Wood - an anatomical structure in the tree and an engineering material in industry:

Prediction of material properties in managed Scots pine stands in the forest

Daniel Erikssson Faculty of Forest Sciences Department of Forest Ecology and Management Umeå

Doctoral Thesis Swedish University of Agricultural Sciences Umeå 2008 Acta Universitatis Agriculturae Sueciae 2008 : 73

ISSN 1652-6880 ISBN 978-91-86195-06-9 © 2008 Daniel Eriksson, Umeå Tryck: Arkitektkopia, 2008 Wood - an anatomical structure in the tree and an engineering material in industry: Prediction of material properties in managed Scots pine stands in the forest

Abstract

The overall objective of this thesis was to create a system for characterizing the material properties of wood, analogous to those used to specify other materials. In order to obtain homogenous materials prediction methods were evaluated. Therefore, one objective was to evaluate the scope for using structural wood properties to predict mechanical properties (Study I) and further develop the functions (Study IV). Another objective was to evaluate the effects of two different silvicultural regimes on the wood structure characteristics and mechanical properties of different wood tissue types at different heights in the tree, and reasons for these differences (Study II). In addition, a simple, fast and reliable light microscopy method for analyzing microfibril angle was developed, and its utility was assessed (Study III). Further objectives were to evaluate the utility of biorthogonal partial least squares regression models, using near infrared NIR spectral data, for predicting material properties and classifying wood as juvenile or mature wood (Study V). The papers (I, II, IV and V) are also used in this thesis as a basis to discuss how to use structural properties and near infrared spectroscopic data to characterize the material properties of trees in the forest.

In Study IV, in total 200 clear wood samples from 24 Scots pine (Pinus sylvestris L.) trees in 18 stands were used in the analyses, while 100 samples from eight trees in two stands were used in studies I - III and V. The samples (especially those used in Study IV) spanned most of the ranges and combinations of structural and mechanical property values found in Scandinavian forests, thus diminishing the risk of random covariance between predictors and properties of interest. The ability to predict a mechanical property generally increased for each of the three examined resolution levels of determination of structural properties with r² values often reaching 0.9 at the highest resolution (Study I and IV). Silvicultural regimes clearly have great potential to influence material properties, in terms of both differences in average levels and trends in vertical and radial directions within trees (Study II). The method developed for determining microfibril angles provided results that were almost identical to those obtained using a commonly applied method, but it was easier to perform and thus cheaper (Study III). The modelling of near infrared spectra was able to predict both mechanical and structural properties at high accuracy (StudyV). The possibilities with proposed application model modelling material properties of individual annual rings are great combining, e.g., a combination of several material properties according to a property table. Thus, wood could be matched, according to its specific material properties, to appropriate products, and thus increase the income for all parts throughout the forest-wood chain.

Keywords: Property tables, mechanical properties, structural properties, silvicultural regimes, NIR – Spectroscopy.

Author's address: Daniel Eriksson, Swedish University of Agricultural Sciences, Vindeln Experimental Forests, Svartberget Field Station, SE-922 91 Vindeln, Sweden.

Contents

List of Publications	7
Introduction	9
Objectives	13
Material and methods	15
Samples	15
Sub-samples	17
In Study III	19
In Study V	20
Statistics	20
Results and Discussion	22
Material and methods used	22
Study I	25
Study II	26
Study III	27
Study IV	29
Study V	30
Material characterization models – <u>Applications</u>	30
Conclusions and need for further research	36
Acknowledgements	38
References	39

List of Publications

The presented thesis is based on the following papers, which will be referred to in the text by their corresponding Roman numerals.

- I Eriksson, D., Lindberg, H. & Bergsten, U. (2006a) Prediction of mechanical properties of *Pinus sylvestris* wood from wood structure characteristics. In: Kudela, J. & Kurjatko. (ed), *Proceedings of thr 5th IUFRO Symosium Wood structure and properties'06*, Sielnica, 3-6 september, 2006, 217–226. Zvolen, Arbora Publishers.
- II Eriksson, D., Lindberg, H. & Bergsten, U. (2006b) Influence of silvicultural regime on wood structure characteristics and mechanical properties of clear wood in *Pinus sylvestris*. *Silvia Fennica* 40(4), 743–762.
- III Eriksson, D., Lindberg, H. & Bergsten, U. Microscopic determination of microfibril angles in cut sections of macerated tracheids of Scots pine. (Manuscript).
- IV Eriksson, D. Prediction of material properties in managed Scots pine forests using wood and tree structure characteristics. (Manuscript).
- V Lestander, T.A., Lindeberg, J., Eriksson, D. & Bergsten, U. (2008) Prediction of *Pinus sylvestris* clear-wood properties using NIR spectroscopy and biorthogonal partial least squares regression. *Can. J. For. Res.* 38, 2052–2062.

Papers I, II and V are printed with the kind permission from the publishers.

Introduction

Forests are great resources of material with wide ranges of diverse parameters. Further, it is possible to adjust the properties of the wood they produce by applying appropriate silvicultural treatments in order to set stands' density, volume and tree size distributions at various points in time as they grow, and thus influence growth- and material-related parameters (Macdonald & Hubert 2002, Mattsson 2002, Pape 1999, Zhu et al. 2007). In addition, high wood production and use have positive environmental effects since forests fix large amounts of carbon dioxide, and large amounts of energy can be saved if wood is used rather than alternative materials, such as concrete or plastics, in many applications. Moreover, wood has great potential to compete with other materials because its mechanical parameters are very competitive, especially relative to its density (Ashby 2000). However, a major current problem is the lack of thorough characterization of wood, and consequently customer complaints that woody raw materials do not always meet their requirements.

The pulp, paper, sawmill and bioenergy industries use most of the wood produced from boreal forests. Properties of interest for the pulp and paper industries include (*inter alia*) the wood's density, chemical composition, fibre anatomy/morphology and microfibril angle, since they affect the yield and properties of the paper, partly due to their effects on maceration and bleaching processes (Sjöström & Alén 1999). In the sawmill industry the products' usefulness and value strongly depend on the wood's modulus of rupture and risk of dimensional instability, e.g. warps (Saarman 1992). For example, construction timber is graded in classes, in terms of its modulus of rupture (Saarman loc cit.), while density and both the moisture and ash contents of wood greatly affect the amount of energy produced and the service intervals of equipment used in the bioenergy industry.

Many activities in the forest-wood chain are regulated by the grading rules for timber and pulpwood payments decided (in Sweden) by Timber Measurement Associations. Forest owners sell wood according to agreements based on the yields of appropriate products, costs of possible alternatives and the customers' willingness to pay, i.e. essentially the basic laws of supply and demand. Grading are based on logs position in trees and measurements of the sizes and visible features of logs, e.g. their curvature, knots, rotten wood, annual ring width (2 - 8 cm from the pith), handling injuries and other defects (VMR 2008). The determined features (e.g., the type, size and amount of knots) correlate somewhat to strength properties (Dinwoodie 2000). However, even though additional sorting is applied in the board industry, and the old grading rules for payments included more classes and features of logs, Saarman (1992) concluded that the strongest pieces may be three to four times stronger than the declared value of construction timber. The value (and hence price) of wood used to create structural units, such as boards, mainly depends on its clear wood properties, but the volume fraction and angle of knots also affect its value (Xu 2002). Further, due to the poor correlation with clear wood properties, such grading cannot distinguish between juvenile and mature wood, which generally have very different values of various parameters, and if specimens contain a mixture of these tissue types they will be prone to wide variations in dimensional instability (Dinwoodie loc cit.). Furthermore, grades of material such as pulpwood have little relevance to the abovementioned clear wood properties that affect yields and properties of the resulting paper, and hence the true value of the material for papermakers. In addition, sawmill chips are often characterised as by-products, comprising mixtures of all possible wood materials from the source region. Finally, industries later in the forest-wood chain incur large costs due to the need to sort the woody raw materials they handle, since some will not meet their customers' requirements. Therefore, more thorough description of wood in terms of important properties before it leaves the forest is essential in order to specify its properties more comprehensively, sort it accordingly for specific uses and customers, and thus optimise the management of these important resources.

Learning from the successes for steel, plastics and other engineering material wood should be specified according to a material scientific approach and thus wood could be included in common material databases, thereby making it possible to be chosen as an alternative in products (Bowyer 2000). In order to produce and market sophisticated, highly-priced wooden materials highly accurate methods are required that are capable of distinguishing material with specified qualities within narrow ranges of parameters from extremely diverse pools of materials. Cheap, somewhat less accurate methods are probably adequate for today's industry, since the net values of the products are low. However, if convenient, accurate methods for determining wood's material properties were developed they would probably promote the emergence of an increasingly strong and sophisticated wood industry, since its customers/ users would then have greater access to a wider range of materials, and more information about them. The superior clearwood could also be used more exclusively in appropriate products to avoid reducing their potential value, by excluding defects from structural wood (Dinwoodie 2000). Additionally,

as the variability of properties within pieces of clear wood can be low the full potential of the diverse pool of wood could be used, e.g. by identifying clear wood suitable for use as finger-jointed material. More comprehensive analyses of clear wood properties could also provide ways to explain and mitigate the heterogeneity of materials in the current wood industry, and counter problems such as those associated with juvenile wood.

Structural material properties (e.g. density, tracheid cell wall thickness, tracheid length, tracheid diameter, proportion of latewood and microfibril angle) are correlated, to varying degrees, to mechanical material properties (e.g. stiffness, bending strength, compression strength, creep, and Brinell hardness in tangential and longitudinal directions; Bao et al. 2001, Cameron & Dunham 1999, Groom et al. 2002, Holmberg 2000, Mencuccini et al. 1997, Persson 2000, Raymond et al. 2004, Yang & Fortin 2001, Xu et al. 2004). Hence, it should be possible to use readily measured structural parameters to estimate important material properties. In order to reduce the risk of random covariance, and the need for extrapolation, the material used to generate appropriate equations for such purposes should ideally cover large proportions of wood resources, in terms of both material properties and other properties. Thus, potential methods to assess material properties, and silvicultural treatments to adjust them, should be evaluated in tests using material that spans wide ranges of material parameters, and wide ranges and permutations of known nonmaterial variables (e.g. annual ring width, cambial age, heartwood/sapwood tissue type, and tree structural properties, such as the age of the apical meristem, heights in trees, proportion of green crown and distance to the green crown) (Amarasekara & Denne 2002, Brüchert et al. 2000, Groom et al. loc cit., Kliger et al. 1995, Lindström 1996, Macdonald & Hubert 2002, Mencuccini et al. loc cit., Olesen 1982, Pape 1999, Zhu et al. 2007). The non-material properties considered in the cited studies to explain material properties are sets of growth-related parameters, such as the age of the trees, silvicultural treatments, site indexes and climatic factors. However, in all of the studies, including those using structural material properties (see above), only one or a few of the properties were used to explain any given material property. Instead multiparametric equations should be used as, e.g., values on microfibril angle can explain some of the variations in stiffness not explained by values on density (Persson 2000). Possible options for improving predictions include the development and use of a light microscopic method to measure microfibril angle that is less laborious than the method presented by Senft & Bendtsen (1985), but cheaper than the X-ray diffraction technique proposed by Evans (1999), which requires expensive equipment. The technique of Senft & Bendtsen (loc cit.) involves inducing checks and precipitating iodine crystals in cavities in the tracheid cell wall. The orientation of the microfibrils can then be discerned by the iodine crystals that tend to follow the microfibril

angle. In addition, the scope for using near infrared (NIR) spectroscopy to estimate structural properties (e.g. microfibril angle, Schimleck et al. 2001a; and mechanical properties, Gindl et al. 2001, Kelly et al. 2004 Thumm & Meder 2001) should be further explored, since it is fast, non-destructive and highly suitable for online measurements (Osborne et al. 1993).

Objectives

The overall objective of the studies this thesis is based upon was to develop a scientifically sound method for characterizing the material properties of wood, analogous to those used to specify the properties of other materials, such as steels and plastics. Specific objectives of the studies were as follows.

Study I

The objective was to evaluate the scope for using structural wood properties to predict modulus of elasticity (MOE), bending strength (f_{m}), compression strength (f_{c}) and creep in bending (Rel. MOE_{t1000h}) all at longitudinal direction and Brinell hardness at both tangential ($H_{B,90}$) and longitudinal directions ($H_{B,0}$). The general hypothesis was that the higher the resolution used in determinations of structural properties (from wood tissue type, through age of cambium and apical meristem, to ring and fibre characteristics), the better the predictions of mechanical properties should be. The presented analyses were set at three resolution levels in order to give customers an opportunity to choose the cost and accuracy of prediction. Structural characteristics, annual ring width and density. The second level analyses included first level parameters and tracheid cell morphological characteristics. The third level analyses included first and second level parameters in combination with more laborious analyses of the proportion of latewood and microfibril angle.

Study II

The objective was to evaluate the effects of two different silvicultural regimes on the wood structure characteristics and mechanical properties in study I of heartwood (Hw) and sapwood (Sw) at stump height (S) and intermediate top height (T) in the trees, and reasons for these differences. The first hypothesis was that the expected major differences in annual ring width and proportion of green crown between the chosen regimes are associated with substantial differences in the structural and mechanical properties of the wood. The second hypothesis was that the wood structure characteristics could explain the major differences in mechanical properties between the regimes. The third hypothesis was that distance to the green crown or proportion of green crown in combination with annual ring width and age characteristics could be used to explain the differences in wood structure over time between the regimes.

Study III

The objectives were to develop a simple, fast and reliable light microscopy method for analyzing microfibril angle that yielded comparable results to those obtained using the commonly applied method presented by Senft & Bendtsen (1985) and to assess its utility. The first hypothesis tested was that values acquired by the methods compared would be equal. Another tested hypothesis was that the developed method would be able to detect differences in microfibril angle between samples with expected differences, such as samples of latewood tracheids and earlywood tracheids, samples from trees grown under different silvicultural regimes and samples differing in apical meristem and cambial ages.

Study IV

The main objective was to further develop the functions based on tree characteristics and structural characteristics generated in study I to predict mechanical properties. Analyses were performed at three levels of resolution, as in study I, but structural properties, such as the trees' crown characteristics and height in trees were added to all of the functions. As in study I, the main hypothesis was that increasing the resolution of determination of structural properties would improve predictions of mechanical properties. The second hypothesis was that adding wood samples from trees representing more silvicultural treatments, tree rank classes, site fertility indices and climates to the material analyzed in study I would improve the prediction of material properties. The last hypothesis was that including structural non-material properties is important for obtaining highly accurate results.

Study V

The objectives were to evaluate biorthogonal partial least squares regression models, using near infrared spectral data, to predict the abovementioned mechanical properties, tracheid cell morphology parameters, proportion of latewood, mean microfibril angle, microfibril angle of latewood and earlywood tracheids and classify wood as juvenile or mature wood.

The papers (I, II, IV and V) are also used in this thesis as a basis to discuss how to use structural properties and near infrared spectroscopic data to characterize the material properties of logs while they are still in the forest.

Material and methods

Samples

In total 200 clear wood samples were taken from 24 Scots pine (Pinus sylvestris L.) trees in 18 stands (Fig. 2, Tables 1 and 2). All of the stands had been actively regenerated, pre-commercially thinned and thinned at least twice (except for the widely spaced stand, which had not been thinned due to its wide spacing), but they were not older than the normal clear-cutting age for pine stands in Sweden. All stands were in Sweden except numbers 17 and 18, which were in the windy south-west part of Norway. The site index, the expected height of dominant trees at 100 years old, was estimated for each of the stands according to Hägglund & Lundmark (1982), except for stand 17 (indicated by asterisks in Table 1), where the measured productivity was used to estimate the site index as if it had been grown at a wind-protected site. Additionally, within pairs of stands (see below) the estimated site index of the stand thinned from below was used to estimate the index in the comparable stand thinned from above. Basal area weighted diameter at breast height was used to define average trees, and the stem diameter at breast height was used to classify sample trees to tree rank classes as small (S), medium (M) or large (L) by measuring the distribution of stem diameters in the stand and by dividing the distribution into three classes. The samples spanned a large part of the variation found within Scandinavia due to variations between sites in terms of silvicultural regimes, geographical origins, site indices, and exposure to wind.

The material was divided into two sets. In studies I-III and V the first set was used and in study IV both the first and second sets were used. The first consisted of 100 of the 200 samples, taken from four dominant trees in the widely spaced stand and four intermediate trees in the densely spaced stand (stands 7 and 8, Tables 1 and 2). From each of these trees stem sections were taken at both stump and intermediate height (20 and 30 years up in the widely spaced and densely spaced trees, respectively). Six samples were taken from each of these trees in both stands at stump height and from the widely spaced trees at intermediate height, and seven samples were taken from each of the densely spaced trees at intermediate height.

The second set contained 100 samples from seven stands thinned from above and nine stands thinned from below (stands 1-6 and 9-18, respectively). Each of the seven sampled stands thinned from above was paired with a comparable stand thinned from below in terms of geographical location, site index, age etc. (stands 1 through 6 and 9 through 16, respectively). These pairs of stands were selected to represent three geographical areas, ranging from 59° to 67° N latitude, and a substantial range of site indices in each area (Table 1). The other two stands thinned from below were in a windy area, one in a sheltered and one in an exposed location (stands 17 and 18). Sixteen trees were sampled, one from each of these stands (randomly selected in terms of rank class). From each of these selected trees stem sections were taken at stump-, breast-, four meter -, nine meter - and 15 meter- (except for smaller trees) heights, and hence sections representing four (or five) apical meristem ages were obtained. One or two samples were taken from each stem section so, in total, six or seven samples were taken from each tree.

From each selected tree in each of the two sets, both sapwood and heartwood samples were taken with both high cambial ages (>20 years) and low cambial ages representing each wood tissue type. Stem sections were taken between two branch whorls except that sections taken at stump height contained some few branch whorls. Samples for testing wood properties related to cambial age were taken from different positions in radial directions from the stem sections, representing specific cambial ages (figure 1).

Stand no.	Latitude	Elevation	Site index	Thinning type	Remarks
	N°	(m a s)	$H_{_{100}}$		
1	67	240	18.4	Below	
2	67	240	18.4	Above	
3	66	180	24.3	Below	
4	66	180	24.3	Above	
5	64	330	19.2	Below	
6	64	330	19.2	Above	
7	64	190	22	Above	Densely spaced
8	64	170	18	None	Widely spaced
9	63	200	24.7	Below	, ,
10	63	200	24.7	Above	
11	60	180	30.5	Below	
12	60	180	30.5	Above	
13	59	80	24.3	Below	
14	59	80	24.3	Above	
15	59	80	22.1	Below	
16	59	80	22.1	Above	
17	58	300	21*	Below	Wind exposed
18	58	110	25	Below	Wind protected

Table 1. Stand characteristics of the sampled pine stands. Thinning types Below and Above refer to thinning from below and thinning from above, respectively. The asterisk (\star) indicates a site for which the site index was estimated in a different way than for the other sites, as detailed in the text.

Stand no.	Age	Trees/ha	Height of dominant	Diameter at breast height	Rank classes of
	(years)		trees (m)	of average trees (cm)	sample trees
1	78	720	15.7	18.1	L
2	78	1240	15.1	13.6	L
3	56	720	18.3	19.5	М
4	56	1480	16.6	13.6	L
5	89	460	18.1	20.0	S
6	89	930	16.0	13.8	S
7	85	1425	20.6	15.7	М
8	56	100	12.1	36.2	L
9	65	650	20.0	23.9	S
10	65	984	19.5	19.6	М
11	62	598	25.6	27.6	S
12	62	1202	22.5	19.4	S
13	80	420	22.0	25.2	L
14	80	810	20.4	18.4	М
15	79	340	20.1	22.8	S
16	79	440	17.7	20.0	S
17	84	754	13.6	18.2	L
18	81	535	22.7	22.4	L

Table 2. Stand characteristics of the sampled pine stands, and rank classes of sample trees. L indicates a large tree relative to the average tree; M indicates a medium sized tree; S indicates a small tree.

Sub-samples

Sub-samples (representing specific cambial ages) were taken from every sample for measurements of mechanical properties, as illustrated in figure 1. The same sub-samples, taken from one side at position A, were used to test longitudinal stiffness in bending and longitudinal bending strength, while sub-samples to test longitudinal creep in bending were taken from the adjacent side in the tangential direction. Sub-samples for measuring compression strength at longitudinal direction and Brinell hardness at the tangential direction were taken from either side at position B. Stem discs from position C were taken for measurements of Brinell hardness at the longitudinal direction. The subsamples consisted of clear wood without structural defects. Bending subsamples from stump height stem sections in test set 1 (see above) occasionally contained defects, but only at positions where stress was low. For five bending sub-samples with non-straight grains, it was necessary to correct the measured values using a modified form of the equation developed by Hankinson (in Dinwoodie 2000) to estimate the stiffness and bending strength values the samples would have had if they had been straight-grained. These values were only adjusted if the calculated difference between the adjusted value and nonadjusted value was 2.5% or more. Measurements of creep were performed on sub-samples of 153 samples, and all of the other tests on sub-samples of 200 samples, conditioned at 20°C and 65% air humidity according to standard method EN 408 (1995). However, sub-samples were taken for measurements of creep from all 200 samples.



Figure 1. Sampling of clear wood in stem sections and corresponding coordinate system in radial (R), tangential (T), and longitudinal (L) directions, perpendicular to each other. Wood sections A and B and stem disc C show positions at which sub-samples for different measurements were taken (see text).

The material was divided into two equal sized test sets (see above), which were conditioned and analyzed at different times, but otherwise treated as identically as possible. However, a Hounsfield 5000 Universal Testing Instrument was used for the stiffness, bending strength, compression strength and Brinell hardness tests of the first test set, while an Instron 4410 universal testing instrument was used for the other set. Creep, measured as the ratio between the initial stiffness and stiffness after 1000 hours of loading, was determined in an apparatus in which weights were hung on the samples, equivalent to ca. 20% of the maximum stress calculated from the measured bending strength. Stiffness, bending strength, creep and compression strength were measured using the protocols recommended in EN 408 (1995), except that in measurements of the first test set's stiffness the samples' deflection in the middle of the span (W_{middle}) was calculated from the deflection at the loading points ($W_{loading points}$), see eq. 1. The equation includes the sample-specific ratio between its Brinell hardness at the tangential direction $(H_{B.90})$ and stiffness at longitudinal direction calculated with an assumption of 15 percent differences in deflection between the points (MOE_{15%}). The relationship between deflection in the middle of the span and deflection at the loading points was measured for the test samples for which stiffness was assessed using the Instron 4410 instrument.

$$W_{\text{middle}}/W_{\text{loading points}} = 6.3492 \star (H_{B,90} / \text{MOE}_{15\%}) + 1.0856$$
 (1)

Brinell hardness in both the tangential and longitudinal directions was measured according to Holmberg (2000), with the diameter of indentations measured only in the radial direction.

In sub-samples taken for the creep measurements (200 in total), the tracheid cell morphology, proportion of latewood and microfibril angle of the earlywood tracheids and latewood tracheids were examined after they had been conditioned according to standard method EN 408 (1995). In addition, the annual ring width and density of every sub-sample were measured after it had been conditioned. The measured latewood proportions of sub-samples taken for measuring creep were used to estimate latewood proportions in comparable sub-samples, by adjusting according to the differences in density between them. Mean microfibril angles were calculated from the mean angles of earlywood tracheids and latewood tracheids, and the proportion of latewood in each sub-sample. Parameters derived by increasing the proportional weights of latewood tracheids' microfibril angles four-fold and six-fold were tested to evaluate whether angles of latewood tracheids are more important than angles of earlywood tracheids for predictions of mechanical properties (cf. Senft & Bendtsen 1985). Light microscopy was used for measurements of microfibril angles and proportions of latewood. Measurements of tracheid length (mean measured length-weighted contour length), tracheid cell wall thickness and tracheid diameter were acquired with a Kajaani FiberLab 3.5 optical fiber dimension analyzer (Metso Automation Inc.). Annual ring widths were measured with a digital caliper (± 0.01 mm). Density was determined by measuring the weight and volume of the samples (± 0.001 g), the latter being determined using the water-displacement method. Heartwood and sapwood were visually distinguished on the basis of the generally darker appearance of heartwood.

In study III

To compare different methods for measuring microfibril angles in terms of their speed and the quality of the results they provide, a wood sample with a cambial age of 20 years taken (at stump height) from a tree in a densely spaced stand (Tables 1 and 2) was divided into two paired specimens: one for cutting sections and one for macerating tracheids. The microfibril angles in the S₂layers of earlywood tracheid cell walls in the sub-samples were then analysed by light microscopy. Methods tested were maceration according to Franklin (1945), cutting thin sections and iodide staining according to Senft & Bendtsen (1985), repeated hydration/dehydration cycles to induce cracks according to Stamm (1964) and use of dry versus wet samples during microscopy. Finally, the utility of an extended analysis based on a method involving maceration of tracheids, iodide staining, repeated hydration/dehydration cycles and keeping samples wet during microscopic examination was assessed. Analyses of both earlywood tracheids and latewood tracheids and measurements of the proportion of latewood were performed to obtain mean microfibril angle values for all samples from the densely and widely spaced trees (Tables 1 and 2).

In study V

Reference variables for the NIR data obtained in this study were tissue type (mature or juvenile wood, threshold cambial age 20 years) and the abovementioned mechanical properties, tracheid cell morphology parameters, proportion of latewood, mean microfibril angle and microfibril angle of latewood and earlywood tracheids. A near infrared spectrometer (Foss NIRSystems 6500, Höganäs, Sweden) with a fibre-optic probe (measurement diameter 4 mm) was used to collect reflectance spectral data. Measurements were performed orthogonally to the radial tangential plane on sub-samples to measure creep. Mean spectra, 780 - 2380 nm (resolution, 2 nm), obtained from 32 scans were collected for each measurement.

Statistics

Linear regression analyses were performed in studies I and IV to determine the correlations between mechanical properties and structural properties, and in study II to explore correlations between structural material properties and other structural properties. Optimization of r² and standard deviation values together with normality of the parameters' distributions were used to select parameters to include in the predictive functions. In study IV, in order to improve the robustness of the functions presented in study I, cross-validation tests were performed and the maximal number of parameters in functions was set to seven (apart from the constant parameter). In addition, in study IV the most appropriate structural parameters, transformations and interaction terms were selected by examining their relationships to the residuals of analysis with the help of graphs and Pearson correlation coefficients. The risk of the correlations between mechanical properties and structural properties being affected by the use of the two sets of samples considered in study IV, conditioned and measured at different times, was diminished by including test set as an indicator parameter during the development of the functions.

In study II, analysis of variance using General Linear Model was carried out to test if there were significant differences in structural characteristics and mechanical properties between trees subjected to different silvicultural regimes. In study III, Tukey's pair-wise comparison tests were performed to test whether there were significant between-method differences in the results obtained. In study V, the correlations between NIR data and the variables of interest were explored by biorthogonal partial least squares regressions. All data were included in the models and the criterion for setting the number of principal components was that each one included should considerably improve the RMSEP value. Two types of validation were applied: cross-validation of regression models and distance to the global model centre, using principal component analysis. Spectral data were mean-centred before biorthogonal modelling. Software used in study I – III was MINITAB 13 (Minitab inc. 2000) and software used in study IV was MINITAB 15 (Minitab inc. 2007). All PLS remodelling was done in Matlab (The Math-works inc.) by using PLS Toolbox (Eigenvector Research; Wise et al. 2003).

Results and Discussion

Material and methods used

Clear wood properties were studied because they are the material properties of interest for the main industries that currently use wood, and they are poorly correlated to the grading classes used for timber and pulpwood payments (see Introduction). Clear wood specimens are also easier to characterize than structural wood since there is less variability in material properties within the pieces. Consequently, in contrast to structural wood, the potential values of material properties are not reduced by weaker parts, e.g., parts with weaker clearwood or the adverse grain angles typically found in knotwood and the surrounding clearwood (Dinwoodie 2000, Xu 2002). Further, homogeneity within pieces is important inter alia to avoid wide variations in dimensional instability due to (for instance) variability in microfibril angles and mixtures of heartwood and sapwood (Dinwoodie loc cit.). In addition, it is important to ensure that batches classified as having a certain quality are as homogenous as possible since customers may prefer wood they purchase to have high or low values of certain properties. Thus, rigorous classification of wood with homogenous characteristics would facilitate fuller exploitation of the potential of wood in both traditional products and applications in which it is currently more rarely used than steel, plastics and other materials (Bowyer 2000). It should also be noted that the local mechanical properties of structural wood could be estimated to a high degree by using clear wood properties and together with three-dimensional measurements of the angles of knots and their volume fractions, or more easily measured knot:stem area ratios, the accuracy increases further (Xu loc cit.). Further, the mechanical properties of a board or an entire log can be calculated from the properties of their different local parts (Xu loc cit.). Thus, analyses of clear wood properties could be used, together with calculations of minimum and maximum values of material properties, to sort logs into optimal quality classes in the forest, before transporting it to appropriate industrial users with high confidence that it will meet their requirements.

The materials used spanned wide ranges of diverse properties (Fig. 2), e.g. an almost nine-fold range in longitudinal stiffness values, which generally



green crown, distance to green crown, height in trees and age of apical meristem. Wood samples were obtained from trees in stands thinned from below or above, in stands grown at wide or dense spacing and from stands either exposed to or protected from wind in a windy area.

promotes robustness in derived functions. In addition, the risk of random covariance between material properties and other properties was further reduced by the great variation in values between stands and within trees. The material examined in study IV was most diverse, especially in terms of combinations of tree structure characteristics and mechanical properties. More samples were also analyzed (except for longitudinal compression strength which was measured with older test equipment for the first set test, which may have given somewhat biased values, hence the values of this parameter obtained for the first set were removed from the analysis in study IV). The differences between test sets were very small for the other mechanical properties, and insignificant. The stiffness values calculated in studies I - III and V were based on an assumed relationship between deformations at the loading points and the middle of the span that yielded somewhat biased values. However, the relative differences between materials (Fig. 4 and Table 3) and the ability of the functions to explain stiffness (Figs. 3 and 5) were affected very little by the bias, since it was almost proportional to the correct value. The small lack of proportionality between the bias and the correct value was shown in study IV to be related to the ratio between hardness at the tangential direction and stiffness, i.e. an effect of local indentations. In addition, the range of differences in the ratios between deformations at the loading points and the middle of the span was only 1.09 to 1.13. The correlations between structural and mechanical properties were consistent with those found in other studies (see below) and higher accuracy was obtained using all the examined characteristics, suggesting that the methods used for determining the structural characteristics and mechanical properties were accurate. In conclusion, the multiparametric linear regression functions are able to distinguish between materials with different qualities, and hence can identify materials with homogenous qualities, to higher degrees than functions obtained in previous studies. Further, the biorthogonal partial least squares regression functions are highly accurate and hence are excellent tools for distinguishing between materials with differing qualities. The reliability of the applied statistic methods is commonly recognized with high robustness to, e.g., that predicting parameters are more or less correlated with each other. However, to validate the reliability of the functions definitively they should be applied in managed forests. In order to sort wood further research is needed to classify wood and transform the derived mean values and their standard deviations to minimum and maximum values. In addition, the volumetric proportions of wood with different qualities within logs have to be modelled to assess their possible financial values.

Structural characteristics were used to predict material properties since information on many such characteristics is readily available, e.g. in stand data files, and thus convenient for distinguishing wood with different material properties while it is still in the forest. Furthermore, these characteristics are also of strong intrinsic interest. For example: the age of the apical meristem and cambium are equal to the duration of the tree's and wood's development, respectively; annual ring width is strongly correlated to volume growth; heights in trees strongly reflect variations in multiple parameters within trees; and other characteristics like density and microfibril angle are material properties of considerable industrial importance. Near infrared spectroscopy was used to predict material properties since it has been applied online in many other industries (Osborne et al. 1993), and has proven capacity to predict material properties accurately (Axrup et al. 2000, Gindl et al. 2001, Hauksson et al. 2001, Hodge & Woodbridge 2004, Hoffmeyer & Pederson 1995, Jones et al. 2005, Kelly et al. 2004, Marklund et al. 1999, Schimleck et al. 2001a, 2001b, 2002, Shultz & Burns 1990, Thumm & Meder 2001, Wright et al. 1990), even for materials with high water contents (Thygesen & Lundqvist 2000).

Study I

The ability to predict every mechanical property generally increased with increasing wood structure resolution (Fig. 3). Multiparametric equations, based on data related to several structural characteristics, were used to quantify the possible levels of accuracy when making predictions at three different resolution levels (giving end-users the opportunity to select the most appropriate level of prediction and cost). The third resolution level, which would generally be most appropriate, combining information on all characteristics, gave high r^2 values in models to predict longitudinal stiffness (~0.93), Brinell hardness at tangential direction (~0.82) and longitudinal creep (~0.83). Values of r^2 for the second resolution level, including all parameters except microfibril angle and proportion of latewood, were ~0.91, ~0.88 and ~0.89 for longitudinal bending strength, Brinell hardness and compression strength, respectively.

Comparisons between the simple equations with those obtained in other studies on the effects of single structural characteristics on mechanical properties showed strong similarities (Bao et al 2001, Bendtsen & Senft 1986, El-Hosseiny & Page 1975, Groom et al. 2002, Holmberg 2000, Kliger et al. 1995, Mencuccini et al. 1997, Persson 2000, Raymond et al. 2004, Yang & Fortin 2001). However, since more heterogenic material was studied here than in the cited studies, the r² values of the density-based relationships were lower, even though the 100 samples used for determining each mechanical property were partly nested to only eight sampled trees. The main reason for the relatively low r² values is that samples representing different apical meristem ages, cambial ages and wood tissue types were taken from trees with major differences in green crown properties and annual ring width patterns (study II,



Figure 3. The relationships between the observed and calculated longitudinal modulus of elasticity (MOE) at the first-, (upper left) second- and third- (lower left) resolution levels. (R-Sq; 0.82, 0.91 & 0.93). HW, Heartwood; SW, sapwood; S, stump; T, intermediate vertical position in the trees

Amarasekara & Denne 2002, Brüchert et al. 2000, Groom et al. loc cit., Kliger et al. loc cit., Lindström 1996, Macdonald & Hubert 2002, Mencuccini et al. loc cit., Olesen 1982, Pape 1999, Zhu et al. 2007). However, the functions generated when all parameters were included, for the mechanical properties studied, had high r^2 values (~ 0.9); considerably higher than those obtained in previous work. Consequently, the new models presented could be generally applicable for selecting wood with homogenous qualities from heterogeneous sources.

Study II

Wood samples from stands subjected to two contrasting silvicultural regimes (wide spacing and dense spacing, designated PWR and SDR, respectively) differed strongly (Fig. 4), both in structural characteristics (with up to fivefold differences in means for all samples) and mechanical properties (with up to almost three-fold differences in means for all samples). Structural characteristics could be ranked, in terms of their proportional differences (from high to low) between SDR and PWR, in the following order: late wood proportion, distance to green crown, annual ring width, proportion of green crown, microfibril angle, tracheid length, density, tracheid cell wall thickness and tracheid diameter. In addition, characteristics related to cambial age matured more rapidly in SDR than in PWR, and differences between them were weaker at intermediate top height than at stump height. Based on means for all samples, SDR wood had ~150% higher longitudinal stiffness, ~70% higher longitudinal bending strength, ~50% higher longitudinal compression strength, ~30% higher Brinell hardness at tangential and longitudinal directions, and ~10% higher relative $MOE_{t. 1000h}$ (longitudinal creep) than PWR wood. The rankings appear to be related to the proportions of juvenile wood associated with the two regimes, and the higher sensitivity of stiffness and strength properties to maturation of wood characteristics than hardness and creep (Bendtsen & Senft 1986, Dinwoodie 2000, El-Hosseiny & Page 1975, Holmberg 2000, Persson 2000). Another effect of sensitivity to juvenility is that for stiffness and strength properties the rankings for the magnitude of differences between the regimes are similar in the different sub-sample groups, as are the structural characteristics. For hardness properties the rankings were almost the same, but the differences between the regimes were greater in heartwood at intermediate height than at stump height. The largest observed differences in ranking and magnitude of differences between sub-groups of samples were related to creep, for which the differences were greatest in sapwood at stump height, then heartwood at stump and intermediate heights, and lowest in sapwood at intermediate height. The final observed effect of differences in sensitivity to juvenility is that the number of wood characteristics that make major contributions to differences between silvicultural regimes or sub-groups was highest for stiffness, followed by strength properties, then hardness properties and lowest for creep.

Consequently, there is high potential to use silvicultural regimes to control wood structural characteristics and mechanical properties. Differences in green crown parameters and annual ring width between the regimes had high ability to explain the differences in wood structural characteristics, in accordance with Amorasekara & Denne (2002), Groom et al. (2002), Lindström (1996) and Macdonald & Hubert (2002). Furthermore, together with cambial age and apical meristem age, green crown parameters and annual ring width were found to correlate to high degrees, both together and separately, to characteristics of wood samples from the two tested regimes at different ages of the rotation period.

Study III

The method involving analysis of macerated tracheids, following pre-treatment with hydration/dehydration cycles, staining with iodide and keeping the samples wet during microscopic observation was the most useful of the tested



Figure 4. Difference in longitudinal stiffness (MOE) between seeding – dense regime and planting – wide spacing regime (from Eriksson et al. 2006b) in relation to Density; in heartwood (–HW), sapwood (–SW) and in combination with stump (S—) and intermediate top (T—) vertical position in the trees.

methods. Using this method sample preparation and analyses were easier to perform, and thus cheaper, than when using the method developed by Senft & Bendtsen (1985), involving analysis of cut iodide-stained sections. The measured microfibril angles obtained by the proposed method were almost identical to those obtained by the reference (Senft & Bendtsen loc cit.) method. The results are also consistent with other studies, since Fujisawa & Hirakawa (1995) reported that analyses of macerated tracheids and cut sections give equal microfibril angles, and several authors have reported that measured values are not significantly affected by either hydration/dehydration cycles or iodide staining (Senft & Bendtsen loc cit., Stamm 1964). Nearly two-fold differences in microfibril angle were observed, using the proposed method, between earlywood and latewood tracheids of all tested cambial ages, which is also consistent with previous reports (e.g., Herman et al. 1999). Furthermore, up to 3-, 2.6-, and 3.1-fold differences were found using the method between wood grown under different silvicultural regimes, between wood with different apical meristem ages and between wood with different cambial ages, in accordance with study II and results presented by Bruchert (2000); Groom et al. (2002); Lindström (1996); Mencuccini et al. (1997); Olesen (1982). Thus, the method is an excellent tool for defining wood and its properties.

Study IV

The ability to predict the mechanical properties generally increased with successive increases in the three examined resolution levels of determination of structural properties (Fig. 5), with r^2 values often reaching 0.9 at the highest resolution, in accordance with results obtained in Study 1. However, the functions obtained were more robust than those obtained in the previous study, due to the greater ranges and combinations of non-material property values (and, hence, material property values) spanned by the examined samples according to study II, Amarasekara & Denne (2002), Brüchert et al. (2000), Groom et al. (2002), Kliger et al. (1995), Lindström (1996), Macdonald & Hubert (2002), Mencuccini et al. (1997), Olesen (1982), Pape (1999) and Zhu et al. (2007). The range of values differed among mechanical properties from almost nine-fold in stiffness, followed by hardness and strength properties and lowest for creep (with an almost two-fold span), and these variations could to some part be explained by the differences in the strengths of the correlations of the mechanical properties to structural characteristics, as manifested in the accuracy of the functions at the three resolution levels (study I, study II, Bendtsen & Senft 1986, Dinwoodie 2000, El-Hosseiny & Page 1975, Holmberg 2000, Persson 2000). Inclusion of structural non-material properties improved the accuracy of the functions, especially at a low resolution level, which may be due to the particularly strong correlations between those properties and both latewood proportion and microfibril angle (Study II). The functions combing all characteristics at the first resolution level provide cheap tools for describing properties of wood at high accuracy while it is still in the forest.



Figure 5. Prediction accuracy of functions at the three resolution levels of determination of structural properties predicting stiffness (MOE), compression strength ($f_{\rm c}$), bending strength ($f_{\rm m}$), Brinell hardness ($H_{\rm B,0}$) and creep all at longitudinal direction and Brinell hardness at tangential direction ($H_{\rm B,00}$).

Study V

Attempts to relate near infrared reflectance spectra to the measured parameters by biorthogonal partial regression modelling were successful, and indicated that the parameters could be divided into several groups in terms of the strength of their correlations with the NIR data. The longitudinal modulus of elasticity showed the highest correlation to the NIR spectra (r>0.9), followed by bending strength at longitudinal direction, compression strength at longitudinal direction and tracheid length (0.8<r<0.9). The next group had correlations in the range of 0.7-0.8, topped by microfibril angle (mean and earlywood tracheids and latewood tracheids separately) followed by Brinell hardiness at longitudinal direction, proportion of late wood and longitudinal creep. The weakest correlations were found for Brinell hardiness at tangential direction, tracheid diameter and tracheid cell wall thickness (0.4 < r < 0.7). The strong ability of near infrared spectroscopy to predict the material properties of the samples examined is consistent with findings of previous studies (Axrup et al. 2000, Gindl et al. 2001, Hauksson et al. 2001, Hodge & Woodbridge 2004, Hoffmeyer & Pederson 1995, Jones et al. 2005, Kelly et al. 2004, Marklund et al. 1999, Schimleck et al. 2001a, 2001b, 2002, Shultz & Burns 1990, Thumm & Meder 2001, Wright et al. 1990). The results also showed that juvenile (cambial age ≤ 20 years) and mature wood can be distinguished using NIR, supporting the findings by Via et al. (2005). The findings should facilitate attempts to improve the documentation of structural, chemical and mechanical properties of wood, in situ, as it progresses through the forest-wood chain.

Material characterization models - Applications

As can be seen in Figure 2, forests have great potential to supply materials with very wide ranges of properties. However, the visual grading methods generally applied poorly distinguish between different materials and, consequently, heterogeneity is currently a major problem (see *Introduction*). Using structural characteristics (studies I – IV) and/or NIR spectroscopy (study V) it is now possible to predict several material properties of clear wood at high accuracy. Thus, there are great possibilities to characterize wood while it is still in the forest since the functions are reliable and contain parameters which are either forest-related or could be measured on line (see *Material and methods used*). Further, homogenous materials in terms of properties of interest for both traditional and other products can be distinguished (see *Material and methods used*). Thus, the developed functions can be used to create practical methods for scientifically characterizing wood, similar to those used for other materials (e.g. the property table from Eriksson (2005) shown in Table 3), while it is still in the forest.

Modelling material properties of different sized wood specimens can be used as a basis for industrial applications of the functions (see Material and methods used). The functions predict material properties of individual annual rings, which set the size of the smallest described unit within analyzed specimens, and thus the smallest size of material of specific classes/qualities. Thus, there are great possibilities to model the qualities of material of different sizes to fulfil different requirements of specific users. Ideally, a product should contain material in which every small piece has the same quality (see Material and methods) but this would increase detection, modelling and processing costs. However, since the functions predict many material properties of interest for different users the concept of a property table (Table 3) proposed by Eriksson (2005) to define a quality could be applied. After certain qualities have been defined wood could be classified using the functions after transformation of the derived mean values and their deviations to minimum and maximum values. The volumetric proportions of material with different qualities within logs could then be modelled and logs could be sorted accordingly.

In the pulp and paper industry sorting is based on grouping material according to its sources, e.g. sawmill chips, pulpwood, species, large/small logs, wood from thinning and clearcutting. Thus, it should be possible to make improvements by reducing the heterogeneity within the material groups. Improvements in sorting wood's properties prior to delivery to industrial customers should also be possible in the sawmill industry, since structural characteristics can, for instance, explain variations in dimensional instability within specimens (see Material and methods used). Further, Xu (2002) has shown that using clear wood properties to model mechanical properties of different-sized structural wood specimens has great potential (see Material and *methods used*). In addition, products with finger-jointed clearwood pieces are now commonly produced in the sawmill industry. However, measurements of knots and other defects to improve estimates of mechanical properties should preferably be done in sawmills, since the detection and cutting to pieces can be done simultaneously. Promising online measurement methods, such as dual-source 2D X-ray scanning, laser scanning and NIR spectroscopy have become established in sawmills to improve roundwood pre-sorting prior to production, but these techniques are currently only used to obtain accurate automated measurements of logs' external features, e.g. curvature and taper. Additionally, in the future online measurements could be capable of predicting material properties of processed and modified wood (Study V, Osborne et al. 1993). Thus, if wood was described (and sorted) according to Eriksson's (2005) concept of a property table (Table 3) during its progress through the forest-wood chain it could be matched, according to its specific properties, to appropriate products, and thus increase the income for all parts.

Table 3. Table of woods' properties (selected part of the table in Eriksson 2005), showing mean values of mature, average density, clear-free Pinus sylvestris heartwood (HW) from various studies and two compared examples of wood with different material qualities from Paper II (SWSSDR and SWSPWR). Wood with a moisture content of 12% (14% in the two compared examples of material qualities) was tested, in the longitudinal direction, at 20°C if not otherwise specified. Note: – (data missing); Ref (cited studies).

Property	Material			Ref.
	SWSSDR	SWSPWR	HW	
Standard				
Description Delivery Form Additives Special Characteristics	Sapwood from stump height in seeding dense spacing regime Solid wood None	Sapwood from stump height in planting wide spacing regime Solid wood None	Mature heartwood from average density <i>Pinus sylvestris</i> Solid wood None High resistance to decay by biota	
Structural characteristics				
Proportion of green crown	0.31	0.75		
Distance to green crown (m)	10	3.2		
Age of apical meristem (Yr)	15	10		
Cambial age (Yr)	32	34		
Annual ring width (mm)	0.78	4.2		
Density (Kg/m ³)	600	480	350 - 600	11
Percentage latewood (%)	26	4.8	10 - 40	40
Fibre length (mm)	2.2	1.7	2.4	32
Fibre width (µm)	32	28	35	32
Cell wall thickness (µm)	4.6	4.1	3 - 5	40
Microfibril angle (°)	9.8	27	9	18
Mechanical properties Modulus of elasticity, E _L (MPa)	11000	3300	8000	31
General difference $E_{_{\rm T}}$ / $E_{_{\rm L}}$			0.035	21
General difference E_{R} / E_{T}			1.9	21
Moist. content = 6%,			8700	32, 17
Moist. content = 20%			7000	32, 17
$1h (E_{1h}/E_0)$	0.97	0.89	0.97	11
$10h (E_{10h}/E_0)$	0.93	0.88	0.93	11
$100h (E_{100h}/E_0)$	0.88	0.78	0.87	11
$1000h (E_{1000h}/E_0)$	0.77	0.64	0.75	11
<u>E, 20 °C, moist. cont. = 18 %</u>			7200	32,17
General difference $E_{_{\rm T}}$ / $E_{_{\rm L}}$			0.032	17
E _{1h, 10h, 100h & 1000h}			-	-
Bending strength (MPa)	110	58	78.2	37
Hardness, Brinell (N/mm ²)	53	29	48	22
Hardness, Brinell Tang. ——	19	12	18	22
Compression strength (MPa)	43	25	47	27
Moist. content = 6%			55	27
Moist. content = 20%			40	27

Basically, such a table would provide detailed data, obtained from standard tests, specifying minimum values of a large number of the material's properties, similar to the norms and datasheets used to describe the specifications of steels and plastics (EN 10025 1993, M-Base Engineering 2003). Thus, the material would be warranted and customers could rely on wood with similar confidence to steel products, plastics and other products for which abundant information is available (Bowyer 2000). This is essential in many applications. For instance, engineers require knowledge of the properties of materials used in various constructions, e.g. if the material has to withstand certain compressive or bending forces one time (strength) (Dinwoodie 2000). Strength parameters for repeated loading can be calculated if the material's resistance to deformation (modulus of elasticity/stiffness) and the critical deformation at which structural damage starts to occur (the elastic limit) are known (Dinwoodie loc cit.). Since wood is a viscoelastic material, it creeps (i.e. deforms to a degree related to the duration of loading) and thus it is important to ensure that its elastic limit is not exceeded (Dinwoodie loc cit.). In addition, homogeneity is important since, for instance high values of hardness may be undesirable during processing, but essential in products that must be resistant to bucking/local indentations, e.g., floorboards. Consequently, it is important to have information regarding a wide range of parameters, and to sort the material accordingly, to meet the needs of many different customers. Finally, the concept of property tables would also promote the development of new wood-based products and materials with highly-specified properties, which could greatly increase the economic value of wood.

Starting in the forest, thorough description of the materials' properties would allow the forest owner to be paid more according to the quality of the wood produced, since appropriate industrial customers would purchase it, and be able to sort wood of the desired quality from other wood. In addition, more information on the effects of different silvicultural treatments would help owners to choose appropriate treatments with respect to trade-offs between the expected value of the wood produced and costs. For instance, in study II the densely-spaced trees had much higher values, for many applications, than the widely spaced trees, due to the effects of the silvicultural practices applied at various phases of their development.

Prediction of material properties in stands is likely to be especially important in early (pre-trade) phases, e.g. before thinning and clear cutting. The results presented in study II suggest that, in addition to major differences in mean values of the parameters, there were also variations in the material properties in both vertical and radial directions, and between stands, due to differences in growth conditions resulting from the different silvicultural regimes. Variations in the material properties between trees within stands can be recognized earlier (Lindström 1996, Macdonald & Hubert 2002, Mattsson 2002). Consequently, prediction of the properties of material in stands requires prediction of the variation within trees, among trees and between stands.

Functions linking mechanical properties (study IV) and structural properties (study II) based on crown characteristics, height in the tree, age of apical meristem, wood tissue type, cambial age, annual ring width and density are useful for predicting material and other properties. In the future it may be possible to automate near infrared spectroscopic analyses (study V) using drillholes, or drillcores, in the forest, which should provide even more accurate estimates of both material and other properties. Cambial age and annual ring are expected to be estimated by NIR spectroscopy in the near future since NIR data can detect variations in microfibril angles (study V), and there is a sharp boundary between latewood and earlywood (and their associated microfibril angles) between two annual rings (Senft & Bendtsen 1985). Near infrared spectroscopy is also useful for estimating density (Schimleck et al. 2001b, 2002) and chemical parameters of the wood, and thus probably heartwood formation (Shulz & Burns 1990). In order to estimate mechanical properties using structural properties, without having performed NIR spectroscopy at the specific height of a certain tree, the distribution of the width of annual rings has to be predicted using the abovementioned non-material properties. The width of annual rings can then be corrected by taking the ratio between the predicted diameter based on the sum of annual ring widths and predicted (see below) or measured stem diameters at different heights. The density can then be predicted from the annual ring width and other non-material properties. In addition, if NIR spectroscopy is not performed at breast height, the cambial age and distance to pith at the boundary between sapwood and heartwood has to be estimated using factors such as growth rate, needle biomass, growth conditions, site index and position of the stem (cf. Albrektson 1984, Bamber 1976). If NIR spectroscopy is performed the boundary can be assumed to be at the same number of annual rings from the bark as measured at breast height. Finally, material properties at heights other than breast height can be predicted more accurately since the NIR-predicted values can be used following correction based on the relative difference between the heights predicted using non-material properties at both breast height and other heights.

A flow scheme for quantifying material properties of wood specimens in the forest is shown in Figure 6. The first step is to determine the requirements of the analysis, and the second is to consider if sufficient data are already available for modelling the material qualities of interest. An inventory of the stand may be needed, in which case sample plots will have to be positioned to describe the variation in different parts of the stand. In order to obtain representative sample trees of the plots, trees should be selected from each tree rank class, either determined by measuring the distribution of stem diameters or by applying appropriate assumptions. The first decision to make regards the heights at which predictions should be made for the sample trees, then predictions are made for properties in "cross sections" at the selected heights, based on data collected for each annual ring, and finally predictions for points between measuring heights are made by interpolation. A suggestion is to choose stump height, breast height and at least one more height further up in the tree. Stem diameter and annual ring width have to be estimated to determine the area of cross-sections and annual rings in order to predict distributions of different material properties. The distance to pith, annual ring width, wood tissue type and material properties are predicted for each annual ring in each examined cross-section by NIR spectroscopy (only at breast height) or functions based on structural properties (see above). Each of the sample trees' proportion of green crown, distance to green crown, height in tree, and stem diameter at both breast height and stump height are measured. Stem diameters at other heights can be predicted from the measured diameters and taper functions. The age of the apical meristem is calculated by subtracting the number of annual rings in the cross-section from the tree's age, which is easily calculated in even-aged stands from the year of establishment. The number of annual rings in the cross-section at breast height is predicted if NIR spectroscopy is performed, which can be useful when calculating the numbers of annual rings at other heights. Other indications of age can be gained by counting the number of branch whorls from the top to the specific height in the tree, or using functions describing normal height development for the specific site index (Hägglund & Lundmark 1982). All parameters needed in modelling material properties (see above) have then been collected. Dissimilarities between different parts of the stand should be accounted for by dividing it into appropriate sections.



Figure 6. Flow path for the quantification of material properties of wood in the forest.

The numbers of sample plots, rank classes, sample trees and the sizes of sample plots that should be used depend on the required accuracy of prediction, heterogeneity of the stand and the amount of information already available. In addition, the use of NIR spectroscopy in drillholes/on drillcores increases the accuracy and should decrease the costs of the analyses. Reductions in accuracy will be associated with deviations from the average tree, to varying degrees for different functions, but such deviations should be of minor importance provided sufficient trees are sampled within a stand and/or tree rank class. The proposed data collection methods and functions provide estimates of material properties of individual annual rings, and thus great scope for users to choose the resolution of the resulting modelling of many different properties. One example is to divide the material into either juvenile or mature wood using, for instance, a threshold microfibril angle in annual rings as a criterion (study II), and this alone may account for substantial proportions of the variations among logs within a tree, among trees within a stand and among stands. Compared to modelling at the resolution of individual annual rings the variation of properties within juvenile and mature wood is not explained, but the prediction is more accurate since the volumetric proportion of juvenile wood within a log is negligibly affected by errors (within a few annual rings) in predictions of the location of its border with mature wood. Combining data on variations in properties of interest within a log is also possible, e.g., bending strength in classes within mature wood, or a combination of several material properties according to a property table (Eriksson 2005).

Conclusions and need for further research

Silvicultural regimes clearly have great potential to influence the material properties of wood, in terms not only of average levels of the properties, but also trends in vertical and radial directions within the trees. They also strongly affect growth-related, non-material properties like crown characteristics and annual ring width, which together with cambial age and apical meristem are correlated to great degrees to structural both material- and non material-properties. Thus, functions using such readily available parameters provide valuable tools for selecting materials.

Functions for predicting mechanical properties from structural properties were developed at three resolution levels, and the ability to predict the mechanical properties generally increased with successive increases in these resolution levels of determination of structural properties, with r^2 values often reaching 0.9 at the highest resolution. The samples used to develop the functions represented most of the variation found in Scandinavian forests, spanning both large ranges and combinations of different property values, this diminishing the risk of random covariance between material properties and non-material properties. Addition of structural non-material properties improved the functions, especially at a low resolution level, providing cheap tools for describing mechanical properties of wood at high accuracy while it was still in the forest.

In addition, a method was developed for measuring microfibril angles that yielded values that were almost identical to those obtained using a commonly applied method, but was easier to perform and thus cheaper. The reliability of the method was further demonstrated by its ability to detect differences in fibril angle between samples with different types of fibre, samples from trees grown under different silvicultural regimes and samples differing in apical meristem and cambial ages. Thus, the method is an excellent tool for defining wood and its properties.

Clearly biorthogonal partial least square regression modelling of near infrared (NIR) reflectance spectra is capable of providing highly accurate predictions of both mechanical and structural properties. Thus, NIR spectroscopy offers a combination of high speed, accuracy, robustness of equipment and low sensitivity to environmental disturbances, making it ideal tool for use *in situ*, e.g., in the forest.

A practical approach for characterising wood is proposed, using functions linking mechanical properties and structural properties, based on characteristics of strong intrinsic interest that are readily available, e.g. in stand data files and possibly, in the near future, automated near infrared spectroscopic analyses using drillholes, or drillcores, in the forest. The possibilities of modelling material properties of individual annual rings could be used to define qualities tailored for specific customers, and to sort logs in the forest accordingly. For example, mature wood within logs could be classified according to its bending strength (and thus financial value), or possibly according to a combination of several material properties using a property table (Eriksson 2005). Hence, wood could be matched, according to its specific properties, to appropriate products, and thus increase the prices fetched for wood at all stages throughout the forest-wood chain.

Further applications of NIR-spectroscopy could include use of structural non-material properties of strong intrinsic interest in modelling NIR-spectral data and analysis in real time in the forest and at industrial sites. Further, minimum and maximum vales of designed qualities should be specified from mean values and their deviations obtained using predictive functions. Finally, property tables should be compiled for processed and modified wood to provide guarantees regarding wood's material properties at delivery for users later in the forest-wood chain.

Acknowledgements

First I would like to thank my supervisors Urban Bergsten and Henrik Lindberg for guidance and support. I would also like to thank personnel at Swedish University of Agricultural Sciences, Svartberget Research Station for great services. In addition, I am thankful to Sören Holm for guidance in statistics, and to Sees-editing Ltd (www.sees-editing.co.uk) for help with the written English. Great thanks are also due to Per Hansson and Iwan Wästerlund at the Swedish University of Agricultural Sciences for lending equipments. Financial support was received from the Kempe Foundations.

References

- Albrektson, A. (1984) Sapwood basal area and needle mass of Scots Pine (*Pinus sylvestris* L.) trees in central Sweden. *Forestry* 57(1), 35–43.
- Amorasekara, H. & Denne, M. P. (2002) Effects of crown size on wood characteristics of Corsican pine in relation to definitions of juvenile wood, crown formed wood and core wood. *Forestry* 75(1), 51–61.
- Ashby, M. F. (2000) *Materials selection in mechanical design*. Butterworth, Heinemann, Oxford. ISBN 0750643579. 502 pp.
- Axrup, L., Markides, K. & Nilsson, T. (2000) Using miniature diode array NIR spectrometers for analysing wood chips and bark samples in motion. *Journal of Chemometrics* 14(5-6), 561-572.
- Bamber, R.K. (1976) Heartwood, its function and formation. Wood Science and Technology 10, 1-8.
- Bao, F.C., Jiang, Z.H., Jiang, X.M., Lu, X.X., Luo, X.Q. & Zhang, S.Y. (2001) Differences in wood properties between juvenile and mature wood in 10 species grown in China. Wood Sci Technol 35, 363–375.
- Bendtsen, B.A. & Senft, J. (1986) Mechanical and anatomical properties in individual growth rings of plantation-grown eastern cotton-wood and loblolly pine. *Wood Fiber Sci.* 18(1), 23–38.
- Bowyer, J.L. (2000) Wood science in a changing world Where are we headed? *Wood Sci Technol* 34, 175–181.
- Brüchert, F., Becker, G. & Speck, T. (2000) The mechanics of Norway spruce (*Picea abies* (L.) Karst): mechanical properties of standing trees from different thinning regimes. *Forest Ecology* and Management 135, 45–62.
- Cameron, A.D. & Dunham, R.A. (1999) Strength properties of wind- and snow- damaged stems of *Picea sitchensis* and *Pinus sylvestris* in comparison with undamaged trees. *Can. J. For. Res.* 29, 595–599.
- Dinwoodie, J. M. (2000) Timber: Its nature and behaviour. Second edition. E&FN SPON. ISBN 0 419 23580 9.
- El-Hosseiny, F. & Page, D.H. (1975) The mechanical properties of single wood pulp fibres: Theories of strength. *Fibre Sci Technol.* 8, 21–31.
- EN., 408. (1995) Timber structures Structural timber and glued laminated timber Determination of some physical and mechanical properties. 19 pp.
- EN., 10025. (1993) Hot rolled products of non-alloy structural steels technical deliver conditions including amendment AI. 26 pp.
- Eriksson, D. (2005) Scots pine wood: an anatomical structure in the tree and an engineering material in industry. Licentiate Thesis. Umeå: Swedish University of agricultural Sciences. ISBN 91– 576-6853-1.

- Gindl, W., Teischinger, A., Schwanninger, M. & Hinterstoisser, B. (2001) The relationship between near infrared spectra of radial wood surfaces and wood mechanical properties. *Journal of Near Infrared Spectroscopy* 9(4), 255–261.
- Groom, L., Shaler, S. & Mott, L. (2002) Mechanical properties of individual southern pine fibers. Part III: Global relationships between fiber properties and fiber location within an individual tree. *Wood and Fiber Science* 34(2), 238–250.
- Franklin, G.L. (1945) Preparation of thin sections of synthetic resins and wood resin composites and a new macerating method for wood. *Nature* 155, 1-51.
- Fujisawa, Y. & Hirakawa, Y. (1995) The relationships between microfibril angles of the S-2 layer and latewood tracheid lengths in elite Sugi tree (*Cryptomeria japonica*) clones. *Mokuzai Gakkaishi* 41(2), 123-131.
- Hauksson, J.B., Bergqvist, G., Bergsten, U., Sjöström, M. & Edlund, U. (2001) Prediction of basic wood properties for Norway spruce. Interpretation of Near Infrared Spectroscopy data using partial least squares regression. *Wood Science and Technology* 35(6), 475–485.
- Herman, M., Dutilleui, P. & Avella-Shaw, T. (1999) Growth rate effects on intra-ring and interring trajectories of microfibril angle in Norway spruce (*Picea abies*). IAWA Journal 20, 3-21.

Hodge, G.R. & Woodbridge, W.C. (2004) Use of near infrared spectroscopy to predict lignin content in tropical and sub-tropical pines. *Journal of Near Infrared Spectroscopy* 12, 381–390.

Hoffmeyer, P. & Pederson, J. (1995) Evaluation of density and strength of Norway spruce wood by near infrared reflectance spectroscopy. *Holz Roh Werkst.* 53, 165-170.

Holmberg, H. (2000) Influence of grain angle on Brinnell hardness of Scots pine (*Pinus sylvestris* L.). *Holz als Roh- und Werkstoff* 58, 91–95.

- Hägglund, B. & Lundmark, J.E. (1982) Handledning i bonitering med Skogshögskolans boniteringssystem, del 2. Skogsstyrelsen. 70 pp.
- Jones, P.D., Schimleck, L.R., Peter, G.F., Daniels, R.F. & Clark, A. (2005) Nondestructive estimation of *Pinus taeda* L. wood properties for samples from a wide range of sites in Georgia. *Canadian Journal of Forest Research* 35, 85–92.
- Kelly, S.S., Rials, T.G., Snell, R., Groom, L.H. & Sluiter, A. (2004) Use near infrared spectroscopy to measure the chemical and mechanical properties of solid wood. *Wood Sci Technol* 38, 257-276.
- Kliger, I.R., Perstorper, M., Johansson, G. & Pellicane, P.J. (1995) Quality of timber products from Norway spruce. Part 3. Influence of spatial position and growth characteristics on bending stiffness and strength. *Wood Sci Technol* 29(6), 397–410.
- Lindström, H. (1996) Basic density in Norway spruce. Part III. Development from pith outwards. *Wood and fiber Science* 28(4), 391–405.
- Macdonald, E. & Hubert, J.A (2002) Review of the effects of silviculture on timber quality of Sitka spruce. *Forestry* 75(2), 107-138.
- Marklund, A., Paper, M., Hauksson, J.B. & Sjöström, M. (1999) Prediction of strength parameters for softwood kraft pulps. Multivariate data analysis based on orthogonal signal correction and near infrared spectroscopy. *Nordic Pulp & Paper Research Journal* 14(2), 140-148.
- Mattsson, S. (2002) Effects of Site Preparation on Stem Growth and Clear Wood Properties in Boreal Pinus sylvestris and Pinus contorta. Diss. Umeå: Swedish University of Agricultural Sciences.
 M-Base Engineering, 2003-06-01. http://www.m-base.de/com/demo.html
- Mencuccini, M., Grace, J. & Fioravanti, M. (1997) Biomechanical and hydraulic determinants of tree structure in Scots pine: anatomical characteristics. *Tree Physiology* 17, 105–113.

Minitab Inc. (2000) *Minitab user's guide 2: Data analysis and quality tools: Release 13 for Windows.* 58 pp.

Minitab Inc. (2007) Meet Minitab 15 for Windows. 142 pp.

- Olesen, P.O. (1982) The effect of cyclophysis on tracheid width and basic density in Norway spruce. Diss. Den kgl. Veterinaer- og LandbohØjsk.
- Osborne, B.G., Fearn T. & Hindle, P.H. (1993) Practical NIR spectroscopy with applications in food and beverage analysis. 2nd ed. Longman Scientific & Technical, Harlow, Essex, England.
- Pape, R. (1999) Effects of thinning regime on the wood properties and stem quality of *Picea* abies. Scandinavian Journal of Forest Research 14(1), 38-50.
- Persson, K. (2000) Micromechanical modelling of wood and fibre properties. Diss. Lund University.
- Raymond, C.A., Dickson, R., Rowell, D., Blakemore, P., Clark, N., Williams, M., Freischmidt, G. & Joe, B. (2004) *Wood and fibre properties of dryland conifers*. RIRDC Publication 99(4).
- Saarman, E. (1992) Träkunskap. Sveriges skogsindustriförbund, Markaryd Sweden.
- Schimleck, L.R., Evans, R. & Ilic, J. (2001a) Estimation of *Eucalyptus delegatensis* wood properties by near infrared spectroscopy. *Canadian Journal of Forest Research* 31, 1671-1675.
- Schimleck, L.R., Evans, R. & Ilic, J. (2001b) Application of near infrared spectroscopy to a diverse range of species demonstrating wide density and stiffness variation. *IAWA Journal* 22(4), 415-429.
- Schimleck, L.R., Evans, R. & Matheson, A.C. (2002) Estimation of Pinus radiata D. Don clear wood properties by near-infrared spectroscopy. *Journal of Wood Science* 48(2), 132-137.
- Senft, J.F. & Bendtsen, B.A. (1985) Measuring microfibrillar angles using light microscopy. Wood and Fiber Science 17(4), 564–567.
- Shultz, T.P. & Burns, D.A. (1990) Rapid secondary analysis of lignocellulose: comparison of near infrared (NIR) and fourier transform infrared (FTIR). *TAPPI J.* 73, 209–212.
- Stamm, A.J. (1964) Wood and cellulose science. The Ronald Press Company, New York. 549p.
- Sjöström, E. & Alén, R. (1999) Analytical Methods in Wood Chemistry, Pulping and Papermaking. Springer – Verlag, Berlin, Heidelberg.
- Thumm, A. & Meder, R. (2001) Stiffness prediction of radiata pine clearwood test pieces using near infrared spectroscopy. *Journal of Near Infrared Spectroscopy* 9(2), 117-122.
- Thygesen, L.G. & Lundqvist, S.O. (2000) NIR measurement of moisture content in wood under unstable temperature conditions. Part 1. Thermal effects in near infrared spectra of wood. *Journal of Near Infrared Spectroscopy* 8(3), 183–189.
- Via, B.K., Shupe, T.F., Stine, M., So, C.L. & Groom, L.H. (2005) Tracheid length prediction in Pinus palustris by means of near infrared spectroscopy: the influence of age. *Holz als Rohund Werkstoff* 63, 231–236.
- Wise B.M., Gallagher, N.B., Bro, R. & Shaver, J.M. (2003) *PLS_Toolbox 3.0*. Eigenvector Research Inc., Manson, WA, USA.
- VMR. (2008). Mätning av Barrsågtimmer. VMR, Sundsvall, Sweden. 4 pp.
- Wright, J.A., Birkett, M.D. & Gambino, M.J.T. (1990) Prediction of pulp yield and cellulose content from wood samples using near-infrared reflectance spectroscopy. *TAPPI J.* 73, 164–166.
- Xu, P. (2002) Estimating the influence of knots on the local longitudinal stiffness in radiata pine structural timber. *Wood science and technology* 36(6), 501–509.
- Xu, P., Donaldson, L., Walker, J., Evans, R. & Downes, G. (2004) Effects of density and microfibril orientation on the vertical variation of low-stiffness wood in radiate pine butt logs. *Holzforshung* 58, 673–677.

- Yang, J.L., Fortin, Y. (2001) Evaluating strength properties of *Pinus radiata* from ultrasonic measurements on increment cores. *Holzforschung* 55(6), 606–610.
- Zhu, J.Y., Scott, C,T., Scallon, K.L. & Myers, G.C. (2007) Effects of plantation density on wood density and anatomical properties of red pine (*Pinus resinosa Ait.*). *Wood Fiber Sci* 39(3), 502-512.

The overall objective of the studies this thesis is based upon was to develop a scientifically sound method for characterizing the material properties of wood, analogous to those used to specify the properties of other materials, such as steels and plastics.

Daniel Eriksson, the author of this thesis, received his graduate education at the Department of Forest Ecology and Management, SLU, Umeå. His undergraduate degree is from SLU, Umeå.

ISSN 1652-6880 ISBN 978-91-86195-06-9