

PRELIMINARY STUDY OF WOOD SPECIES IDENTIFICATION BY NIR SPECTROSCOPY

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ABSTRACT

A method for determination of hardwood wood species in chips form by NIR reflectance spectroscopy has been investigated. A series of measurements on chips from seven wood species (beech, acacia, hornbeam, ash-tree, aspen, birch and cherry) with various, controlled amounts of moisture, has been carried out and the spectra were subjected to discriminant analysis (PCA). Wet wood chips had higher rates of successful identification as compared to dry wood chips, probably due to significant variation of moisture content from species to species rather than minute differences in chemical composition. Acacia, birch, hornbeam and aspen were identified with high accuracy. Low identification accuracy has been observed with cherry and ash-tree. The predicted versus actual relationship for all tested wood species is linear with a coefficient of determination $R^2= 0,8054$.

KEY WORDS: NIR spectroscopy, wood species, wood chips, moisture content, PCA

INTRODUCTION

Several pulp mills use a mixture of hardwoods or softwoods for pulp production. A fast and reliable qualitative and quantitative determination of wood mixture composition entering the pulping process is of utmost importance for optimisation of production. Near infrared (NIR) reflectance spectroscopy has been used to predict many wood properties. Wood is a very complex organic material composed of cellulose, hemicelluloses, lignin and extractives. These compounds generate absorption bands in the NIR region. These absorption bands occurring in the NIR region of wood are primarily overtone and combination bands of O-H, N-H and C-H functional groups (Shenk et al. 1992). Application of NIR spectroscopy in wood and pulp research was reviewed by Tsuchikawa (Tsuchikawa 2007). For example, pulp yield, determined by cellulose and lignin content, has been successfully predicted. Wood species can be also determined by NIR reflectance spectroscopy evaluated with chemometric methods such Partial least squares projection to latent structures (PLS) and Principal component analysis (PCA). NIR reflectance spectroscopy is a suitable, fast, non destructive tool for measuring composition of wood chips mixtures (Antti et al. 1996) offering a possibility of online measurement.

A prerequisite to obtain a reliable calibration model with a limited number of samples is application of a statistical experimental design for selecting the composition of the calibration

samples (Box and Draper 1997). Composition of a three component mixture consisting of Swedish pine, Swedish spruce and Polish pine was predicted with high accuracy by evaluation of NIR reflectance spectra of chips ground to powder and evaluation of spectra by PLS (Antti et al. 1996).

The objective of this preliminary study was to verify the possibility of wood species determination in a multi component mixture of hardwood chips by NIR spectroscopy. The results will be used in development of an on-line method for determination of wood species entering the digester for pulp production.

MATERIAL AND METHODS

Material

Seven different species of wood samples in form of chips were tested: beech (*Fagus silvatica* L.), acacia (*Robinia pseudoacacia* L.), hornbeam (*Carpinus betulus* L.), ash-tree (*Fraxinus excelsior* L.), aspen (*Populus tremula* L.), birch (*Betula alba* L.) and cherry (*Cerasus avium* L.). For NIR measurements ten chips for each wood species were selected as standards for discriminant analysis calibration, based on two criteria. Firstly, the measured side of chips was selected as uniform as possible. Secondly, chips exhibiting a range of shades within a single wood species were selected such that they would represent this range of shades.

The first series of standards were *wet chips* from mill wood yard. These chips were either measured immediately after arrival or were stored in a refrigerator. The second series of chips were dried at laboratory temperature for a period of at least 48 hours. These are referred to as *air-dry* chips. The third series of chips were dried in an oven at 105°C until reaching constant weight. These chips were measured immediately after taken out of the oven or kept in sealed plastic bags to avoid absorption of moisture. These chips are referred to as *oven-dry*.

For preparation of the calibration models the following standards were measured: ten standards of seven wood species of tree different moisture content - wet, air-dry and oven-dry wood chips were tested. Unknown wood samples were selected for each specific condition, however, focus was laid on wet chips.

Instrument

Infrared spectrometer iS 10 by Nicolet was used for analysis. For NIR spectrometric measurements, Integrat IR extension by Pike Technologies was used.

Software

The NIR spectral data was collected using Omnic version 8.0 software by Thermo Scientific. The experiment setup was the following: Number of scans: 128; resolution: 4; data spacing 0.482 cm⁻¹; final format: absorbance (Log (1/R)). The collected spectra were analyzed and evaluated using TQ Analyst version. 8.0 software by Thermo Scientific. The PCA method for discriminant analysis was incorporated in the TQ Analyst software.

RESULTS AND DISCUSSION

NIR spectra of tested wood species

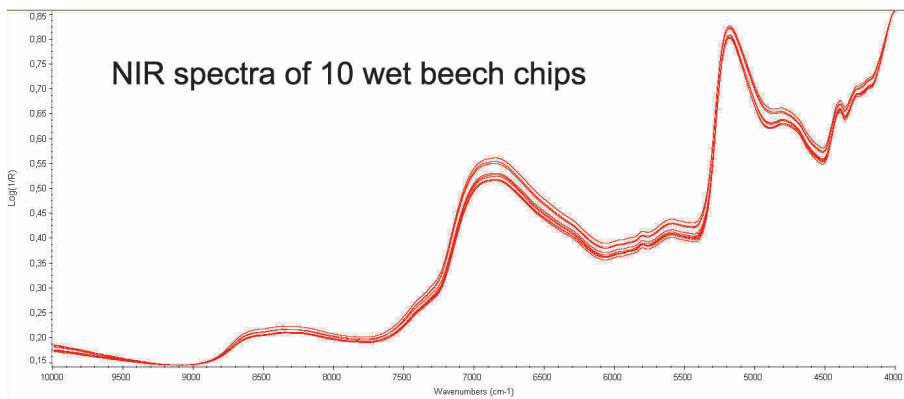
For every material an IR-spectra can be obtained by measuring the reflection from its surface. The spectrum depends, e.g. on chemical composition, density, and the moisture content of

a material. NIR penetrates the sample deeper than other IR regions (a few centimeters) and is therefore useful for measuring wood chips.

The infrared spectra of various wood species are basically very similar due to cellulose being the dominating component. The differences in spectra related to variances in chemical compositions are rather small and in most instances invisible by simple visual inspection of the spectra. Water is the second most important component of wood that is detected in the NIR region.

Fig. 1 shows NIR spectra of ten individual wet beech chips. Between 10000 and 4000 cm^{-1} wave numbers are two maximum associated with water (5500-7000 cm^{-1} and 4000-5000 cm^{-1}). This figure depicts one important aspect that must be considered at all times. Moisture content variations are significant not only between different wood species but chips of same species have various moisture content depending on the location of the chip in the chip pile in the wood yard. The deeper is a chip located the more likely it is able to retain constant moisture. The closer the chip is to the surface more, the is affected by weather conditions, especially excess heat, drought or heavy rain. Evaporation and re-condensation is also an important factor to consider, especially when storing selected samples in a watertight storage container. Condensed water on the chip surface may lead to much higher moisture content readings in spectra than the actual content.

Fig. 1: Spectra of ten individual wet chips of beech wood



The moisture content of chips from various wood species used in this investigation in wet conditions and after air drying is presented in Tab. 1. The difference in moisture content of wet chips of various wood species is significantly different, however the moisture content of air-dry chips of wood species was nearly constant. Acacia is the driest and aspen is the wettest of the species.

The moisture content has a significant influence on NIR spectra of various wood species. Differences of NIR spectra of various wood species in wet, air-dry and oven-dry conditions are shown on Figs. 2-4. On Fig. 2 are spectra of seventy wet chips of seven wood species at various moisture content. These stacked spectra show rather significant variation in moisture content, especially in the interval ranging between 5500-7000 cm^{-1} and 4000-5000 cm^{-1} wave numbers.

Tab. 1: Moisture content in selected wet and air-dry wood species

| Wood species | Moisture content, % | |
|--------------|---------------------|---------------|
| | Wet chips | Air-dry chips |
| Acacia | 21.2 | 10.6 |
| Birch | 32.8 | 12.7 |
| Beech | 32.1 | 11.6 |
| Cherry | 30.6 | 11.7 |
| Hornbeam | 33.7 | 11.9 |
| Ash-tree | 29.3 | 12.1 |
| Aspen | 42.1 | 11.5 |

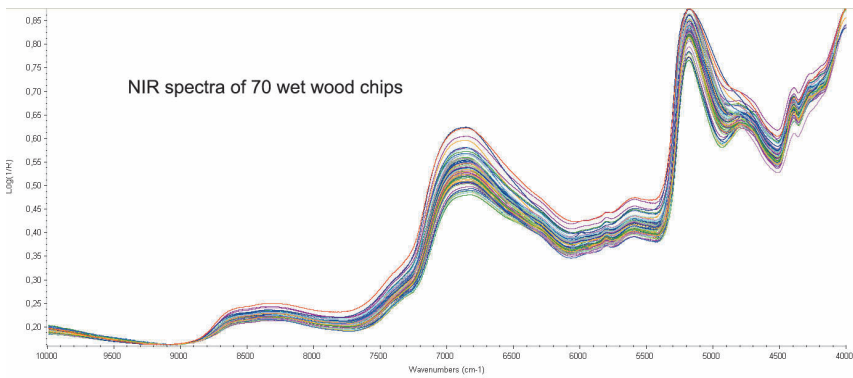


Fig. 2: Spectra of seventy wet chips of seven wood species at various moisture content

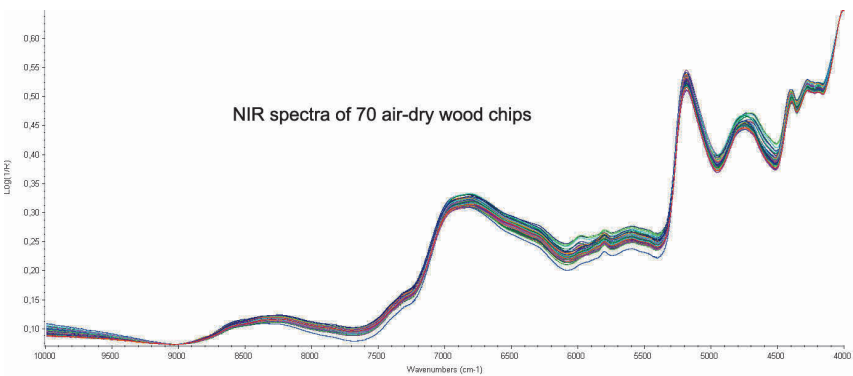


Fig. 3: Spectra of seventy air-dry chips of seven wood species at approximately same moisture content

When we compare Fig. 2 and Fig. 3, a very significant difference of spectra due to moisture content decreases dramatically in case of air-dry wood chips. As Tab. 1 suggests, the moisture content present in these wood chips approaches similar values which explains the extent at which the spectra become so stacked together.

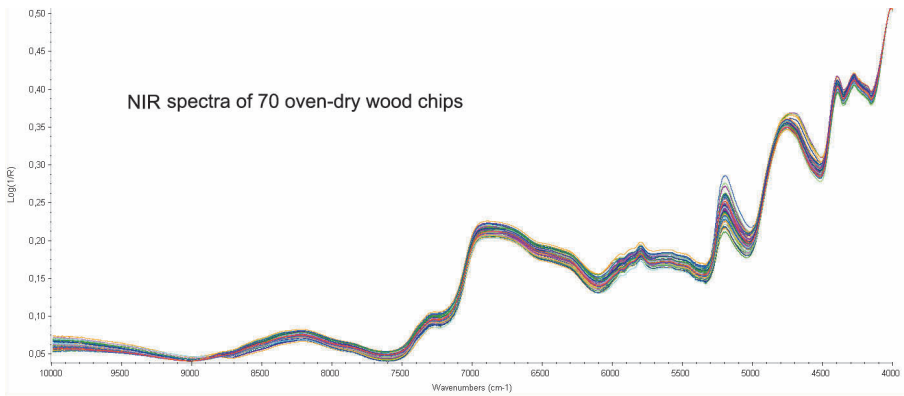


Fig. 4: Spectra of seventy oven-dry wood chips of seven wood species

Extensive drying at 105°C does not make the spectra much more stacked but new peaks are more revealed where water is finally absent (Fig. 4). The difference between Figure 2 and 3 is much more extensive than the difference between the Figure 3 and 4 although there is still a rather profound difference. After a few hours of drying the wood chips at 105°C we expect virtually all water to become absent.

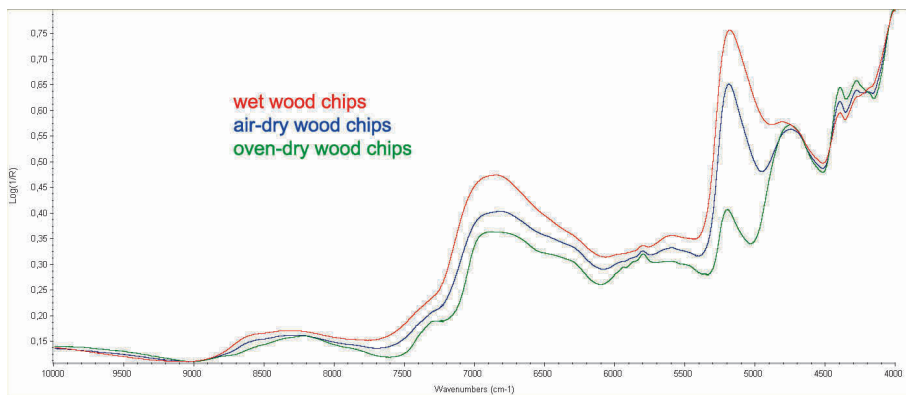


Fig. 5: Average spectrum of wet, air-dry and oven-dry chips of all wood species

The moisture content influences the signal height as well as the overall NIR spectrum shape as shown on Fig. 5. As the chips become dry, influence of water on NIR spectra decreases and

additional peaks become more visible. Drying at 105°C may in fact enhance some partly hidden peaks as well as slightly change the chemical structure. Temperature is high enough to be in fact responsible for some, although not profound changes in chemical composition. We suspected that the alteration of chemical composition may have a positive effect on identification. However, wet samples were distinguished more easily. Differences in moisture content seem to be the key factor for PCA discriminant analysis as compared to minute composition differences between various wood species.

Average NIR spectra of wet and air-dry chips for all seven wood species are shown on Figs 6 and 7. The average spectra of all air-dry wood species are more stacked together than NIR spectra of wet chips.

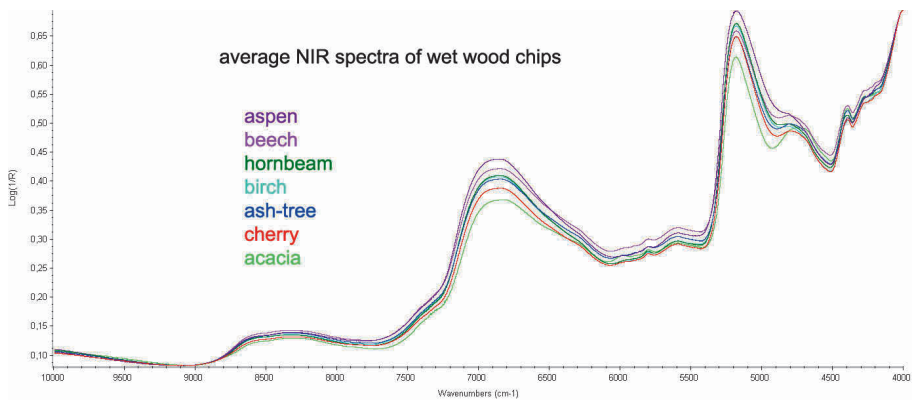


Fig. 6: Average spectra of seven wood species wet chips

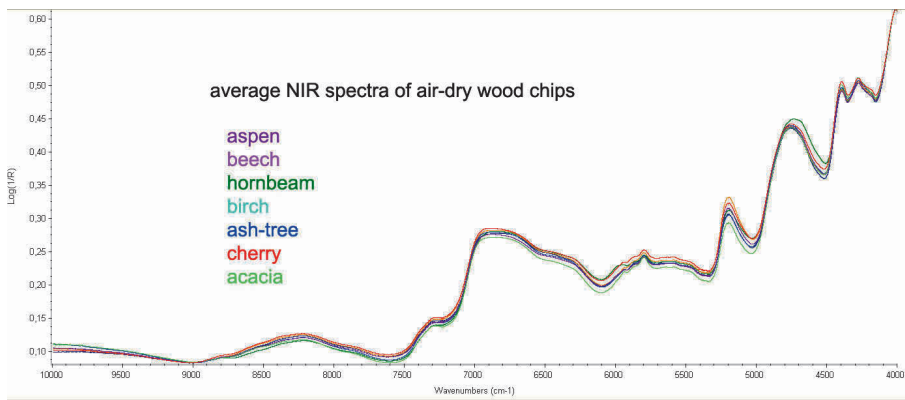


Fig. 7: Average spectra of seven wood species air-dry chips

NIR spectra evaluation

For identification of wood species in a mixture of all seven wood species NIR spectra of wet chips were evaluated as these are showing more distinct differences between wood species (Fig. 6).

The PCA method was used as a feature of TQ_Analyst software. When carefully inspecting spectra or even focusing on particular segments of spectra in detail, there are no visible distinctions between these spectra with the exception of moisture content variations. Distinguishing wood species simply by visual evaluation of these spectra regardless of details is practically impossible. Instead, the PCA method is a means of distinguishing different spectra based on similarities and differences revealed in complex mathematical algorithms that are otherwise not visible to the naked eye. These mathematical operations take into account larger or smaller spectra segments and their characteristics such as the gradient of incline, enabling to operate at first or second derivative of spectra, looking for particular trends. The first or second derivative sometimes enhances the trend or diminishes, if there is any trend at all. In our case, we created three PCA models with seven, four and three variables.

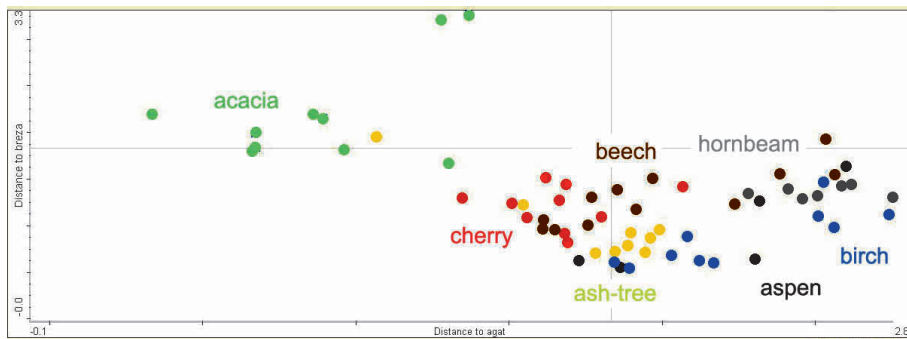


Fig. 8: PCA diagram of seven wet wood species (a seven variable model)

The first model, depicted in Fig. 8, consider all seven wood species as separate variables. The model is rather complicated and clusters are quite scattered, merging to one another to some extent. These results decrease level of correct wood species specification. Surprisingly according to our calculations the success hit rate in positive wood identification exceeded 70% in unknown samples. As Fig. 8 suggests, acacia creates the most separate and the best defined cluster. Thus acacia is most definitely the easiest distinguished wood species, as compared to the other present wood species with a success rate of over 90%.

Fig. 9 is a PCA model of four variables, where the three most abundant wood species are presented as separate variables and the remaining four wood species are put together as one distinct variable, marked as “other”. The situation in this model becomes much simpler. Again acacia is the easiest wood species to be distinguished from the group, but otherwise there is not a significant improvement of separation by individual wood species as compared with the first model depicted in Fig. 8.

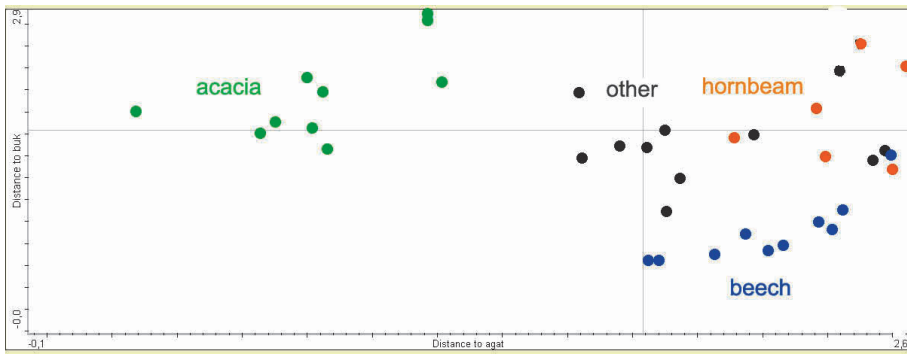


Fig. 9: PCA diagram of three wood species and the rest as a separate variable (a four variable mode)

Fig. 10 depicts the simplest PCA model with 3 variables only. The variable “other” is excluded and the only wood species participating in this model are acacia, beech and hornbeam. The clusters are quite distinct at this point, however, acacia is the best distinguished wood species in this case as well. In fact beech and hornbeam were exchanged and were in many unknown samples not identified positively. Acacia was again the most distinguished wood species with a success rate of over 90% (Tab. 2 and Fig. 11).

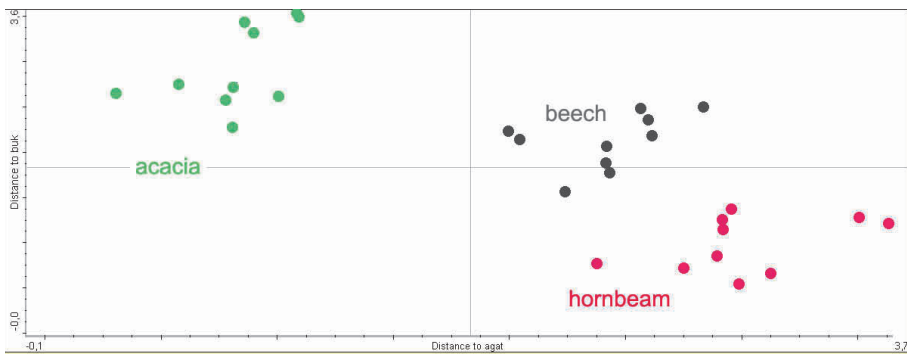


Fig. 10: PCA diagram of three wood species (a three variable model)

Tab. 2: Predicted versus actual count of wood chips per wood species in unknown samples based on the first model, depicted in Fig. 8

| Wood species | Actual | Predicted | Difference |
|--------------|--------|-----------|------------|
| Acacia | 13 | 13 | 0 |
| Birch | 8 | 7 | -1 |
| Beech | 42 | 27 | -15 |
| Cherry | 8 | 13 | 5 |
| Hornbeam | 13 | 15 | 2 |
| Ash-tree | 8 | 14 | 6 |
| Aspen | 8 | 6 | -2 |

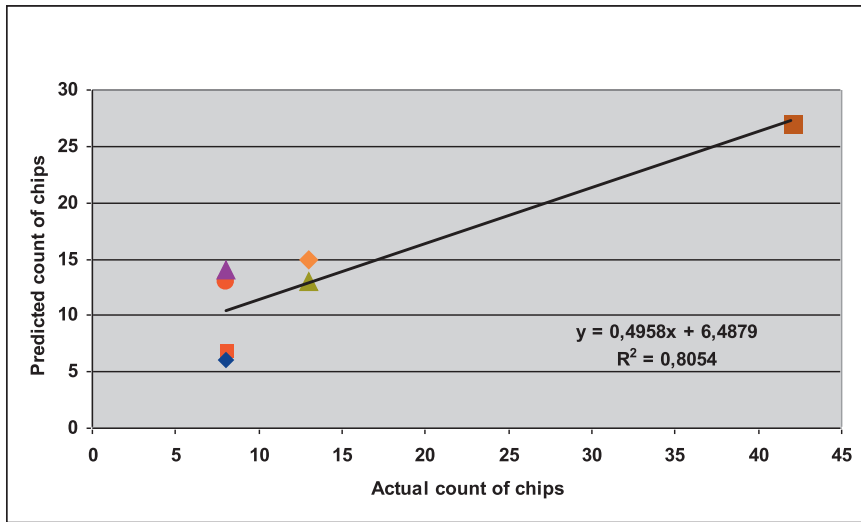


Fig. 11: Predicted versus actual count of wood chips per wood species in unknown samples

CONCLUSIONS

NIR reflectance spectra of seven wood species (beech, acacia, hornbeam, ash-tree, aspen, birch and cherry) with various, controlled amounts of moisture have been measured in the range between 10000 and 4000 wave numbers in order to determine the number of chips of individual species in a mixture. A PCA model with three, four and seven variables was used in an attempt to distinguish between various wood species. Preliminary data suggests that various wood species are easier to distinguish with wet chips as stored in a mill wood yard as compared to air-dry or oven-dry chips. Results of measurement on wet chips showed that with a varying degree of success, it is possible to separate as many as seven wood species. The success rate varies between different species, with lesser impact on how many variables are used in the model. These preliminary results indicate a possibility of on-line identification of individual wood species by NIR reflectance spectroscopy. This will be investigated in further studies.

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