering effects.

Mechanical testing

Bending MOE and MOR were determined according to DIN 52186 on an all-round test machine by Zwick-Roell (Z 250). Density was determined according to DIN 52182, moisture content according to DIN 52183.

PLS Regression

All, reference (67) and thermally modified (203) samples, were used for analysis. The samples were divided into two sets, one consisting of the reference samples and one consisting of all modified samples. Only the sample set used for evaluating the moisture content includes just the 200 °C and 220 °C treatment. This is due to the appearance of different clusters according to moisture content due to treatment intensity. Using samples of all treatments would have induced a non described area around 5 % moisture content. Therefore the both of the most severe treatments were combined for modelling moisture content. The samples were divided randomly into a calibration and a validation set. Every third sample was included in the validation set. For both calibration and validation models r^2 and root mean square error (RMSEC, RMSEP) were calculated. For spectral pre-treatment and PLS-1 regression the Unscrambler 9.7 (Camo Process AS, Oslo, Norway) software package was used.

RESULTS AND DISCUSSION

The PLSR model parameters of thermally modified ash samples (Table 1) differ from the ones of untreated reference samples (not shown). Generally the models of only unmodified ash samples showed better r^2 - and RMSEP values than those of thermally modified samples. The models of MOE, MOR and oven-dry density based on radial face spectra perform better than those based on tangential face spectra. Only moisture content models show similar results with spectra from both faces. All radial face based models of MOE show r^2 -values of > 0.60 for calibration and validation models. The r^2 -values of MOR are lower namely 0.50 and higher for calibration and 0.39 for the best validation model (SNV). The oven-dry density models perform best with the 1st Derivative having an r^2 of 0.82 for calibration and 0.58 for validation. The corresponding values for the unmodified sample set are 0.84 (calibration) and 0.76 (validation) (not shown). Moisture content could be modelled with an r^2 of 0.87

(calibration) and 0.60 (validation) on the radial face. The superior results of the radial face can be related to the fact that the early and late wood are represented, whereas on the tangential face the risk of an over representation of either early or late wood is possible. Besides, the large vessels of the early wood can influence the acquisition of the FT-NIR spectra on the tangential face more than on the radial face when the growth ring is cut through the early wood zone. The RMSEP of the models based on radial face spectra is less than the RMSEP from models based on tangential face spectra. The ratio of RMSEP/SDD is < 1 with all MOE and MOR calibration and validation models and with radial oven-dry density models. RMSEP/SDD is around 1 with the models of oven-dry density and moisture content.

Table 1: Summary of PLS-1 model parameters of calibration and test models of MOE, MOR, oven-dry density and moisture content from averaged spectra taken from the radial and tangential face of bending test samples

	radial					tangential				
	PCs	r^2_{cal}	RMSEC	r^2_{val}	RMSEP	PCs	r^2_{cal}	RMSEC	r^2_{val}	RMSEP
MOE										
raw	10	0.74	810.13	0.73	844.88	10	0.45	1222.58	0.30	1334.75
norm.	8	0.69	911.84	0.62	991.11	10	0.44	1235.74	0.27	1366.07
SNV	7	0.65	1007.50	0.62	974.77	9	0.41	1255.61	0.36	1284.91
1 st Der.	8	0.81	712.75	0.77	766.19	7	0.51	1146.29	0.35	1298.35
2 nd Der.	5	0.69	912.10	0.62	987.88	4	0.42	1253.48	0.32	1334.83
MOR										
raw	7	0.50	14.49	0.34	14.17	5	0.34	17.54	0.33	15.32
norm.	10	0.56	14.34	0.38	14.63	5	0.33	17.63	0.32	15.43
SNV	7	0.50	15.32	0.39	14.57	4	0.32	17.71	0.36	14.91
1 st Der.	8	0.60	13.64	0.36	15.01	5	0.35	17.39	0.35	15.06
2 nd Der.	8	0.74	11.11	0.27	16.03	5	0.37	17.08	0.30	15.66
oven-dry density										
raw	10	0.73	0.03	0.53	0.03	2	0.37	0.04	0.18	0.04
norm.	9	0.58	0.03	0.32	0.03	4	0.35	0.04	0.01	0.04
SNV	7	0.54	0.03	0.31	0.03	5	0.41	0.04	0.11	0.04
1 st Der.	9	0.82	0.02	0.58	0.03	2	0.34	0.04	0.27	0.04
2 nd Der.	6	0.72	0.03	0.49	0.03	2	0.34	0.04	0.28	0.04
Moisture content 20/65 (only 200°C / 220°C samples)										
raw	6	0.87	0.08	0.60	0.10	7	0.78	0.11	0.63	0.10
norm.	3	0.79	0.10	0.63	0.10	6	0.75	0.11	0.65	0.09
SNV	2	0.77	0.11	0.63	0.10	6	0.76	0.11	0.63	0.10
1 st Der.	2	0.77	0.11	0.63	0.10	5	0.76	0.11	0.65	0.09
2 nd Der.	3	0.80	0.10	0.64	0.09	2	0.66	0.13	0.53	0.11

However, the RMSEP for the thermally modified sample models is always higher than the RMSEP of the reference sample models. Especially the results of MOR of the thermally modified samples differ from those of the reference samples. It is known that MOR is linked to MOE and density. NIR is sensitive to both parameters as well as chemical composition; however, MOR is not described equally well. One reason could be the influence of internal cracks in the cell wall structure induced by thermal treatment. The results also show that the correlation of MOE and MOR becomes weaker after thermal treatment. The results of the models based on the unmodified spectra show generally good results. Spectral pre-treatment mostly results in a lower number of latent variables (PCs). The latent variables contain chemical and physical information

necessary for modelling the measured parameters. Increasing numbers of latent variables improve the models, but increase the ratio of noise being modelled (overfitting). By spectral pre-treatment r^2 and RMSEP could be improved while using less PCs in some cases.

CONCLUSIONS

Prediction errors and regression coefficients of calibration and validation models based on FT-NIR spectra of thermally modified ash bending samples show that very good models can be achieved with r^2 -values of > 0.60 for MOE calibration and validation models. Also MOR, oven-dry density and moisture content could also successfully be evaluated. Models based on FT-NIR spectra taken from the radial face performed better than models based on spectra from the tangential face. Spectral pre-treatment could improve the results, especially the number of latent variables. However, no general assumption can be made, since the untreated raw spectra generally show good results. The results indicate that NIR-spectroscopy and multivariate regression can be used for quality control of thermally modified wood. An application focussed pre-sorting system for TMT might be based on this technique for samples before and after treatment to guarantee constant product quality. Further studies might be focussed on the use of NIR-imaging system for quality control and the application of an optical fibre probe for process control during the actual treatment.

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REFERENCES

- Esteves, B. and Pereira, H. (2008). Quality assessment of heat-treated wood by NIR spectroscopy. *Holz als Roh- und Werkstoff*, **66**, 323-332.
- Hanger, J., Huber, H. Lackner, R. and Wimmer, R. (2002). Physikalische Eigenschaften heimischer Holzarten nach Wärmebehandlung. *Holzforschung und Holzverwertung*, **54**, 111-113.
- Osborne, B.G., Fearn, T. and Hindle, P. (1993). *Practical NIR Spectroscopy with Applications in Food and Beverage Analysis*. Longman Scientific & Technical, Harlow, Essex, England.
- So, C.-L.; Via, B.K.; Groom, L.H.; Schimleck, L.R.; Shupe, T.F.; Kelley, S.S.; Rials, T.G. (2004). Near infrared spectroscopy in the forest products industry. *Forest Products Journal*, **54**, 6-16.
- Tsuchikawa, S. (2007). A review of recent near infrared research for wood and paper. *Applied Spectroscopy Reviews*, **42**, 43-71.