Evaluation of density and strength of Norway spruce wood by near infrared reflectance spectroscopy

P. Hoffmeyer, J. G. Pedersen

The non-destructive evaluation of wood properties by Near Infrared Reflectance Spectroscopy (NIR) has been assessed. The surfaces of specimens of clear wood *(Picea abies)* were NIR-scanned, and the results compared to such properties as moisture content, density, compression strength and chemical and biological degradation. In addition, the NIRscans of clear wood specimens were compared to the bending strength of the structural timber from which it had been cut. The NIR dependency of surface roughness was investigated and found to be of minor importance.

NIR calibrations for moisture content (MC < 30%), density, compression strength and chemical degradation proved that the NIR technique is an excellent non-destructive method $(r^2 \ge 0.9$; independent test set). Even biological degradation was determined with a most promising accuracy ($r^2 = 0.75$; independent test set). For the prediction of the bending strength of timber NIR proved less efficient. However, NIR still contributed to timber strength prediction at the same level as annual ring width, the parameter which is presently visually assessed by timber graders.

It is concluded that the NIR method is very versatile in the non-destructive evaluation of wood. The results merit further investigations in order to develop proper models and instrumentation for commercial use.

Abschiitzen der Dichte und Festigkeit von Fichtenholz mittels NIR-5pektroskopie

Die Möglichkeit, Holzeigenschaften mit Hilfe der zerstörungsfreien NIR-Spektroskopie zu bestimmen, wurde überprüft. Die Oberfläche von fehlerfreiem Fichtenholz wurde mittels NIR abgetastet. Die Meflwerte wurden zu folgenden Holzeigenschaften in Beziehung gebracht: Feuchte, Druckfestigkeit, Dichte sowie chemischen und biologischem Abbau. Zusätzlich wurden die NIR-Ergebnisse noch verglichen mit der Biegefestigkeit des Bauholzes aus dem die Proben entnommen worden waren. Auch die Rauheit der Holzoberflächen wurde mit einbezogen. Sie erwies sich als rel. unbedeutend. Die NIR-Messungen erwiesen sich als hochkorreliert ($r^2 \ge 0.9$) mit der Feuchte (MC < 30%), der Dichte, der Druckfestigkeit und dem Abbaugrad (durch Chemikalien), so daß die NIR-Technik als vorzügliche zerstörungsfreie Bestimmungsmethode anzusehen ist. Sogar der biologische Abbau konnte mit vielversprechender Genauigkeit ($r^2 = 0.75$) bestimmt werden. Für die Vorhersage der Biegefestigkeit des Bauholzes war die NIR-Methode weniger geeignet. Immerhin erlaubt die NIR-Technik das Abschätzen der Biegefestigkeit

P. Hoffmeyer

J. G. Pedersen

Building Materials Laboratory, Technical University of Denmark, Building t 18, DK-2800 Lyngby, Denmark

mit der gleichen Zuverlässigkeit wie das Abschätzen der Jahrringbreiten, wie es zur Zeit noch bei der visuellen Holzsortierung benutzt wird. Die NIR-Spektroskopie erweist sich als vielseitig nutzbare Methode zum zerst6rungsfreien Ermitteln yon Holzeigenschaften. Die Ergebnisse ermutigen weitere Untersuchungen, um daraus geeignete Modelle und Geräte für industrielle Anwendungen zu entwickeln.

Introduction

1

Near Infrared Reflectance Spectroscopy (NIR) is a technique for the non-destructive evaluation of such organic materials where particularly $-CH$, $-OH$ and $-MH$ groups influence the properties to be assessed. NIR has a widespread application in agriculture and food industry for the determination of the content of starch, protein, moisture etc. In the wood industry NIR is now used on a routine basis for the in-line determination of moisture content during production of MDF- or particle boards.

The basis of the NIR technique is the quantification, according to the Lambert-Beer law, of the absorption (A) of near infrared light by the material. The near infrared light is confined to wavelengths from 700 nm to 2500 nm. In practice the reflectance (R) of the light is measured, and the relationship $A = \log(1/R)$ is used to calculate the absorption.

The NIR technique is confined to the evaluation of surface layers. However, light can be emitted, and reflectance recorded, by the use of optical fibres, and the interior of wood thereby made accessible to measurements through the drilling of small holes.

NIR has been explored for the determination of constituents in dried, ground wood (Mroczyk et al. 1992; Murray 1992; Schultz and Burns 1992). The potential for the characterization of pulp has been demonstrated (Birkett & Gambino 1989; Easty et al. 1990; Wallbäcks et al. 1991a and b). NIR has been used for the determination of moisture content and density of wood (Pedersen et al. 1993) and for wood identification (Nair and Lodder 1993). The influence of surface roughness and fibre angle relative to the duration of the incident light has been studied (Tsuchikawa et al. 1992), and NIR has been used for the characterization of glue/chip mixtures (Kniest 1992; Niemz et al. 1992).

The aim of the present study was to evaluate the potential of NIR for the measurement of density and strength of wood with special reference to the study of chemically and biologically degraded wood. The paper emphazised the wood technological aspects of the study, whereas the statistical analysis is treated more thoroughly elsewhere (Pedersen et al. 1993). A presentation of preliminary results was given earlier (Hoffmeyer 1992).

Material

2

5 different experiments were carried out, all of which were based on the measurement of NIR, moisture content, compres-

Biotechnological Institute, Holbergsvej 10, 6000 Kolding, Denmark

sion strength and density of Norway spruce *(Picea abies).* Experiments 1-4 included specimens of dimensions $20 \times 20 \times$ 60 mm while experiment 5 used cross sections from structural timber beams of dimensions $45 \times 95 \times 1800$ mm.

2.1

Experiment 1

The aim of this experiment was to assess the potential of NIR with respect to wood of different moisture contents and densities.

166

127 specimens were produced from a total of 28 boards representing a range of annual ring widths from 0.5 mm to 10 mm and a density range from 350 kg/m³ to 580 kg/m³. 4-5 specimens were prepared from each board. The specimens sides were planed and cross sections cut with a fine-tooth circular saw. 7 specimens were conditioned at each of 11 different moisture content levels ranging from dry to almost fibre saturated condition. 50 of the 127 specimens were conditioned specifically to reach a moisture content of 12%.

2.2

Experiment 2

The aim of this experiment was to assess the potential of NIR with respect to detecting strength differences for wood of equal density.

81 specimens were produced from one board in such a way that the same annual rings were, as far as possible, represented in all specimens. The densities ranged from 450 kg/m^3 to 500 kg/m³. The specimens were treated with gaseous hydrogen fluoride for various lengths of time in order to produce a series of specimens representing the full range of chemical decomposition of the cell wall material. Moisture content was approximately 12%.

2.3

Experiment 3

The aim of this experiment was to assess the influence of surface roughness on NIR precision.

52 specimens of the same type as used in experiment no. 1 were conditioned to 12% moisture content. After compression testing the specimens were cross cut in order to give 3 different surface roughness conditions: coarse-tooth band saw, coarse tooth circular saw and sanding with grade P500 sandpaper.

2.4

Experiment 4

The aim of this experiment was to assess the potential of NIR with respect to decay detection.

A sample of 120 prisms was prepared from discs taken from the ground level location of old salt treated transmission poles. It was attempted to have a wide range of degree of decay represented in the sample. Most of the decay was soft rot, but also extensive "orange rot" *(Rigidoporus vitreus)* was present in some specimens. Two specimen sides were planed. Two sides and the two cross sections were cut with a fine-tooth circular saw. The specimens were then conditioned at 90% RH.

2.5

Experiment 5

The aim of this experiment was to assess the potential of NIR with respect to density and strength of structural timber.

57 boards, having a moisture content of approximately 20%, were tested to bending failure (third point loading). Two discs were cut close to the location of failure. The cross cuts were made with a coarse-tooth circular saw. One disc from each board was used for NIR measurements. The other was used for density and moisture measurements.

3 Methods

3.1

Chemical treatment

The chemical treatment (experiment 2) was carried out by placing each wood prism in a closed polypropylene container. The prisms were then exposed to the vapors of hydrogen fluoride for 48 hours in amounts rising in steps of 1% from 0% to 40% of the dry prism weight corresponding to 41 specimens.

3.2

NIR measurements

Reflectance was measured with a Quantum 1200I Plus instrument from LT Industries that measures at 1201 wavelengths from 1200 nm to 2400 nm. Each prism was measured twice and each measurement represented 30 scans. Only measurements on cross sections are reported here.

3.3

Calibration

Each measurement consists of 1201 reflectance values which in turn, represent an average of 30 scans. Pre-treatment of these spectra was, as a rule, carried out as follows: Background correction with reference ceramic, logarithm, normalization and derivation. The multivariate methods PLSR (Partial Least Square Regression) and PCR (Principal Component Regression) were used for calibrating the spectra against the reference values.

The results from each experiment were sub-divided into a calibration set (75-80% of the specimens) which produces a model and a test-set (20-25% of the specimens) which validates the model. SEE (Standard Error of Estimate) is calculated for the calibration set. SEP (Standard Error of Prediction) and the coefficient of determination (r^2) is subsequently calculated for the test-set.

Statistical methods were used to identify a few outliers which were then removed from both the calibration sets and the test sets.

4

Results

4.1

Experiment 1

The results of the statistical analysis are shown in Table 1. The columns in italic identify the models used (number of factors) for the calibration. These models are illustrated in Figs. 1-3.

4.2

Experiment 2

The results of the statistical analysis are shown in Table 2. The model which includes 6 factors is shown in Fig. 4.

There is found no correlation between density and NIR as density is not significantly influenced by the chemical treatment.

4.3

Experiment 3

The results of the statistical analysis are shown in Table 3. The model which includes 6 factors and all specimens with no regards to surface conditions is shown in Fig. 5.

4.4

Experiment 4

The results of the statistical analysis are shown in Table 4. The model which includes 4 factors is shown in Fig. 6.

Table 1.3 calibrations selected for the final validation of models for moisture content, density and compression strength

Table 2. 3 calibrations selected for the final validation of compression strength

No. of factors			14	
SEE (MPa) (calibration set) 3.7		3.2	1.0	
SEP (MPa) (test set)	4.0	3.3	2.8	
r^2 (test set)	0.93	0.95	0.97	

Table 3. Calibration developed from data from band sawn, circular sawn, and sanded cross sections

Surface roughness No. of factors		Band saw Circular saw	Sanded	- All
SEE (MPa) calibration 1.4	2.1	1.1	2.9	2.1
SEP (MPa) validation		19	32	23

Table 4.2 calibrations selected for the final validation of compression strength

4.5

Experiment 5

The results of the statistical analysis are shown in Table 5. The selected models (non-shaded columns) are illustrated in Figs. 7 and 8.

5

Discussion and conclusions

Throughout the following discussion of results it should be noted that, as a rule, the r^2 values presented express how well one part of a population of test results (20-25%) is described by a model derived independently from the remaining part of the same population (75-80%). Clearly, the r^2 values for the whole population would have been higher.

From Experiment 1 it can be concluded that moisture content of solid wood below fibre saturation is assessed with a remarkable accuracy ($r^2 > 0.99$). This finding extends the use of

Fig. 1. Relationship between NIR determined moisture content (%) and measured moisture content (experiment 1: varying density and moisture content)

Bild 1. Beziehung zwischen spektroskopisch (NIR) gemessener und tatsächlicher Feuchte (in %). Versuch 1: Variation der Dichte und Feuchte

Fig. 2. Relationship between NIR determined density ($kg/m³$) and measured density (experiment 1: varying density and moisture content)

Bild 2. Beziehung zwischen spektroskopisch (NIR) bestimmter und tatsächlicher Dichte (in Kg/m³). Versuch 1: Variation der Dichte und Feuchte

NIR beyond its present field of application which has been moisture content of pulp or wood particles. The results do not permit any conclusions regarding the assessment of moisture content above the fibre saturation point.

The results also show that both density and compression strength are easily detectable by NIR. With calibrations including only 4 factors, density and compression strength show NIR correlations of $r^2 = 0.76$ and $r^2 = 0.92$, respectively; for an 8-factor calibration both correlations exceed r^2 =0.94. Compression

> Table 5. Calibrations selected for the final validation of models for density and bending strength of structural timber

167

Fig. 3. Relationship between NIR determined compression strength (MPa) and measured compression strength (experiment l: varying density and moisture content)

Bild 3. Beziehung zwischen spekroskopisch (NIR) bestimmter und tatsächlicher Druckfestigkeit (in MPa). Versuch 1: Variation der Dichte und Feuchte

Fig. 6. Relationship between NIR determined compression strength (MPa) and tested compression strength (experiment 4: varying degree of decay, varying density, constant moisture content) Bild 6. Beziehung zwischen spektroskopisch (NIR) bestimmter und tatsächlicher Druckfestigkeit (in MPa). Versuch 4: Variation von abbaugrad und Dichte bei konstanter Feuchte

Fig. 4. Relationship between NIR determined compression strength (MPa) and measured compression strength (experiment 2: varying degree of chemical treatment, constant density, constant moisture content)

Bild 4. Beziehung zwischen spektroskopisch (NIR) bestimmter und tatsächlicher Druckfestigkeit (in MPa). Versuch 2: Variation der chemischen Behandlung bei konstanter Dichte und konstanter Feuchte

Fig. 5. Relationship between NIR determined compression strength (MPa) and tested compression strength for specimens of 3 different surface roughness conditions (experiment 3: varying density, constant moisture content)

Bild 5. Beziehung zwischen spekroskopisch (NIR) bestimmter und tatsächlicher Druckfestigkeit (in MPa) für Proben mit 3 verschiedenen Rauheitsgraden der oberfläche. Versuch 3: Variation der Dichte bei konstanter Feuchte

Fig. 7. Relationship between NIR determined density (kg/m^3) and measured density (experiment 5: structural timber, varying density, constant moisture content)

Bild 7. Beziehung zwischen spektrokopisch (NIR) bestimmter und tatsächlicher Dichte (in Kg/m³). Versuch 5: Bauholz, Variation der Dichte bei konstanter Feuchte

Fig. 8. Relationship between NIR determined bending strength (MPa) and measured bending strength (experiment 5: structural timber, varying density, constant moisture content)

Bild 8. Beziehung zwischen spektroskopisch (NIR) bestimmter und tatsächlicher Biegefestigkeit (in MPa). Versuch 5: Bauholz, Variation der Dichte bei konstanter Feuchte

strength seems to be assessed with a higher accuracy than density. However, this finding probably just reflects the fact that compression strength is highly dependent on moisture content which dry density obviously is not. Compression strength, therefore, is assessed on the basis of two parameters (moisture and chemical content) and dry density on the basis of only one (chemical content). An introductory test showed that at constant moisture content compression strength and density are highly correlated and also NIR-assessed with almost the same accuracy. Experiment 1 thus fails to prove that compression strength is NIR-assessed independently of density.

In Experiment 2, specimens of equal density were chemically treated in order to alter strength without, at the same time, altering density. In this test he convincing correlation of NIR versus strength from experiment 1 was confirmed and almost identical r^2 values produced. At the same time the NIR/density correlation was virtually non-existent. This then proves that a direct NIR assessment of strength is possible.

The potential of using NIR as a strength indicator was then further pursued in Experiment 4, which included biologically decayed specimens. The link between Experiment 2 and Experiment 4 is that chemical bonds are hydrolyzed in both series of specimens, either chemically or biologically. Once again, a 4-factor calibration of NIR against strength turns out very favourably although the correlation is somewhat weaker $(r^2 = 0.81)$ than that obtained in Experiment 2 ($r^2 = 0.93$). On the other hand, the standard error of estimate ($SEP = 2.0 MPa$) is only half the value of that of Experiment 2.

The results are encouraging, particularly because there were two basically different types of decay included among the specimens. Some specimens contained soft rot, while other specimens suffered from white rot *(Rigidoporus vitreus);* the former degrades cellulose, while the latter prefers lignin. The results indicate that the model encompassed both types of degradation. The results also exceed by far those from other commercially available non-destructive instruments. It is concluded that NIR might well be a reliable indicator of soft rot; however, a larger sample size is required in order to produce definitive evidence.

While density of small specimens seems to be reliably detected by NIR, the potential of the technique for larger specimens remained to be assessed. In Experiment 5 the density of structural size cross-sections was determined with at least the same precision as that found for the small cross-sections (Experiment 1). A 4-factor model of NIR against density produced a r^2 value of 0.77 as compared to 0.76 for Experiment 1. The corresponding standard error of estimate values of 11 kg/m³ and 37 kg/m³ was clearly in favour of the large sized specimens.

The attempt to use NIR also as a predictor of the strength of structural timber turned out to be much less convincing than density prediction. A 3-factor model produces a r^2 value of only 0.29 and a standard error of estimate of 3.5 MPa. This, however, was expected since the strength of structural sized timber is governed by other factors than density such as knot size, grain angle, compression wood, etc.

Strength grading is mandatory for all timber used for load bearing purposes. Such grading, when carried out visually, is normally based on models which combine a number of parameters each of which has no significant predicting power of its own. Such multiple parameter models will normally produce coefficients of multiple determination of the order r^2 = 0.40-0.45. The two most important visual grading parameters defined in most Timber Codes are annual ring width and knot size. The correlation to strength for these parameters is expressed by a coefficient of simple determination normally of the order $r^2 = 0.20 - 0.27$ for annual ring width and $r^2 =$ 0.16-0.20 for knot size (Hoffmeyer 1990).

Against this background, the above NIR-strength correlation, however weak, seems to be at least as powerful as the presently used annual ring width as predictor of strength (or density). A regression of annual ring width against strength for the present test including 54 specimens results in a slightly higher (r^2 = 0.34) correlation than for the NIR prediction, but also in a slightly higher standard error of estimate (4.3 MPa) than for the NIR prediction. In addition, it should be recalled that the above values for annual ring width correlation are obtained from the whole test set, whereas the NIR values are obtained by using a test set of 11 specimens on a model derived from the remaining 33 specimens. In conclusion, therefore, NIR-measurement is believed to present a realistic alternative to the present visual grading of annual ring width.

The results of test no. 5 are particularly encouraging when looking a little ahead. Eurocode 5 on timber structures requires that a certain characteristic density is guaranteed for visually graded timber. However, there is presently no way of visually grading according to density. The NIR-assessment of density may come in very handy in this connection.

With respect to industrial use it is important to know how sensitive NIR is to the quality of the surface to be measured. Experiment 3 addressed this question by including three different surface qualities, circular sawn, band sawn and sanded. The results indicate that all three surface qualities may be encompassed by the same model. Thus, there seems to be no basic difficulty in handling different surface qualities; however, the present investigation does not offer a complete analysis of this question.

In conclusion, the NIR reflectance method has been proven to offer a fast, accurate and multipurpose tool for the nondestructive evaluation of basic wood properties. The method, obviously, is confined to such measurements where the surface is representative of the interior.

6

References

Birkett, M. D.; Gambino, M. I. T. 1989: Estimation of pulp kappa number with near-infrared spectroscopy. Tappi Journal 72 (9): 193- 197

Easty, D. B.; Berben, S. A.; DeThomas, F. A.; Brimmer, P. J. 1990: Nearinfrared spectroscopy for the analysis of wood pulp: quantifying hardwood-softwood mixtures and estimating lignin content. Tappi Journal 73 (10): 257-261

Hoffmeyer, P. 1990: Failure of wood as influenced by moisture and duration of load. Doctoral dissertation. State University of N. Y., College of Environmental Sci. and Forestry, Syracuse.

Hoffmeyer, P. 1992: Evaluation of wood by near infrared reflectance spectroscopy. Proceedings of IUFRO-Timber Engineering meeting, Bordeaux and Nancy. Building Materials Laboratory. Technical University of Denmark

Kniest, C. 1992: Charakterisierung yon Span-Leim-Gemischen mittels NIT-Spektroskopie. Holz Roh-Werkstoff 50: 73-78.

Mroczyk, W. B.; Kasprzyk, H.; Gawecki, T. 1992: The application of NIRS for the determination of the composition of wood samples of *Fagus silvatica,* pp 566-568 in I. Murray & I. A. Cowe, ed. Making Light Work: Advances in Near Infrared Spectroscopy. VCH Verlagsgesellschaft, Weinheim

Murray, Ian; Cave, lan A. 1992: Making light work. Advances in near infrared spectroscopy. ISBN 3-527-28498-2, ISBN 1-56081-264-8, pp 566-576

Nair, P.; Lodder, R. A. 1993: Near-IR Identification of Woods for Restoration of Historic Buildings and Furniture. Applied Spectroscopy 47 (3): 287-291

Niemz, P.; K6rner, S.; Wienhaus, O.; Flamme, W.; Balmer, M. 1992: Orientierende Untersuchungen zur Anwendung der NIR-Spektroskopie für die Beurteilung des Mischungverhältnisses

Laubholz/Nadelholz und des Klebstoffanteils in Spangemischen. Holz Roh-Werkstoff. 50:25-28

Pedersen, J. G.; Hoffmeyer, P.; Jacobsen, U. G.; Reffstrup, T. 1993: Non-destructive evaluation of wood by near infrared reflectance spectroscopy. Report no. 93-1-1, Biotechnological Institute, Kolding, Denmark

Schultz, T. P.; Burns, D. A. 1992: Rapid secondary analysis of lignocellulose: comparison of near infrared (NIR) and fourier transform infrared (FTIR). Tappi Journal 73 (5): 209-212

Tsuchikawa, S.; Hayashi, K.; Tsutsumi, S. 1992: Application of NIRS to wood. pp 569-576 in: Murray, I.; Cowe, I. A.: Making Light Work: Advances in Near Infrared Spectroscopy. Weinheim 1992

170

Wallbäcks, L.; Edlund, U.; Nordén, B.; Iversen, T. 1991a. Multivariate characterization of pulp. Part 1. Spectroscopic characterization of physical and chemical differences between pulps using 13C, CP/MAS, NMR, FT-IR, NIR and multivariate data analysis. Nordic Pulp and Paper Research Journal 6 (2): 74-80, 94

Wallbäcks, L.; Edlund, U.; Nordén, B.; Iversen, T. 1991b: Multivariate characterization of pulp. Part 2. Interpretation and prediction of beating processes. Ibid. 6(3): 104-109