# Discrimination of wood origin with near-infrared spectroscopy

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#### Introduction

Spruce (*Picea abies* L. Karst.) is one of the most important woods in Europe, widely used for constructive purposes and paper manufacture as well. There are three main regions of spruce occurrence in Europe (Figure 1).

However according to recent forest inventories; the actual distribution of *Picea abies* is much wider than its natural range.<sup>1</sup>

Many wood properties such as wood density, yearly ring width, chemical composition and other physical properties, depend strongly on environmental, genetic and tree-growth related factors. It has always been considered a difficult task to identify the origin of timber and no effective tools are presently available for this purpose. The goal of this study was to verify if Fourier Transform Near-Infrared Spectroscopy (FT-NIR) is able to detect significant differences due to the provenance of samples of the same wood species.

FT-NIR is a promising alternative to several traditional analytical techniques (such as timeconsuming and expensive "wet" chemical analysis). It has been successfully applied in wood characterisation. The most recent review on FT-NIR applications into wood, forest products, paper and pulp research has been provided by Tsuchikawa.<sup>2</sup>

### Materials and methods

Wood samples investigated in this project were of Norway spruce (*Picea abies* L. Karst.) growing in different locations and representing borders of the spruce range in Europe. Five sites have been selected for sample collection: central Finland, southern Estonia, northern Poland, southern Poland and northern Italy. The range of Norway spruce and sample locations are presented in Figure 1. A total of 469 samples has been collected. A small block (~20mm width) was cut out from each undercut slab, assuring the opening of the radial plane. The samples were carefully conditioned to guarantee constant moisture content (~12%MC), minimising an effect of differences in the water signal measured by the NIR. All of the experimental samples were measured by using VECTOR 22-N produced by Bruker Optics GmbH, equipped with a standard fiber-optic



Figure 1. Norway spruce range and sample location in Europe.

probe, germanium-diode detector and the thermoplastic resin Spectralon as a reference. The spectral range measured was between  $4000 \text{ cm}^{-1}$  and  $12000 \text{ cm}^{-1}$ . The spectral resolution of the spectrophotometer was  $8 \text{ cm}^{-1}$ , each spectrum has been computed as an average of 32 successive measurements. Five separate spectra were measured on the radial plane of each wood sample. In addition powdered samples (fraction <0.5 mm) and chemical components obtained from wood such as lignin, cellulose and holocellulose were analysed. OPUS 6.5<sup>6</sup> and LabView software have been used for spectra processing. Cluster Analysis (CA), Principal Component Analysis (PCA), Identity Test and 2D correlation techniques were applied for data evaluation. Traditional "wet"

Country	Soluble in hot H <sub>2</sub> O	Soluble in 1% NaOH	Solvent extractives	Cellulose	Holocellulose	Lignin	Ash
Finland	2,05	11,48	1,82	45,16	70,24	28,82	0,28
Estonia	0,65	9,75	1,97	45,62	67,25	27,06	0,29
Poland N	0,93	10,87	1,51	45,52	67,36	26,92	0,31
Poland S	1,02	11,54	2,45	45,49	71,82	27,91	0,27
Italy	0,87	10,65	1,15	45,49	70,75	29,34	0,31

Table 1. Chemical composition of wood samples from different provenances.

chemical analysis was performed in parallel to the spectroscopic measurements. The concentration of cellulose was determined according to the Seifert procedure (by using acetylacetonedioxane-hydrochloric acid). Holocellulose content was obtained by wood delignification by sodium chlorite with addition of acetic acid.<sup>3</sup> Amounts of lignin, solvent extractives and ash were determined according to Tappi standards.<sup>4</sup> Density of samples was measured by both stereometric and X-ray methods. Ages of wooden samples and early/late wood ratios were determined by custom image analysis software developed in LabView.

## **Results and discussion**

Results of chemical analysis performed on milled woods coming from different provenances are summarised in Table 1.

Some minor differences in content can be observed; especially in cases of lignin, extractives and substances soluble in 1%NaOH. Not much variation was noticed for cellulose and ash contents. Average NIR spectra were computed for each location in order to envisage the differences between woods harvested in different countries, as presented in Figure 2.

The shapes of the curves are very similar; however there was evidence of differences after close examination of some characteristic spectral regions. The band assignments correspond to the work of Tsuchikawa *et al.*<sup>5</sup>

Cluster analysis was performed on spectra collected from powdered wood. It was found that all samples were clearly separated, creating five main clusters covering Finland, Estonia, Poland North, Poland South and Italy. None of the spectra were miss-classified.

Similar results were obtained by PCA. All of the wood samples were clearly separated, and fitting spheres were not overlapping [Figure 3(a)].

Additional PCA analyses were performed on the substances extracted from wood by means of chemical analysis. The separation was not unambiguous (some overlapping between the



**Figure 2.** Second derivative of the averaged spectra for milled wood sample from Finland, Estonia, Poland North, Poland South and Italy.



**Figure 3.** PCA of powdered wood fraction <0.5 mm (a) and lignin (b) obtained from different provenance samples. Note: 2nd derivative + vector normalisation, 5 smoothing points, region 9500–4250 cm<sup>-1</sup>.

provenance groups was observed) in the cases of cellulose and holocellulose. A clear separation between provenance groups was, however, achieved by lignin analysis (Figure 3b). A probable reason for such results might be the complex lignin structure, which is much more variable as a result of provenance than the monomer-like structure of cellulose.

The Identity Test (Opus manual)<sup>6</sup> was adopted for validation of the provenance determination algorithm. 80% of samples were used for model generation, and the remaining 20% of samples were taken for validation. In all cases the hit quality calculated was lower than the threshold; and all of the samples were positively classified. The Identity Test might be therefore applied for sorting timber in relation to its origin. Finally, 2D spectral correlation was performed on averaged spectra. The results confirmed the differences in spectra due to origin, and identified the most significant spectral ranges affected by the provenance.

## Conclusions

FT-NIR spectroscopy, a non-destructive, fast and low-cost technique, in combination with appropriate data managing procedures, offers an effective tool to separate groups of wooden specimens of different origin. Although trees of the same wood species, but growing in various locations, have only slight differences in chemical composition, FT-NIR spectroscopy is sensitive enough to detect such differences. The method presented in this paper has an interesting application in wide areas of forest/wood industry, for tracking wood provenances and as a technical tool for detecting logs coming from protected areas (illegal logging detection).

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