Identification and characterization of Estonian wood using ATR-FTIR spectroscopy combined with multivariate analysis

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Introduction

Figure: Analyzed woods: pine, birch, spruce

The push towards greener alternatives brings lignin into consideration as a sustainable starting material, but if lignin is considered a waste-product, the quality of extracted lignin will be too low to valorize it fully.[1] To compare lignins that are collected in different conditions, better, faster, and more reliable analytical methods are needed. Analytical chemistry provides vital information about lignin extraction process, its yield and lignin purity.[2]

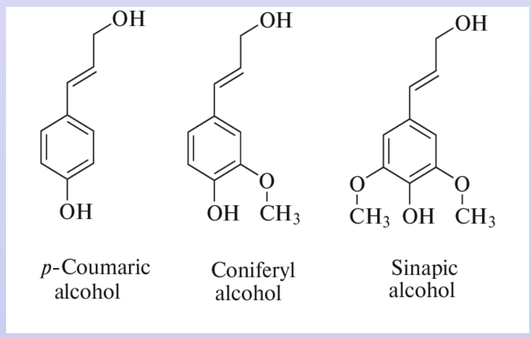

Figure: Lignin monomers (H, G and S units)

Currently, most methods are to do with lignin functional groups, such as hydroxyl, methoxy, carbonyl and carboxyl groups. Elemental analysis, infrared spectroscopy and nuclear magnetic resonance spectroscopy are used to characterize lignin. Spectroscopic methods such as infrared spectroscopy are often used to build chemometric models because of the ease of use and nondestructive nature.[2]

Results & Discussion

Developing new analytical methods is crucial for characterization of lignocellulosic biomass and quality control for valorized products. Composition of lignocellulosic biomass was determined using classical "wet chemistry" and spectroscopic methods and multivariate data was analyzed using chemometric methods. Klason lignin[3,4] and extractives content[5] were measured gravimetrically in 90 wood samples. The accuracy of Klason method was determined using a certified reference material.

It was determined that:

- birch contains 25,8 ± 3,2 % of lignin and 3,5 ± 0,8 % of extractives,
- spruce contains 31,1 ± 3,0 % of lignin and 2,0 ± 0,7 % of extractives,
- pine contains 32,2 ± 3,0 % of lignin and 6,1 ± 3,0 % of extractives.

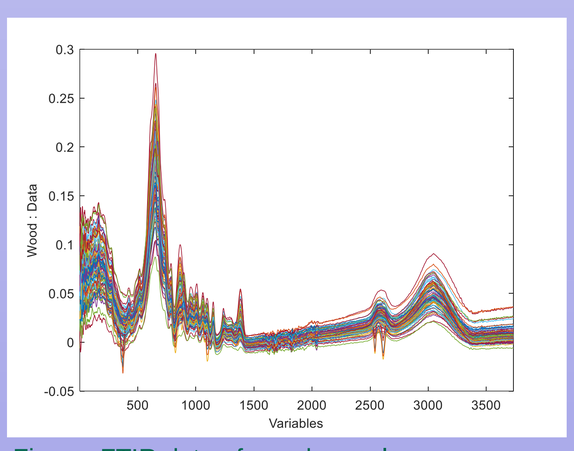

Figure: FTIR data of wood samples

There results were in compliance with data gathered from the literature. Infrared spectra were measured for all samples and the data was used to conduct a PCA analysis, which showed that the samples clearly separated to hardwood and softwood clusters, but the area of logging and wood composition had no correlation.

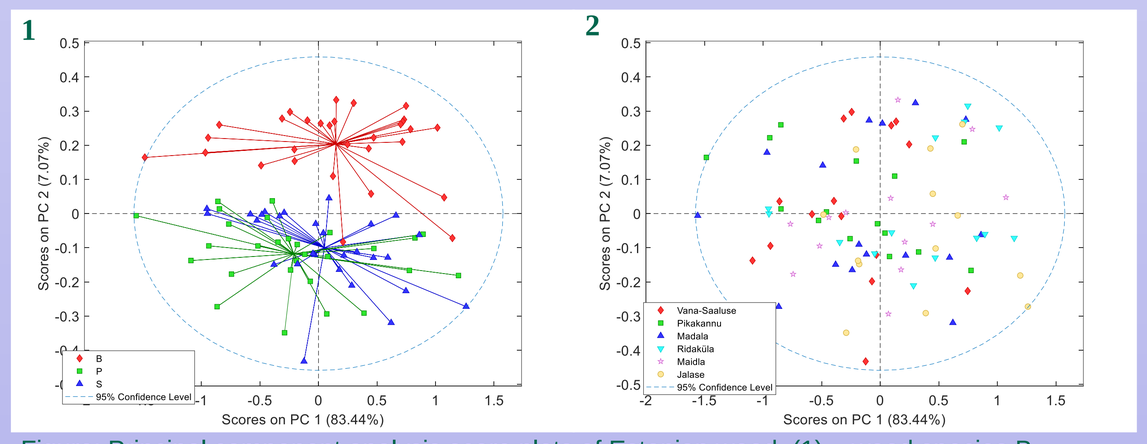

Figure: Principal component analysis score plots of Estonian wood. (1) – wood species B-birch, P-pine, S-spruce. (2) – logging areas

Acid soluble lignin (ASL), Klason lignin (KL) and extractives (EA) amounts were used to build a PLSR model. Five PLS components were used, and the prediction performance was determined using standard cross-validation.

PLS model was able to predict ASL, KL and EA content with a correlation coefficient of R^2~0,9 creating the model and R^2 0,6-0,8 cross-validating. The PLSR model was able to predict ASL, KL and EA concentrations with a relatively low standard deviation (up to 15%) and the predicted concentrations were close to measured concentrations. This shows that multivariate data analysis can be used to replace classical chemistry methods, such as Klason method, in day-to-day wood analysis.

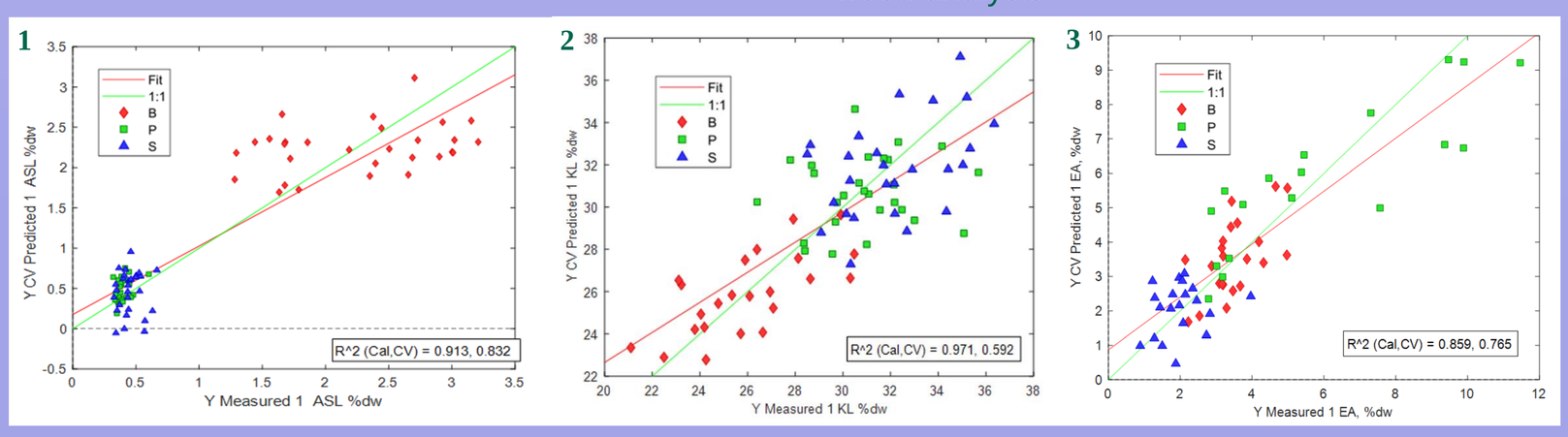

Figure: Partial least squares regression models using 5 latent variables to predict (1) acid-soluble lignin content, (2) Klason lignin content and (3) extractives content

Conclusions

New analytical methods are needed to bring wood chemistry up to current standards. With the help of chemometrics, classical analytical methods can be replaced with a much easier spectroscopic method that takes less time, is less expensive and is nondestructive. Initial data exploration using principal component analysis was promising, and the following partial least squares regression model showed that it is possible to predict acid soluble lignin, Klason lignin and extractives content in Estonian wood biomasses using infrared spectroscopy.

References:

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