RAPID PREDICTION OF BASIC WOOD PROPERTIES BY NEAR INFRARED SPECTROSCOPY*

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ABSTRACT

The optimal utilisation of a wood raw material is dependent on the wood properties. In this study near infrared (NIR) spectroscopy was used to non-destructively predict density, modulus of elasticity, and modulus of rupture for small clear specimens cut from *Picea abies* (L.) Karst. (Norway spruce) trees. NIR spectra were recorded directly on the wood surface of each specimen as close as possible to the fracture developed during the bending test. Models were calibrated using partial least squares regression. The validation method was test set validation by data splitting. The correlation between predicted and measured values was highest for modulus of elasticity (0.86), followed by modulus of rupture (0.84) and density (0.79). The validation of the models showed that the average accuracies of predictions were 20.3 kg/m^3 for density, 1.1 GPa for modulus of elasticity, and 6.1 MPa for modulus of rupture. Results are comparable to what has been found for other tree species, such as *Pinus radiata* D. Don (radiata pine), *Pinus taeda* L. (loblolly pine), and *Larix decidua* Mill. (European larch).

Models for predicting density, modulus of elasticity, and modulus of rupture based on NIR spectra outperformed simple regression models using the mean annual ring width as the independent variable. NIR spectroscopy is a rapid tool for characterising organic materials. It requires minimal sample preparation and spectra are collected on solid wood, rapidly and non-destructively. For these reasons the method should be tested in production lines for lumber. In future research we aim for reliable predictions of mechanical properties of industrially manufactured lumber using models based on NIR spectroscopy and multivariate statistical methods.

Keywords: near infrared spectroscopy; partial least squares; wood density; modulus of elasticity; modulus of rupture; *Picea abies*.

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INTRODUCTION

The properties of wood as a raw material are decisive for the quality of the final products. Wood density is an important indicator of wood physical and mechanical properties, pulp properties, and pulpwood productivity. Many timber products have a load-carrying function and should therefore have sufficient strength to guarantee the desired level of structural safety and sufficient stiffness to meet the stability requirements. Bending stiffness and bending strength are wood properties that strongly affect the success of the products used in load-carrying constructions. Bending stiffness is measured as the modulus of elasticity (MOE), or the beam's resistance to bending. Bending strength is measured destructively as the modulus of rupture (MOR), which measures the maximum load a beam will carry before it ruptures. Modulus of elasticity and modulus of rupture have been shown to be correlated (Kliger *et al.* 1998), and affected by wood properties such as knots (Hoffmeyer 1987), annual ring width (Kliger *et al.* 1995), log taper (Oja *et al.* 2001), wood density (Johansson 1997), spiral grain (Dinwoodie 2000), and microfibril angle (Cave & Walker 1994).

The variation in wood properties is tremendous. In many parts of the world where the raw material to a large degree consists of wood from fast-grown plantations, with significant proportions of juvenile wood, there are serious concerns about decreased stiffness and strength. Slower growth and higher ages at final felling have minimised this problem in Scandinavia. However, wood from fast-grown plantations will in future represent a higher proportion of the wood supply in Scandinavia too.

In lumber manufacturing, testing and sorting are carried out mainly after the sawing operation. Which dimensions to produce is determined from the diameter and length of the log, and to a smaller degree from its internal wood quality. Knowledge about the variation between and within trees can be valuable for the development of tools for prediction of mechanical properties of wood prior to lumber manufacturing. This would improve possibilities for better utilisation of the raw material, as wood can be more appropriately allocated to final products. Several techniques, such as X-ray log scanning (Oja *et al.* 2001), optical log scanning (Jäppinen & Beauregard 2000), and acoustics (Tsehaye *et al.* 2000) have been reported capable of sorting logs according to mechanical properties of the produced lumber. It has also been shown that measurements taken on standing trees in the forest and on unprocessed logs can be utilised successfully in models for predictions of modulus of elasticity and modulus of rupture of the produced lumber, and the mean annual ring width has been identified as an important variable (Haartveit & Flæte 2003). These techniques require either expensive and time-consuming measurements and analyses, or an expensive and complex storing of information about each individual log in an external database.

A requirement for techniques used in industrial applications is for results to be produced fast, so that the speed of the production line is not affected. NIR spectroscopy requires minimal sample preparation, and spectra can be collected rapidly and non-destructively. The method is particularly suited for characterising organic materials. NIR instruments are at present used in production lines, e.g., for sorting organic waste. NIR spectroscopy has also been used to model yield and cellulose content of pulps (Wright *et al.* 1990). A comprehensive overview of applications of NIR spectroscopy on trees and wood has been provided by Kelley *et al.* (2004). NIR spectroscopy has a large potential as an alternative to time-consuming and/or expensive analyses. Several applications of NIR spectroscopy are relevant for this study. NIR spectroscopy has been used to predict wood density of several tree species, for example European larch (Gindl *et al.* 2001), Norway spruce (Thygesen 1994; Hoffmeyer & Pedersen 1995; Hauksson *et al.* 2001), *Eucalyptus delegatensis* R.T.Baker (Schimleck *et al.* 2001), *Eucalyptus globulus* Labill. (Schimleck *et al.* 1999), and loblolly pine (Schimleck *et al.* 2003). Modulus of elasticity of small clear specimens of radiata pine has been modelled using NIR spectroscopy (Thumm & Meder 2001), and Kelley *et al.* (2004) and Gindl *et al.* (2001) used NIR spectroscopy to predict modulus of elasticity and modulus of rupture of loblolly pine and European larch, respectively.

A NIR spectrum contains chemical and physical information about a sample. Various compounds in biological materials have overlapping peaks in the spectra, making multivariate data analysis compulsory. The data from a NIR spectrum may consist of thousands of variables measured for each sample. Each variable corresponds to the reflectance or transmittance measured from each wavelength. Projection to Latent Structures using Partial Least Squares methodology (Martens & Næs 1989) is applied to overcome problems encountered when using numerous and correlated variables as independent variables in the X-matrix for prediction purposes. NIR analysis relies on developing a calibration model that relates the NIR spectra of a large number of samples to their corresponding values for the response variable, measured by a reference method. The calibration model is then used to predict new samples based on their NIR spectra.

The objective of this study was to evaluate NIR spectroscopy combined with multivariate data analysis as a tool to non-destructively predict density, modulus of elasticity, and modulus of rupture of Norway spruce wood.

MATERIALS AND METHODS

Sampling of Wood Specimens

The wood specimens used in these experiments were collected from Norway spruce trees grown in plantations at seven different locations north of the Arctic Circle in Norway.

A 500-m^2 circular sample plot was established in the centre of each stand. Four trees were randomly sampled within each sample plot, except from one stand where five trees were randomly sampled. The mean age of the sampled trees was 49 years, ranging from 30 years to 72 years. Three small clear specimens (20 × 20 mm and 340 mm long) were prepared from air-dried wood from each stem, one from each of three different radial positions in the cross section: close to the pith, in the middle of the cross section, and close to the cambium. The specimens were collected 1.5 –2.5 m above the base of the tree. One of the specimens located close to the cambium had to be discarded because of defects, giving in total 86 small clear specimens.

Measurements of Density, Modulus of Elasticity, and Modulus of Rupture

The specimens were conditioned in standard environment to 12% wood moisture content. Wood density was measured at 12% moisture content according to ISO 3131 (ISO 1975a).

Modulus of elasticity and modulus of rupture were determined according to ISO 3349 (ISO 1975b) and ISO 3133 (ISO 1975c). The load was applied on radial surfaces (tangential bending).

NIR Spectroscopy

After mechanical testing the specimens were stored in a room without regulation of the air humidity at approximately 20ºC. One wood sample (20 × 20 × 30 mm) was cut from each wood specimen as close as possible to the fracture resulting from the modulus of rupture measurements (Fig. 1). NIR spectra were recorded directly on the cross-sectional wood surface adjacent to the fracture of each specimen. Scanning of spectra was performed in reflectance mode, in the 700–2500 nm range, in 0.35-nm steps, by a PerkinElmer Spectrum One NTS system equipped with a

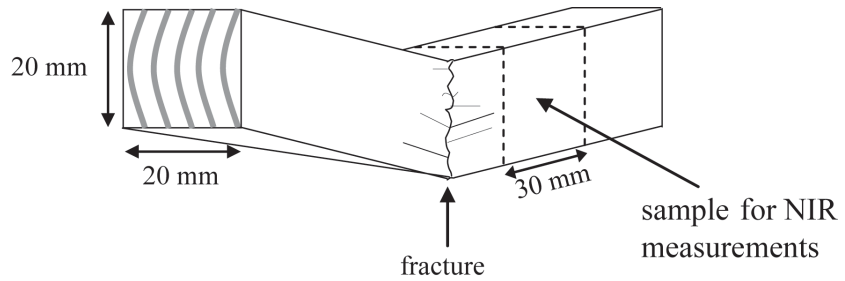

FIG. 1–A wood specimen after modulus of rupture measurements. The sample for NIR measurements is indicated.

Near Infrared Reflectance Accessory package. The specimens were placed directly on the circular sapphire window which had a diameter of 10 mm. For each spectrum 25 scans were collected and averaged. Spectra below 900 nm were discarded because of high levels of noise observed in the region 700–900 nm. Before analysis the wavelength variables were reduced by a factor of 3 by averaging.

Statistical Analyses

The NIR spectra are complex, with numerous broad overlapping bands (Fig. 2), and there are usually strong correlations between variables in the X-matrix. This makes it necessary to use multivariate statistical methods when analysing relationships between the spectra and the response variable.

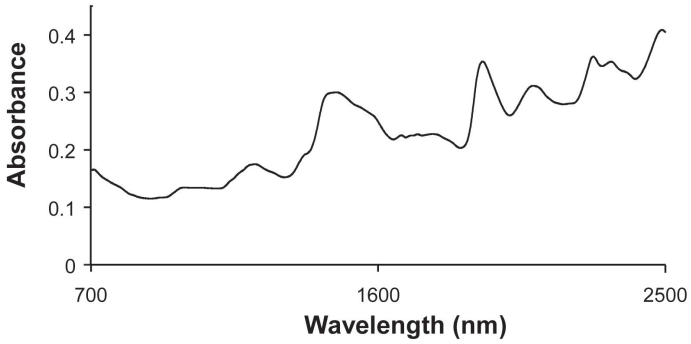

FIG. 2–The average NIR spectrum for the 86 wood specimens.

All models were calibrated and validated using partial least squares 1 regression. Partial least squares 1 is a linear modelling method that compresses the spectral data and projects them on to partial least squares components. The partial least squares 1 method extracts the spectral information with the largest covariance to the dependent variable (Martens & Næs 1989). Partial least squares components are mutually orthogonal, thus avoiding problems related to co-linearity among the variables in the X-matrix. The partial least squares method is also well suited to analyse NIR data with numerous variables in the X-matrix.

The reflectance data were expressed as apparent absorbance (log 1/reflectance). All spectra were centred before analysis. Multiplicative scatter correction was used to transform the data, in order to compensate for multiplicative and additive scatter effects in the spectra (Martens & Næs 1989). Scatter effects are effects caused by physical phenomena, such as particle size, rather than chemical properties. They interfere with the relationship between chemical properties and shape of the spectrum (Esbensen 2000). Multiplicative scatter correction tends to simplify the

calibrated model by reducing the number of partial least squares components required, and in many cases improving prediction results (Næs *et al.* 2002). The analyses were also performed on the original NIR spectra, and first and second derivative spectra. The multiplicative scatter correction pre-treatment gave better results, and only these will be reported.

The mean annual ring width has been an important variable in prediction models for modulus of elasticity and modulus of rupture in lumber (Haartveit & Flæte 2003). For small clear samples the annual ring widths are possible to measure quickly and relatively easily. Prediction models using the mean annual ring width to predict density, modulus of elasticity, and modulus of rupture were therefore compared with the models based on NIR spectroscopy.

All statistical analyses were performed using The Unscrambler® version 9.2.

Model Validation

Model validation was performed using test set validation by data splitting. For each of the three wood properties (density, modulus of elasticity, and modulus of rupture), the test set was selected as follows: first the data were sorted into ascending order according to the wood property of interest. Thereafter every fourth specimen was selected as a test set. This ensured that specimens covering the complete range of values were present in the test set.

The modelling error is estimated as the difference between the calibrated values of the response variable ($\hat{y}_{cal}$) and the measured values of the response variable (y_{cal}) for each wood sample.

$$Modelling\ error = (\hat{y}_{cal} - y_{cal})$$

The sum of the squared differences over all n wood samples gives the calibration residual Y-variance. The square root of the calibration residual Y-variance is referred to as the root mean square error of calibration (RMSEC), and is defined (Esbensen 2000) as:

$$RMSEC = \sqrt{\frac{\sum_{i=1}^{n} (\hat{y}_{cal,i} - y_{cal,i})^2}{n}}$$

where $\hat{y}_{cal,i}$ is the fitted value of sample i,
$y_{cal,i}$ is the observed value of sample i, and
n is the number of samples in the calibration set.

The root mean square error of prediction (RMSEP) estimates the expectation of the average error when predicting new samples, expressed in the same unit as the dependent variable. It is the square root of the residual validation variance and is defined (Esbensen 2000) as:

$$RMSEP = \sqrt{\frac{1}{N} \sum_{i=1}^{N} (\hat{y}_{val,i} - y_{val,i})^2}$$

where $\hat{y}_{val,i}$ is the predicted value of sample i,
$y_{val,i}$ is the measured value of sample i, and
N is the total number of samples in the test set.

The root mean square error of calibration is reduced when the number of partial least squares components added to the model increases. The decision regarding how many to include in the model is therefore difficult to make solely by evaluating the root mean square error of calibration. However, as more components are added to the model, the residual validation variance tends to increase.

The goodness of the models was evaluated using the correlation between observed and fitted values for model calibration (r_{cal}) and between observed and predicted values for model validation (r_{val}) in addition to the root mean square error of calibration and the root mean square error of prediction.

It is generally seen as advantageous for models to have a small number of partial least squares components. As more are added to a model, the residual validation variance decreases to a minimum before it starts increasing. The number of partial least squares components that minimises the residual validation variance is often considered optimal. Software designed to analyse NIR data takes this into account and the model with the lowest residual validation variance determines the optimal number of partial least squares components. Even though addition of further partial least squares components to the model will reduce the residual calibration variance, the prediction errors will increase, and the model becomes overfitted. An overfitted model has incorporated information not relevant to Y into the model structure, resulting in poorer predictions of new samples.

In order to compare the prediction abilities for the three wood properties, the relative prediction error (RPE) was used. Relative prediction error is defined (Martens & Næs 1989) as:

$$RPE = \frac{(VAR_y - MSEP)}{Var_y}$$

where Var_y is the variance of the response variable, and
MSEP is the mean square error of prediction.
RPE values close to 1 indicate excellent prediction ability, and
RPE values close to 0 indicate poor prediction ability.

RESULTS

Descriptive statistics for the calibration set, the test set, and the complete data set are provided in Table 1. For all models, the specimens used for modelling consisted of 86 samples, of which 21 were selected as a test set.

TABLE 1–Basic statistics for wood density, modulus of elasticity, and modulus of rupture for the calibration set, the test set, and the complete data set

Wood property	Data set	n	Mean	Standard deviation	Min	Max
Density (kg/m^3)	Calibration	65	392.9	34.6	317.2	454.9
Density (kg/m^3)	Test	21	394.7	33.1	331.0	453.2
Density (kg/m^3)	Complete	86	393.4	34.1	317.2	454.9
MOE (GPa)	Calibration	65	10.4	2.3	5.5	16.2
MOE (GPa)	Test	21	10.4	2.2	6.7	14.9
MOE (GPa)	Complete	86	10.4	2.3	5.5	16.2
MOR (Mpa)	Calibration	65	71.2	11.9	44.1	96.4
MOR (Mpa)	Test	21	71.8	11.3	51.5	92.3
MOR (Mpa)	Complete	86	71.4	11.7	44.1	96.4

Wood Density

A simple regression model using the mean annual ring width as the independent variable was calibrated and validated. The calibration resulted in root mean square error of calibration = 30.0 kg/m^3 and r_{cal} = 0.49, while model validation resulted in root mean square error of prediction = 29.0 kg/m^3 and r_{val} = 0.47. For the calibrated model based on NIR spectra, the optimal number of partial least squares components was 3. For the model calibration, root mean square error of calibration was 20.3 kg/m^3 and r_{cal} was 0.81. The model validation yielded similar results, as root mean square error of prediction was 20.3 kg/m^3 and r_{val} was 0.79 (Fig. 3). Hence, calibration as well as validation results showed that models using NIR spectra

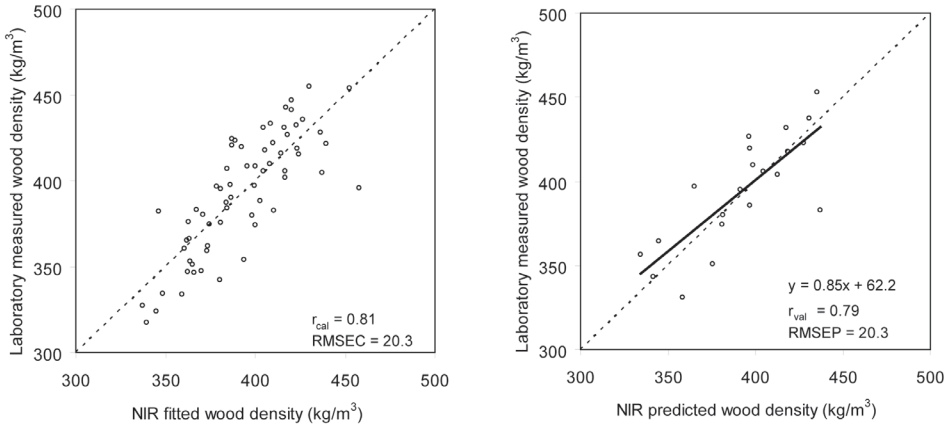

FIG. 3–Results from model calibration (left, n = 65) and validation (right, n = 21) for wood density. The number of partial least squares components is 3. Solid line: regression line for *measured vs. predicted wood density*; dashed line: target line (y = x).

outperformed the simple regression model based on the mean annual ring width as the predictor.

Modulus of Elasticity

A simple regression model using the mean annual ring width as the independent variable was calibrated and validated. The calibration resulted in root mean square error of calibration = 1.75 GPa and r_{cal} = 0.65, while model validation resulted in root mean square error of prediction = 1.71 GPa and r_{val} = 0.61. For the calibrated model based on NIR spectra, the optimal number of partial least squares components was 7. For the model calibration, root mean square error of calibration was 1.16 GPa and r_{cal} was 0.87. The model validation yielded similar results as root mean square error of prediction was 1.09 GPa and r_{val} was 0.86 (Fig. 4). Hence, r_{cal} and r_{val} were higher for modulus of elasticity than for wood density. Also, for modulus of elasticity the model based on NIR spectra outperformed the simple regression model with the mean annual ring width as the independent variable.

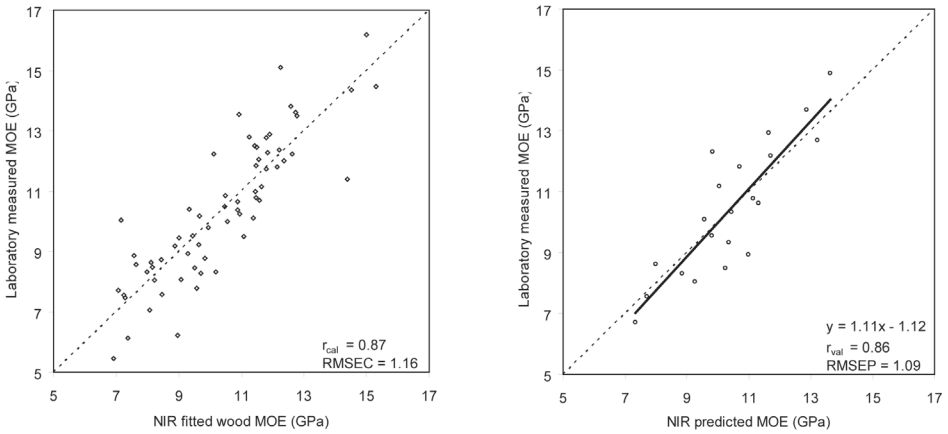

FIG. 4–Results from model calibration (left, n = 65) and validation (right, n = 21) for modulus of elasticity (MOE). The number of partial least squares components is 7. Solid line: regression line for *measured vs predicted modulus of elasticity*; dashed line: target line (y = x).

Modulus of Rupture

A simple regression model using the mean annual ring width as the independent variable was calibrated and validated. The calibration resulted in root mean square error of calibration = 9.43 MPa and r_{cal} = 0.60, while model validation resulted in root mean square error of prediction = 8.36 MPa and r_{val} = 0.72. For the calibrated model based on NIR spectra, the optimal number of partial least squares components was 5. For the model calibration, root mean square error of calibration was 6.33 MPa and r_{cal} was 0.84. The model validation yielded similar results, as root mean

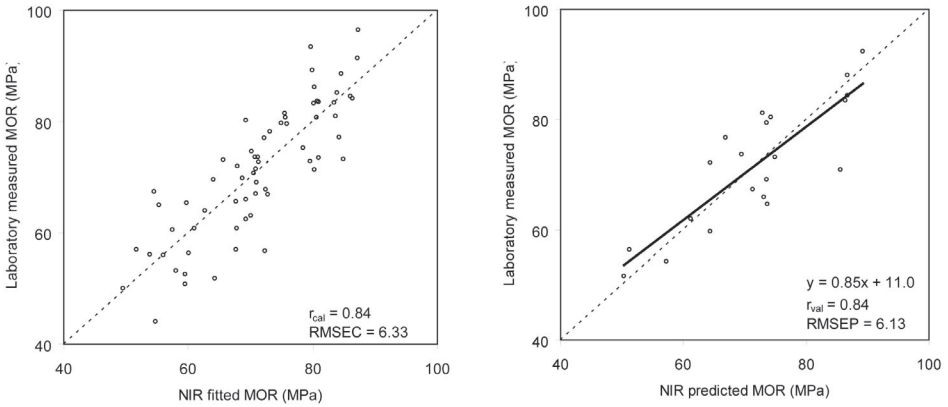

FIG. 5–Results from model calibration (left, n = 65) and validation (right, n = 21) for modulus of rupture (MOR). The number of partial least squares components is 5. Solid line: regression line for *measured vs. predicted modulus of rupture*; dashed line: target line (y = x).

square error of prediction was 6.13 MPa and r_{val} was 0.84 (Fig. 5). Hence, the correlation between predicted and observed values was not as strong as the correlation presented for modulus of elasticity, but was better than the corresponding correlation presented for wood density. For modulus of rupture too, the NIR-based models outperformed the simple regression model using the mean annual ring width as the independent variable.

To compare models for different wood properties, the relative prediction error (RPE) was used. Wood density had the smallest values of relative prediction error (0.64) followed by modulus of rupture (0.72) and modulus of elasticity (0.77).

DISCUSSION

Based on the presented results, we found that NIR spectroscopy combined with partial least squares models is a technique well suited for predictions of wood density, modulus of elasticity, and modulus of rupture. Measured in terms of the validated correlation coefficient (r_{val}), the best predictions were obtained for modulus of elasticity, followed by modulus of rupture and wood density. This is also reflected by the relative prediction errors that were smallest for wood density, followed by modulus of rupture and modulus of elasticity.

Hoffmeyer & Pedersen (1995) evaluated NIR spectroscopy for predicting wood density of Norway spruce clearwood specimens. As in the present study, the spectra were measured on cross sections. Root mean square error of calibration and root mean square error of prediction values were higher than found in the present study when they used a model with 4 partial least squares components and at the same

level as the present study for a model with 8 partial least squares components. In the present study, wood density was predicted with a model using only 3 partial least squares components and the risk of overfitting the model was therefore regarded as small. Hoffmeyer & Pedersen (1995) found a higher correlation between NIR-predicted and measured wood density than in our study. A likely explanation for this is that the range of density was greater (350 kg/m^3–580 kg/m^3) compared with the material in our study (317 kg/m^3–455 kg/m^3).

NIR spectroscopy has also been used for predictions of modulus of elasticity of small clearwood specimens of radiata pine by Thumm & Meder (2001). Spectra were recorded on one radial and one tangential face of each specimen, as the specimens were moved past a NIR detector. Prediction models based on spectra recorded on radial wood faces performed better than models based on spectra recorded on tangential faces. The present study did not allow for a similar comparison.

Kelley *et al.* (2004) measured modulus of elasticity and modulus of rupture of loblolly pine wood in a three-point bending test. NIR spectra collected from the radial face of solid wood specimens were used to construct partial least squares-2 models for modulus of elasticity and modulus of rupture. Their analyses were similar to the present study, although the experimental design was based on collecting all wood specimens from three different trees. The calibrated model was based on full cross validation, and approximately one-third of the samples were randomly selected as a test set. The range of modulus of elasticity and modulus of rupture was slightly wider for Kelley *et al.* (2004) than for our study. The root mean square error of prediction values, as well as the validated correlation coefficients, were comparable to the results reported here.

Gindl *et al.* (2001) used NIR spectra collected on the radial surface of European larch to model wood density, modulus of elasticity, and modulus of rupture. However, Gindl *et al.* (2001) had a material with considerably larger variations with respect to density, modulus of elasticity, and modulus of rupture. The larger range of values observed for all wood properties examined by Gindl *et al.* (2001) explains why their correlations between predicted and observed values were higher than in the present study. When comparing the estimate of the average error of prediction, the present study had slightly lower values than those of Gindl *et al.* (2001). Note, however, that the results reported by those authors were based on full cross validation, which increases the risk of over-estimating the prediction abilities of partial least squares models (Esbensen 2000).

The ranges of wood density, modulus of elasticity, and modulus of rupture (Table 1) were moderate compared with previous Norwegian studies from the same region (Okstad & Kårstad 1985). Despite the moderate range of the response variables, the presented prediction models performed well, even when compared

to studies with a larger range of values with regard to wood density (Hoffmeyer & Pedersen 1995; Gindl *et al.* 2001), modulus of elasticity (Thumm & Meder 2001; Kelley *et al.* 2004), and modulus of rupture (Gindl *et al.* 2001). Increasing the variance of the response variable commonly leads to improved predictions.

It has been reported that for wood specimens of radiata pine with high values of modulus of elasticity, predictions based on NIR measurements result in an under-estimation of modulus of elasticity (Thumm & Meder 2001). It is suggested that this is caused by a kind of saturation effect. This means that spectra of wood specimens with high modulus of elasticity show much smaller responses to differences in modulus of elasticity values than spectra from specimens with lower modulus of elasticity values. We did not find evidence of a similar effect for Norway spruce, but it possibly exists for samples with higher values of modulus of elasticity than found in the present material.

When comparing results from different studies using NIR spectroscopy and partial least squares modelling, model validation is a key issue. The partial least squares methods have been developed in the field of chemometrics (Martens & Næs 1989), and the purpose has commonly been to develop models for prediction purposes. Comparisons between different studies should be made with care if different methods for model validation have been used.

Two methods for model validation are test set validation and cross validation. When using test set validation a new individual and independent sampling of new observations is used for model validation. Test set validation is always the preferred method (Esbensen 2000). When only one sampling exists, an approximation to test set validation is performed by splitting the data set in two parts. The first part is used for model calibration, and the remaining samples are used for model validation. In the absence of a true test set from a new and independent sampling, the present study was validated using test set validation by data splitting. A new independent sampling is, however, a more reliable method of validation (Esbensen 2000; Kozak & Kozak 2003).

Cross validation involves iteratively excluding one or a group of observations from model calibration, and thereafter using the calibrated model to predict the excluded samples. This procedure is repeated until all samples have been excluded and predicted. When only one observation is excluded iteratively it is referred to as full cross validation; excluding a group of observations iteratively is referred to as segmented cross validation. Full cross validation is criticised for over-estimating the predictive capabilities of the regression models, since removing only one observation from the data hardly affects the y-x relationship (Esbensen 2000). When using segmented cross validation, the data are usually separated into segments using random or systematic selection. An alternative is to divide the data

into segments based on natural partitions of the data — for example, when the complete sample is collected from different geographical locations. In such cases the data can be divided into segments based on growth location (Haartveit *et al.* 2003). The root mean square error of prediction values and validated correlation coefficients resulting from any cross validation procedures are averages of the results obtained for all segments. Hence, differences in the model's ability to predict the segments are not discovered.

Conservative validation methods such as test set validation will reduce the probability of over-estimating the predictive capabilities of the models compared to full cross validation. When the objectives are reliable predictions, conservative validation methods are preferred.

CONCLUSIONS

This study showed that, compared with the mean annual ring width, NIR spectroscopy is superior in predicting density, modulus of elasticity, and modulus of rupture of small clear specimens of Norway spruce.

Haartveit & Flæte (2003) used stand and tree characteristics to predict modulus of elasticity and modulus of rupture of Norway spruce structural lumber. The models showed promising results with respect to pre-sorting of logs in order to create a better fit between the quality of logs and the expected properties of the final products. Since NIR spectroscopy is a very rapid technique, it is of interest to investigate if NIR spectra obtained on logs can be used to predict modulus of elasticity and modulus of rupture of the lumber. Another application of interest is to evaluate NIR spectroscopy for on-line strength grading of structural lumber.

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