CHEMICAL PROPERTIES ESTIMATED USING IMAGING NIR – A PRESTUDY

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Objective with the preliminary investigation

- There are several studies available that shows the possibility to use NIR-models to predict chemical properties in wood
  - For characterization of properties such as wood density, chemical composition, lignin and extractive content (Tsuchikawa et al. 2015).
- In spring 2017 we decided to do a pre-study to test possibilities to get a good reference dataset based on drill cores
  - If a NIR-model indicated the same trend at more or less the correct level, we would consider that a good indication that the method could be used
  - NIR-models could then be used to predict chemical properties without use of wet chemistry analysis
- One parameter that we wanted to evaluate was whether the sample size was sufficient for calibration modelling, as the wet chemistry method requires a minimum amount of wood, which also impacts the resolution at which reference measurements can be made
- The pre-study should give answers to :
  - How many samples do we need in this case to get good models?
  - How should the sampling be done? Is it possible to use drill cores?
  - How much sampling material is needed?

The overall goals with the subproject

- Development of methods:
  - Sample preparation (flattening drill cores)
  - NIR models to estimate chemical composition

- Chemical properties included:
  - lignin
  - cellulose
  - hemicellulose
  - sugars
  - extractives

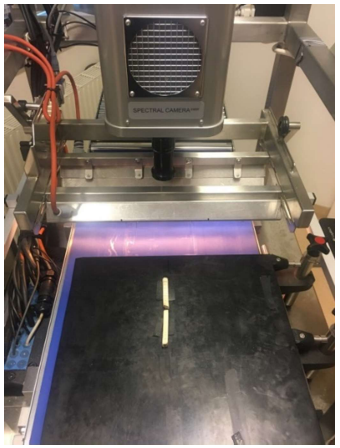
- Use the NIR models to estimate chemical composition for the Gran SSF SilviScan samples

Wood cores

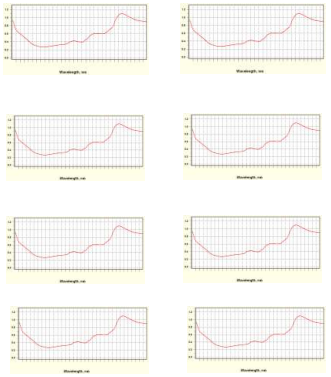
Calibration of chemical models for wood cores – method

Prepared wood cores

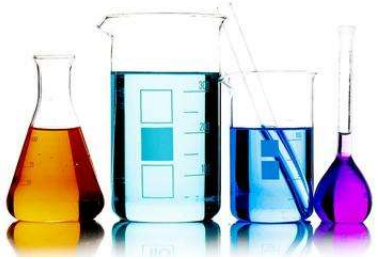
2D NIR measurements



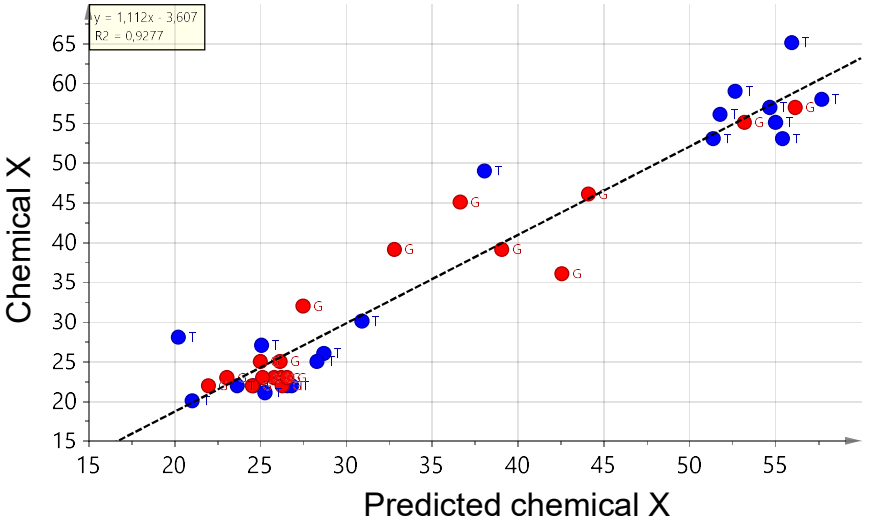
Spectra



Lab analysis



Calibration modelling



RI. SE

Chemical reference – preliminary dataset

- A small dataset with chemical data compiled
- Subsamples from wood cores
  - Each subsample will be obtained from 4-5 rings from a 10 mm increment core.
- The current preliminary dataset:
  5 wood cores => 15 subsamples
  - To few sample to get reliable models
  - The number of samples needed is determined by the variation in the properties and how reliable the reference analysis is

Wet chemistry analysis

- Cellulose (%) and hemicellulose (%)
- Extractives (%)
- Lignin: Klason (% TS), Acid-soluble (%)
- Suger content (g/kg TS): Arabinose, Galactose, Glucose, Xylose, Mannose

- Sample amount:
  - Sample amounts available in the test set: 100-400 mg.
  - Sample amount needed for the analysis:
    Lignin: 1g
    Carbohydrate analysis: 300 mg
    Extractives: several grams
  - Due to the limited amount of sample material the normal methods adjusted
- Chemical analysis done at MORE

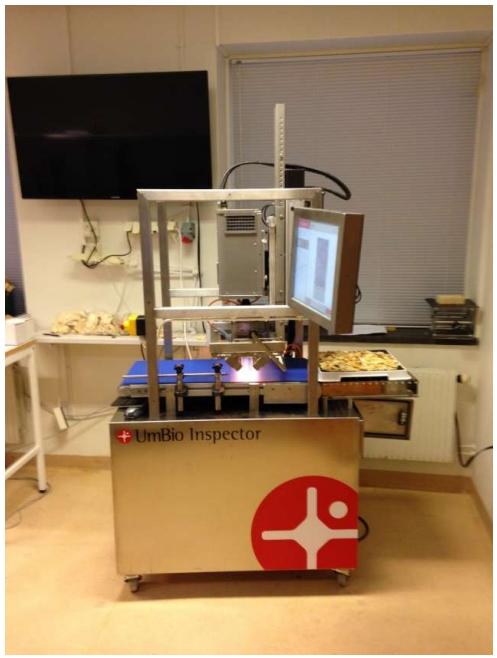
NIR imaging

NIR scanner

- Camera
  - Line scanner, 320 pixels
  - NIR region: 900 – 2500 nm
  - MCT sensor (mercury cadmium telluride)

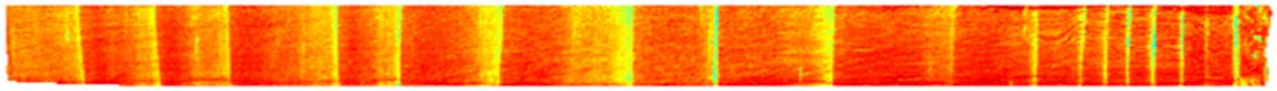
- Changeable objective lenses
  - Macro lens => 30 µm pixel size, FOV 10 mm
  - 22,5 lens => ~.5 mm to 1.2 mm pixel size, FOV 150 mm to 420 mm

- Conveyor belt
  - Sample moves with constant speed in front of the camera with 100 fps
  - Each image produces a line with 320 pixels and 256 wavelengths

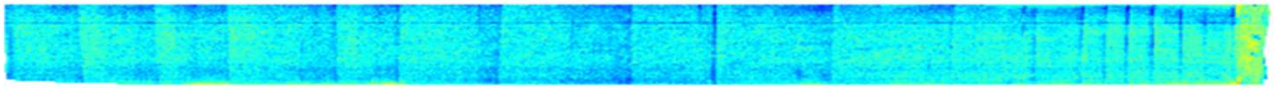


NIR imaging – example of data
Prinicipal component analysis

PC 1



PC 2

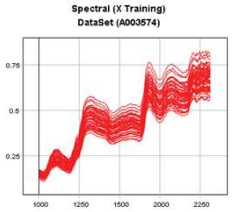


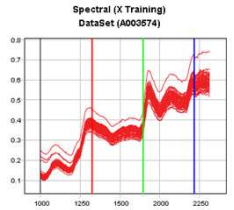
PC 3

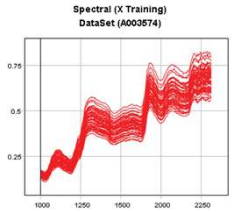
Models

Modelling

- Spectra extracted and grouped into a matrix for each subsample
- Each spectra connected to a chemical reference value for the subsample
- All subsamples compiled into one large calibration matrix
- Multivariate modelling
- Spectral pre-treatment

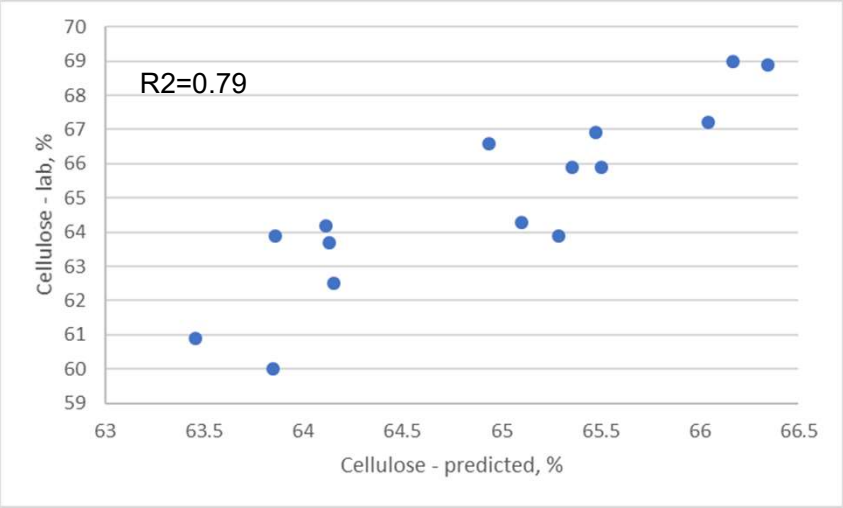




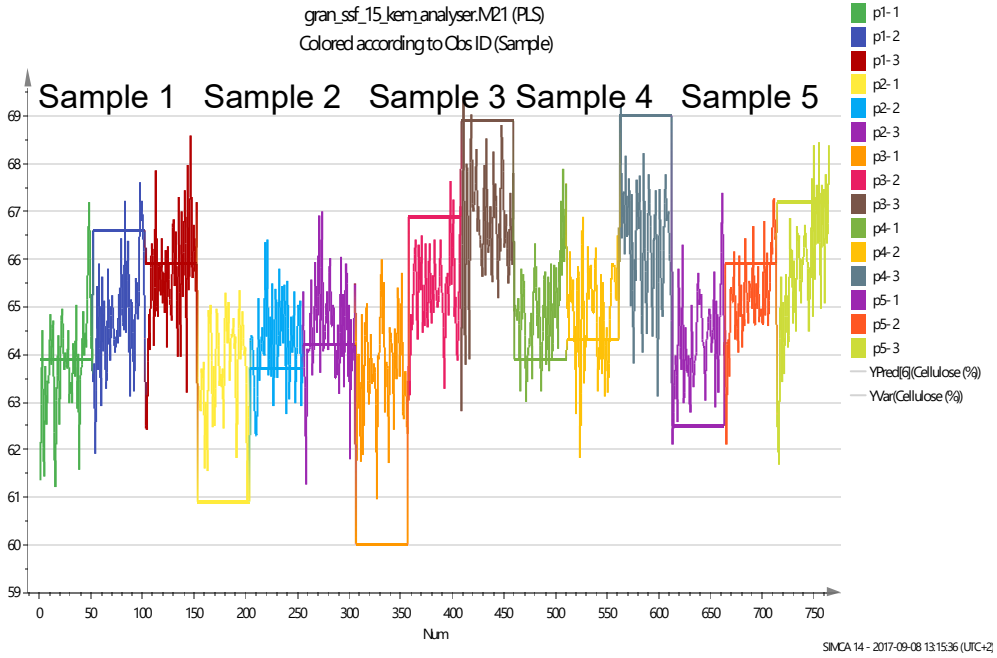


Cellulose

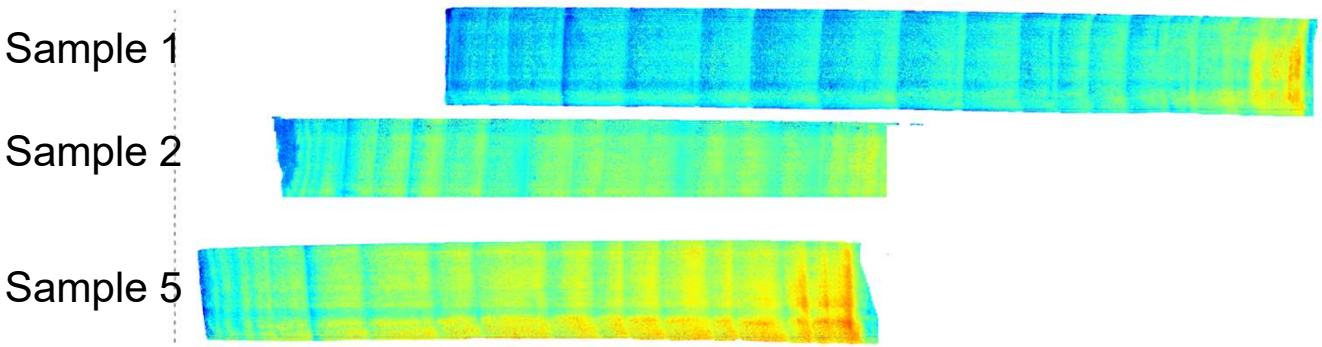
Average prediction per sample

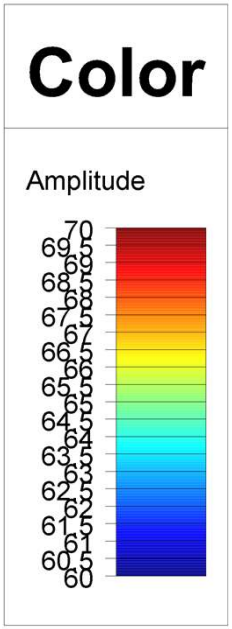


Prediction based on individual spectra



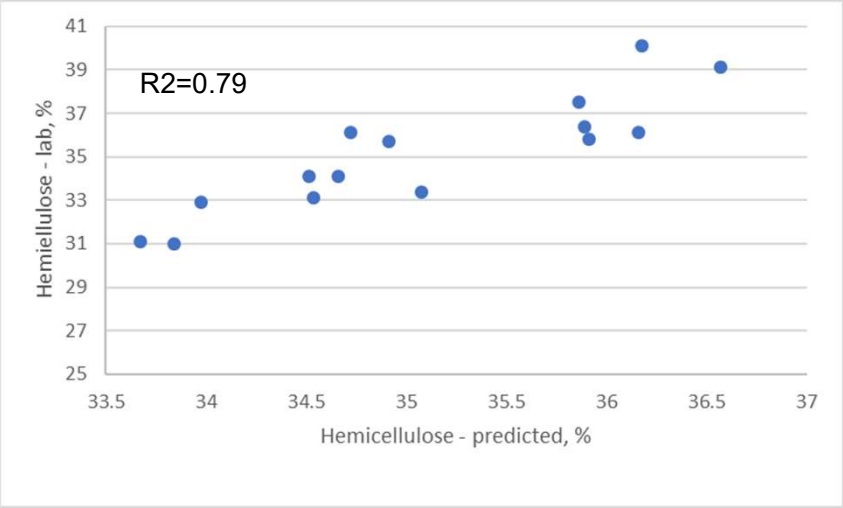
Cellulose


Sample 1
Sample 2
Sample 5

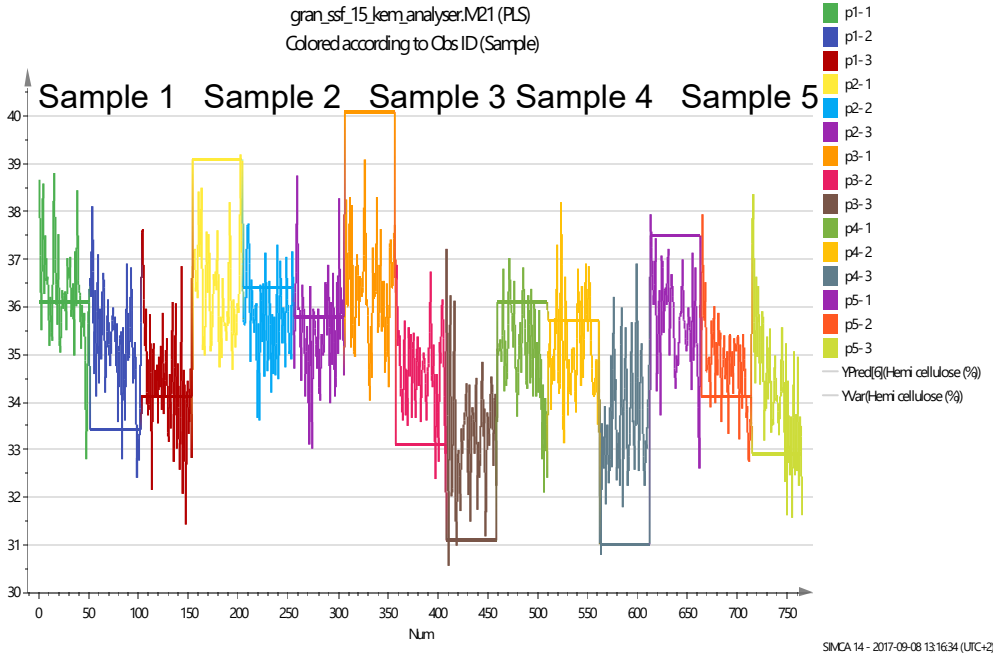

Color
Amplitude
70
69.5
68.5
68
67.5
66.5
66
65.5
64.5
64
63.5
62.5
62
61.5
60.5
60

Hemicellulose

Average prediction per sample



Prediction based on individual spectra

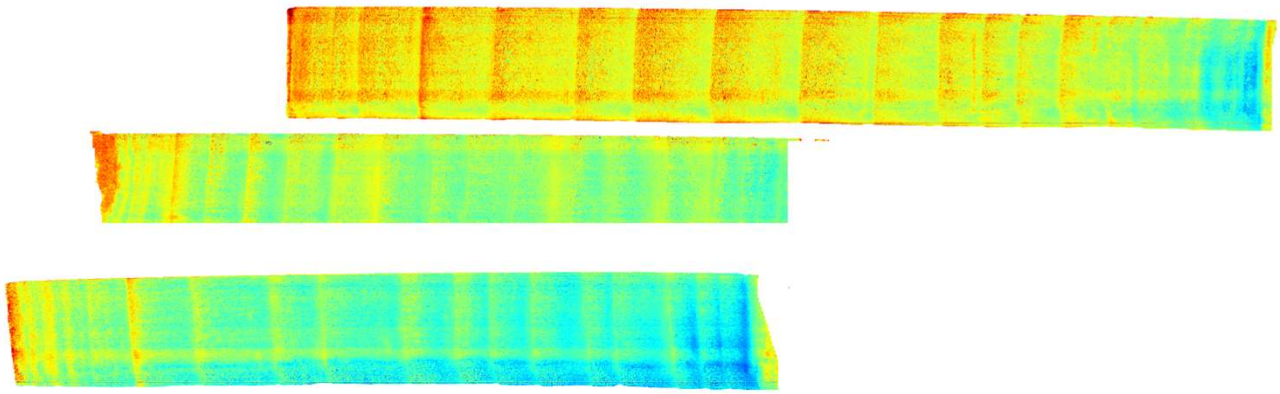


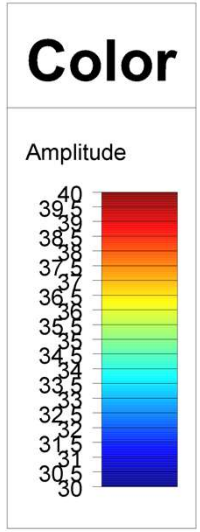
Hemicellulose

Sample 1

Sample 2

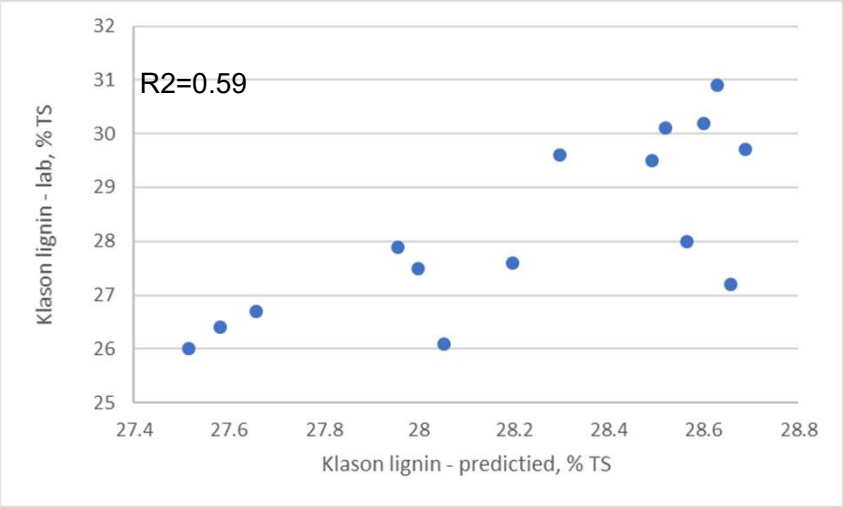
Sample 5



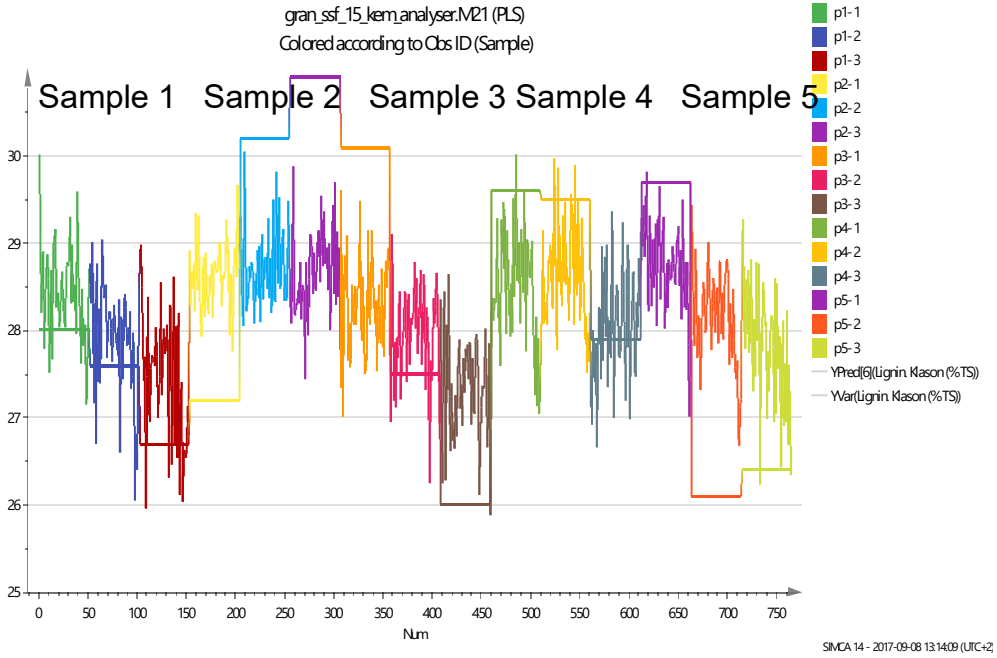


Lignin

Average prediction per sample



Prediction based on individual spectra

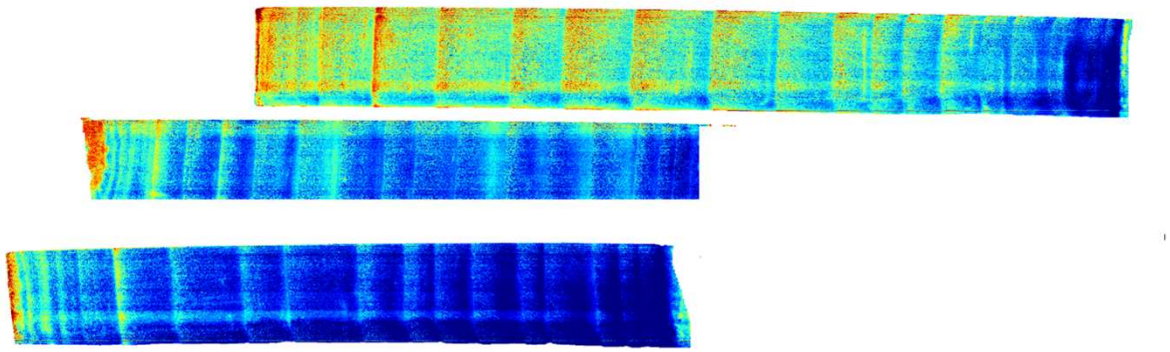


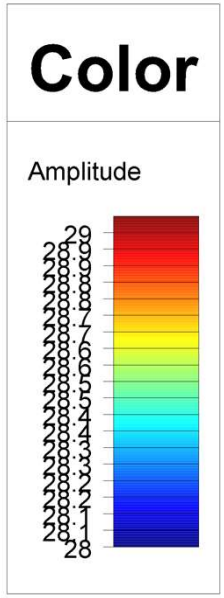
Lignin

Sample 1

Sample 2

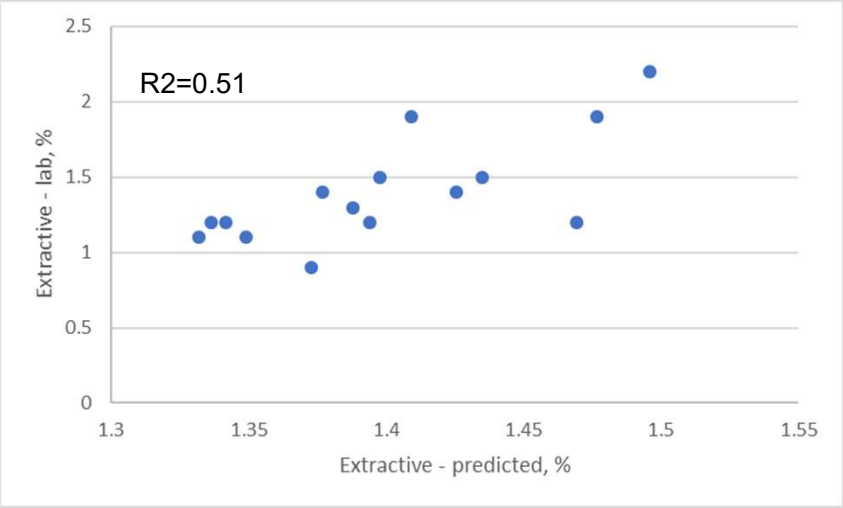
Sample 5



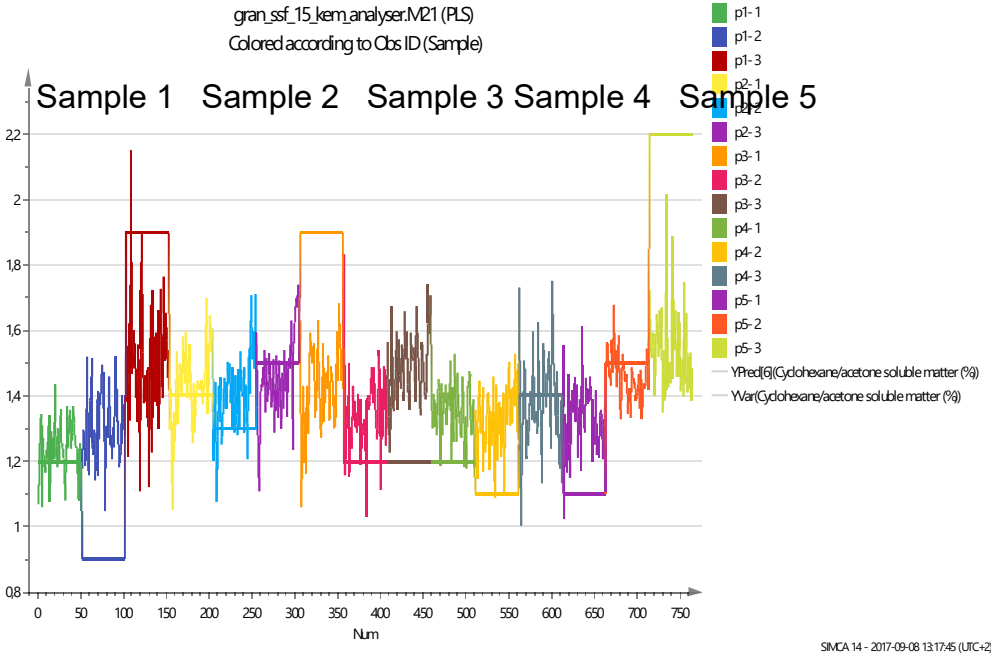


Extractives

Average prediction per sample



Prediction based on individual spectra

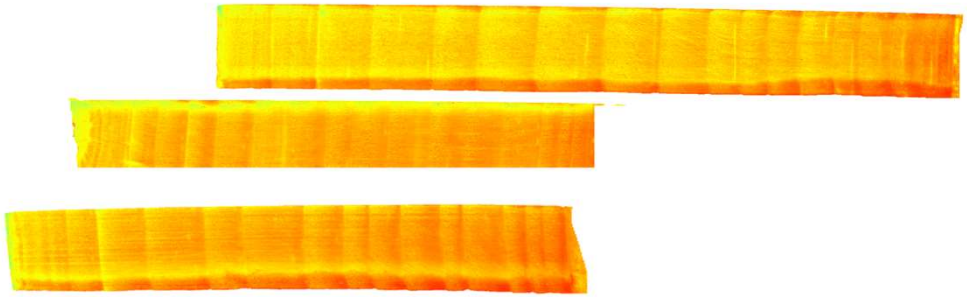


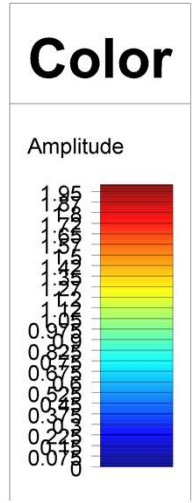
Sample 1

Sample 2

Sample 5

Extractives



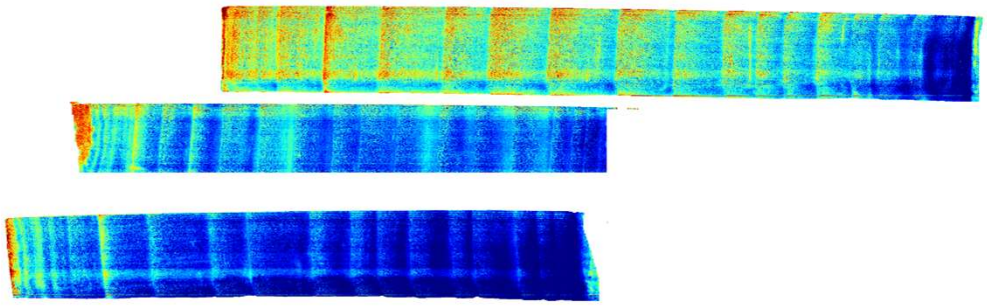


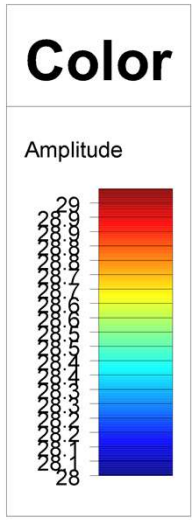
Lignin

Sample 1

Sample 2

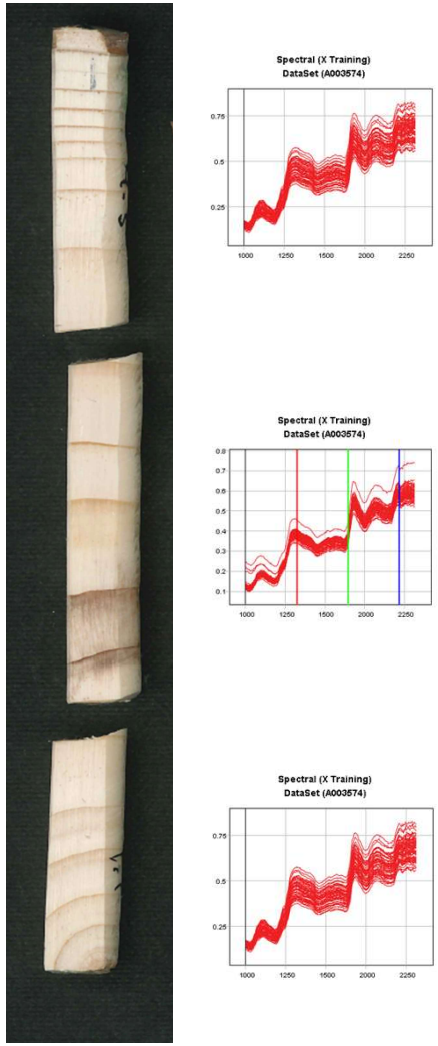
Sample 5





Spectra

- **Spectra extracted and grouped into a matrix for each subsample**
- Yesterday we did an observation!
- Only 50 spectra exported from each subsample!
- They are systematically picked, but probably to few to fully reflect the variation in a piece of wood
- There is thus a significant possibility for improvements
- The developer of the software has been contacted to find out how to solve this



Models

- Predicted trends correlated with wet chemistry data in most cases, but current models are not good enough
- Cellulose and hemicellulose
  - Promising, but the errors are too large
- Lignin
  - Limited variation in available sample, but lignin should be possible
  - Measurement error in reference data may suffer from the limited amount of sample material
- Extractives difficult
  - Limited variation on current dataset
  - Relatively high measurement error in the reference method?
  - Doubtful if models can be developed for extractives, due to the limited variation for this sample material (young trees)?

Conclusion

- Predicted trends correlated with wet chemistry data in most cases
- Current dataset too limited for development of reliable models
- Sample size too small for wet chemistry
  - at least if listed all properties should be analyzed
- There are significant variations in chemistry between year rings =>
  consequently, reference sampling over several year rings decreases resolution and degrades models
- Variation in some properties is possibly too small to make any kind of modelling useful,
  in these cases wet chemistry data could possibly be presented with standard deviations as representative for all samples

- For reliable models we need:
  - More samples – 60 samples preferred
  - Larger sample size for more reliable wet chemistry

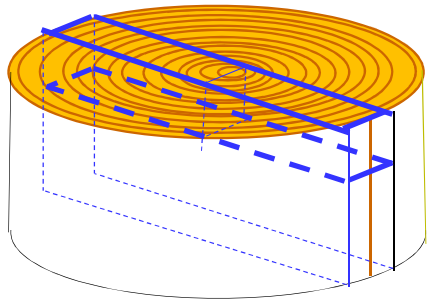
RI. SE

How to get more smaples?

- More samples needed for reliable models
  - Is it possible to use the Ekebo discs for this?
  - Stored deep frozen at Skogforsk
- Larger sample size for more reliable reference analysis
  - Sampling from wood discs gives more sample material
  - Sample from only one yearring possible
    (or even better wood from only earlywood/latewood)
- Sampling of additional samples can be supported by using the current models
  - Selection of suitable sample possible to do

The Ekebo discs

- From the discs one centre piece along the diameter of the sample has been cut out
- The remaining sample material is stored deep frozen in Ekebo – 800 disc
- Selection of 40 discs
  - Sand the surface of the disc “closest to the SilviScan sample”
  - Scan the discs with imagine NIR to get spectra
  - Use the existing NIR models to predict the chemical in the wood discs to get a picture of the variation
  - Based on that select samples to get a well-balanced dataset



- Based on these steps selection of additional sample for chemical reference measurements can be done
  - 60 samples for chemical reference analysis