

NEAR INFRARED SPECTROSCOPY FOR THE QUALITY CONTROL OF FELTING COMBUSTIBLE CARTRIDGE CASES

From samples to analysis

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Raw materials



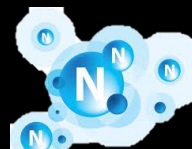
Pulping



Analysis



Nitrogen content



Production process

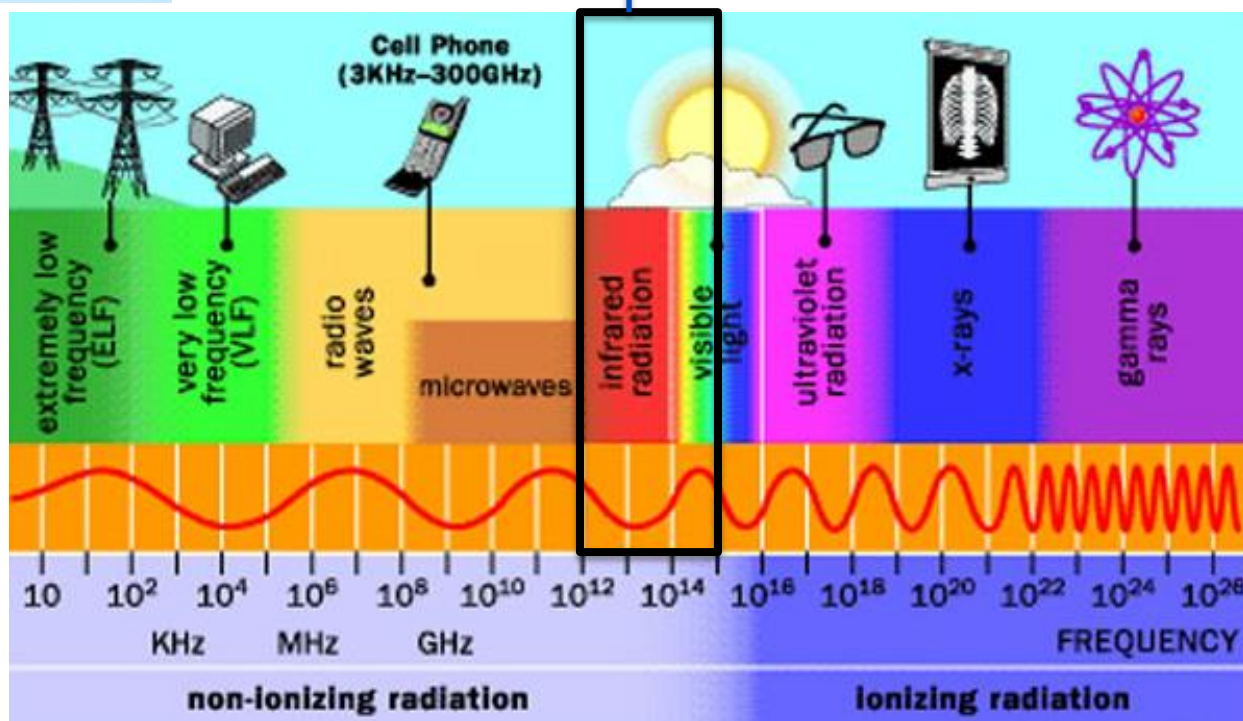
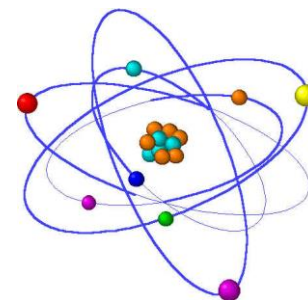
Combustible Cartridge Cases





$$E_{\text{photon}} = h\nu = hc / \lambda$$

Vibration of atomic bonds





Beater additive process for CCC and Nitrogen content analysis



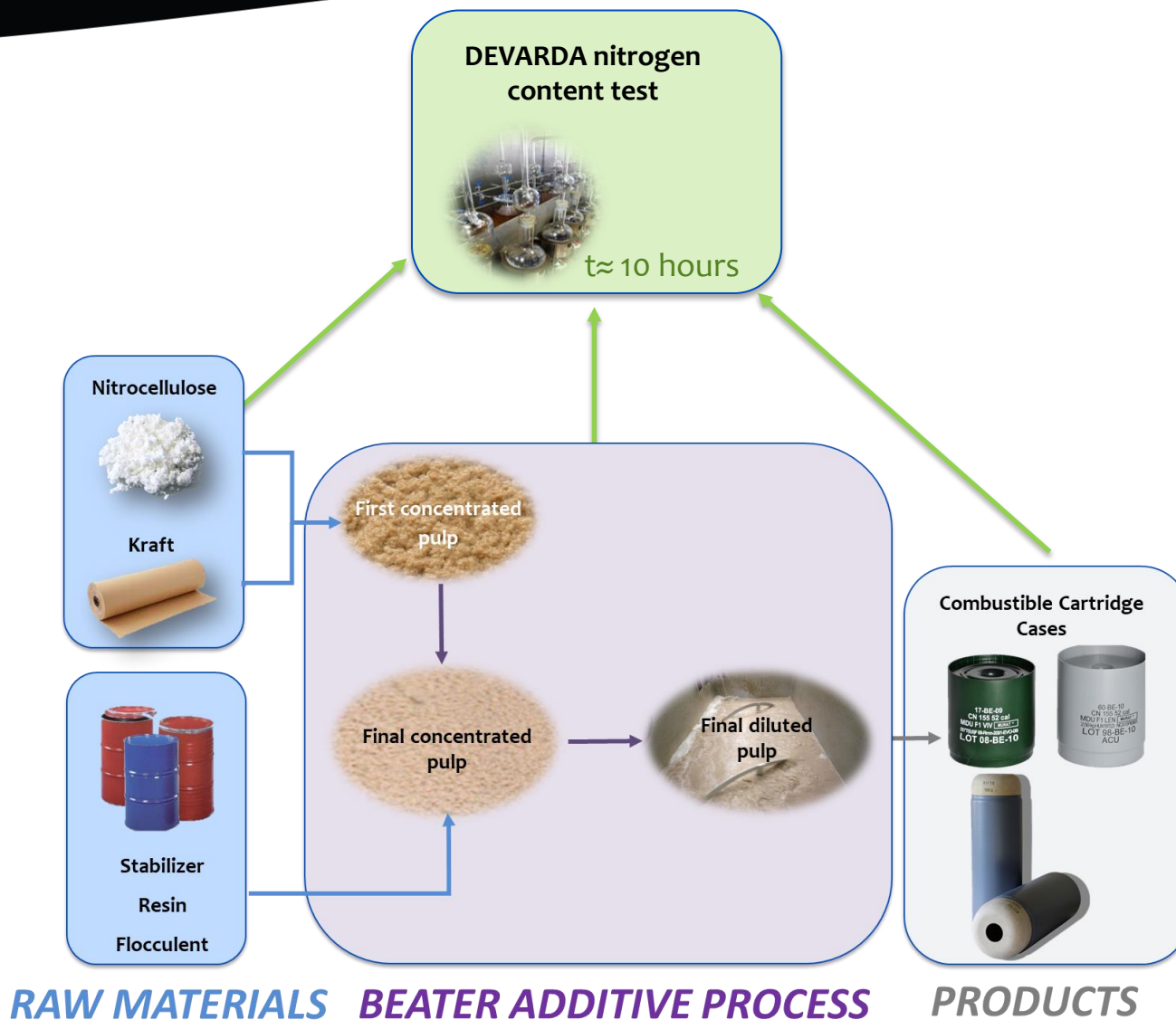
Near Infrared Spectroscopy From samples to analysis

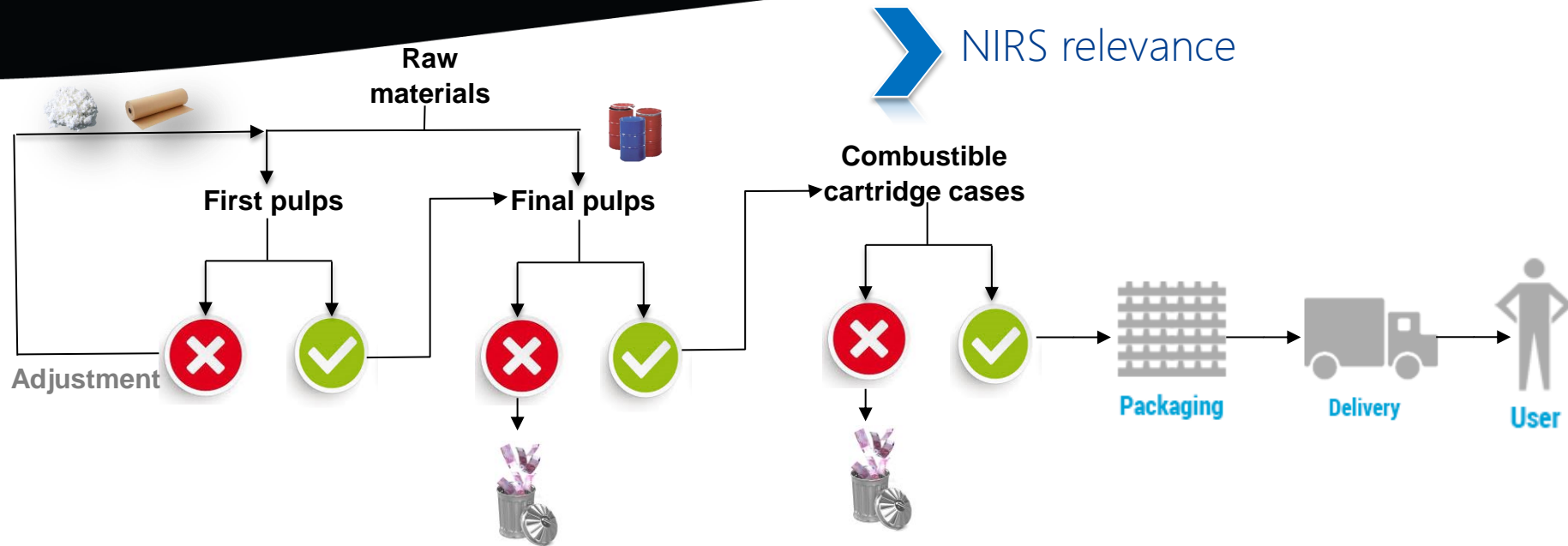


Calibration models and prediction



Conclusion/Outlooks





NIRS relevance

Devarda titration test:



- Implementation time of samples and analysis: 10h (1day)
- Low flexibility in production to adjust pulps
- Non continuous or semi continuous analysis
- Precision: 0,02

Reference analysis

Near Infrared Spectroscopy system (NIRS):

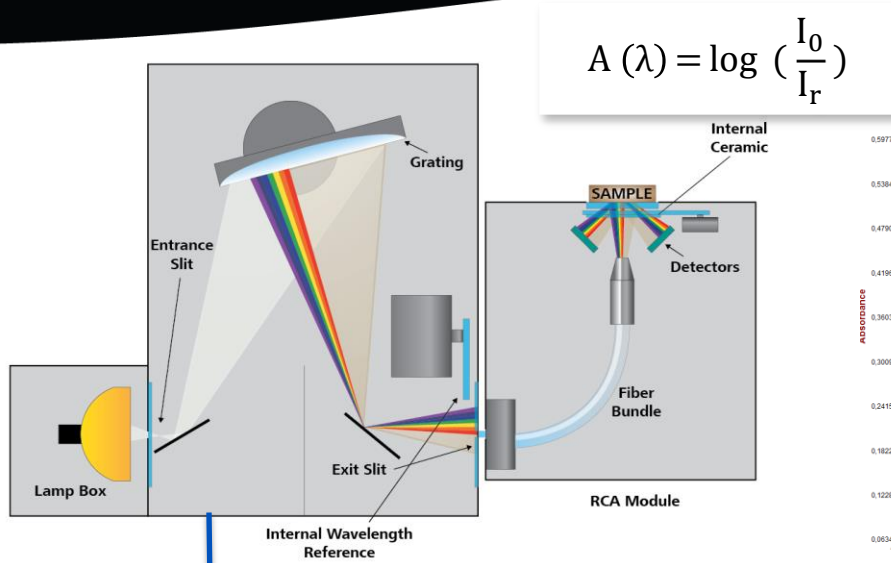


- Indirect analysis method → Calibration time
- Data base samples
- Reliable calibration models
- Precision: >0,02
- Implementation time of samples and analysis: <1h (1day)
- High flexibility in production to adjust pulps
- continuous or semi continuous analysis
- Low cost

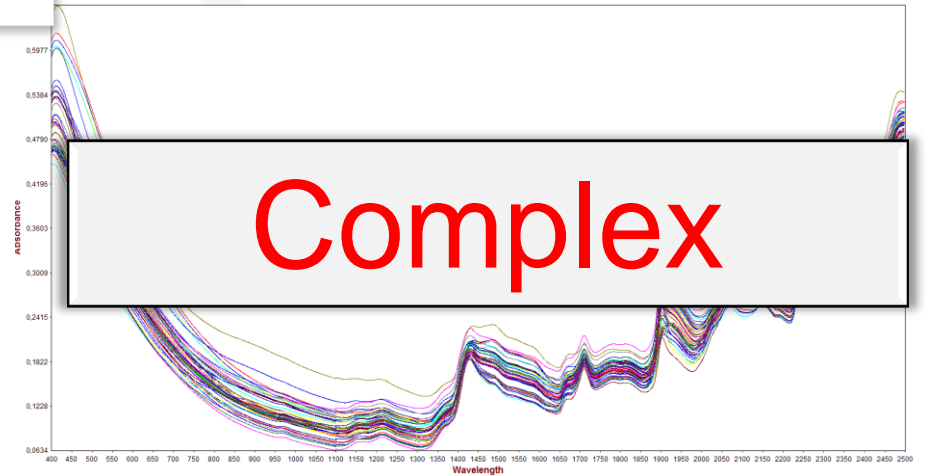




Physico-chemical influences

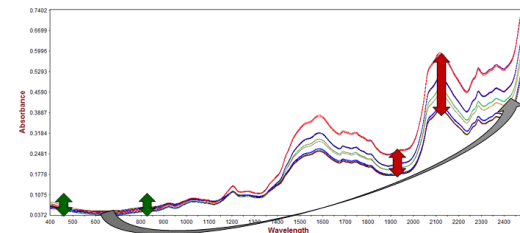


$$A(\lambda) = \log \left(\frac{I_0}{I_r} \right)$$



Physical factors of samples influencing NIR spectrum:

- Density
- Refractive index Surface
- Surface condition
- Cristalline shape...

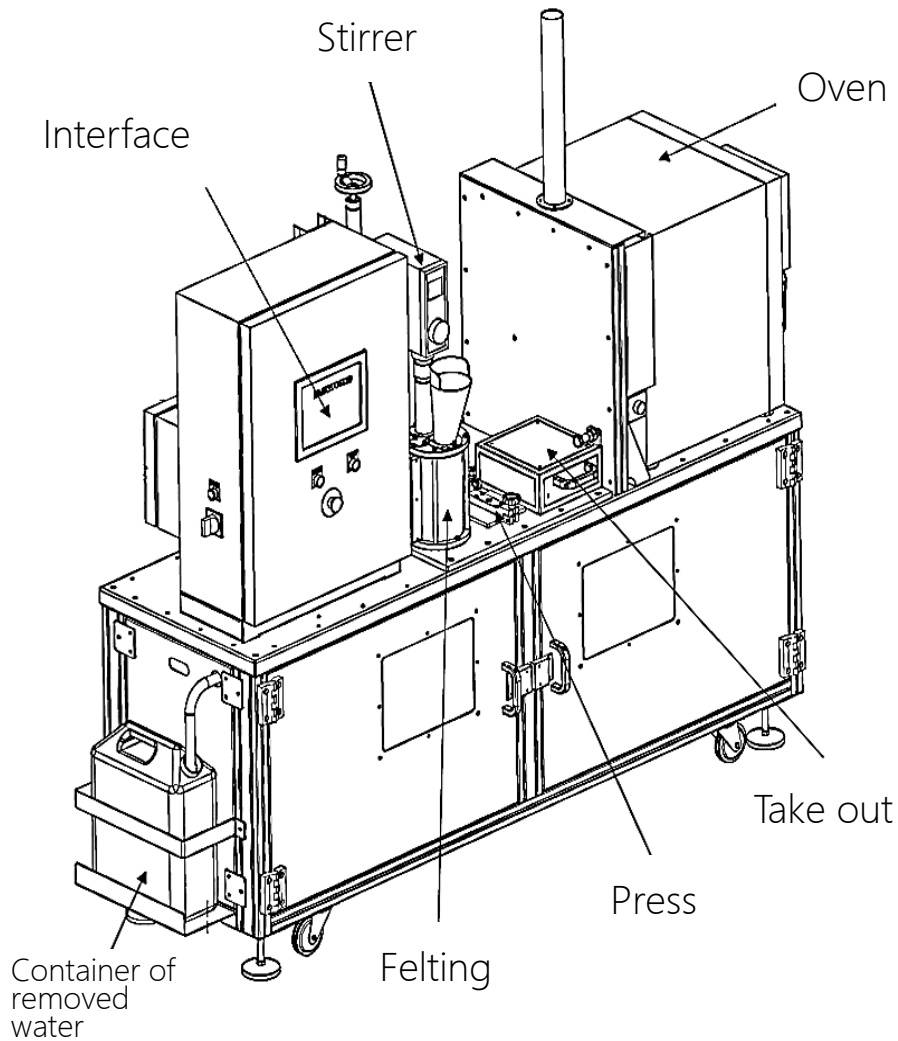


Chemical factors influencing NIR spectrum : %N, Water content ...composition

1st harmonic, 2nd harmonic, 3rd harmonic ... and combinations → impossible to have a direct interpretation of spectrum



Samples definition



Disc of pulps sample

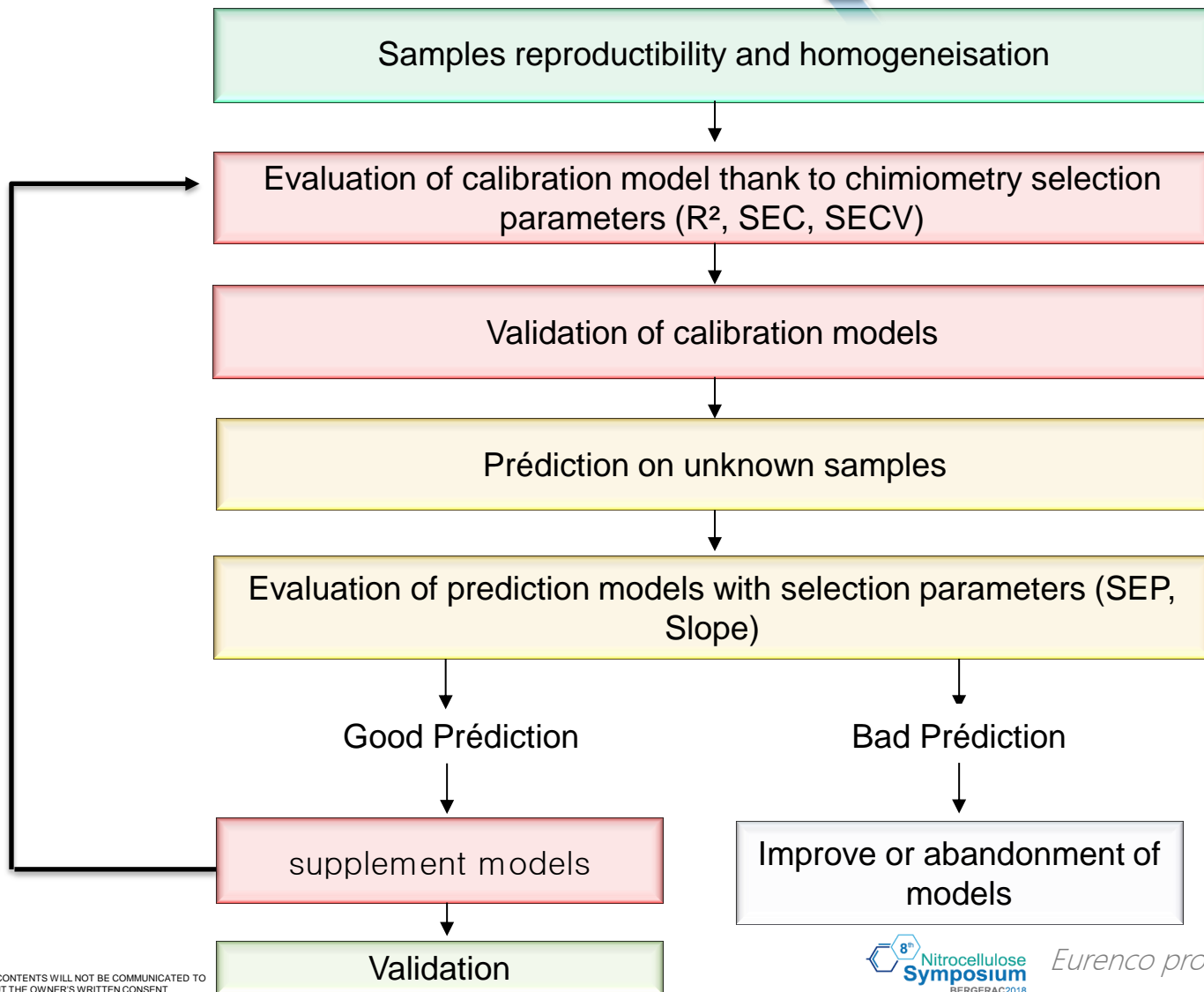


Development of a machine and a method to have a good regularity and reproducibility of:

- Thickness
- Surface condition
- Flatness
- Homogeneity
- Water content



Methodology

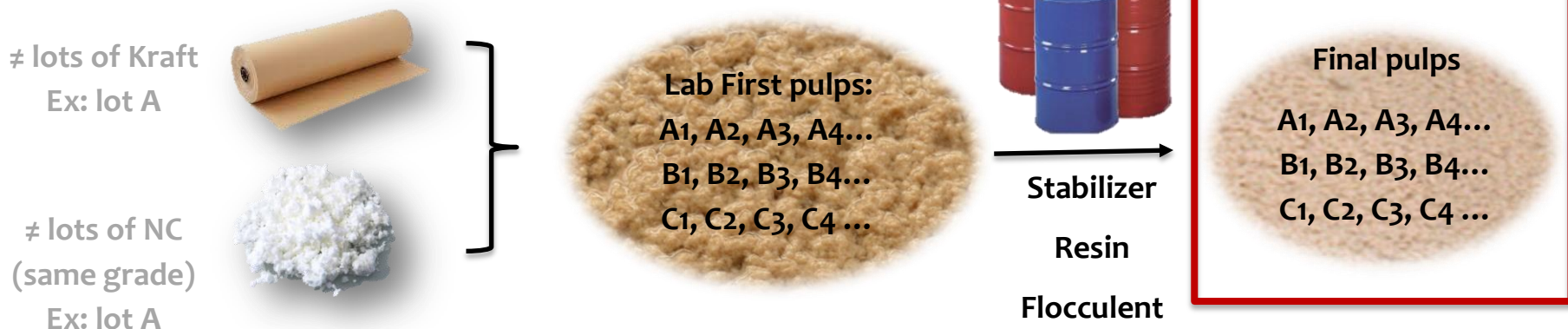




Laboratory scale development of pulps to create a reliable data base for the calibration

Target: Include all variations of the pulp manufacturing process to propose a reliable calibration model

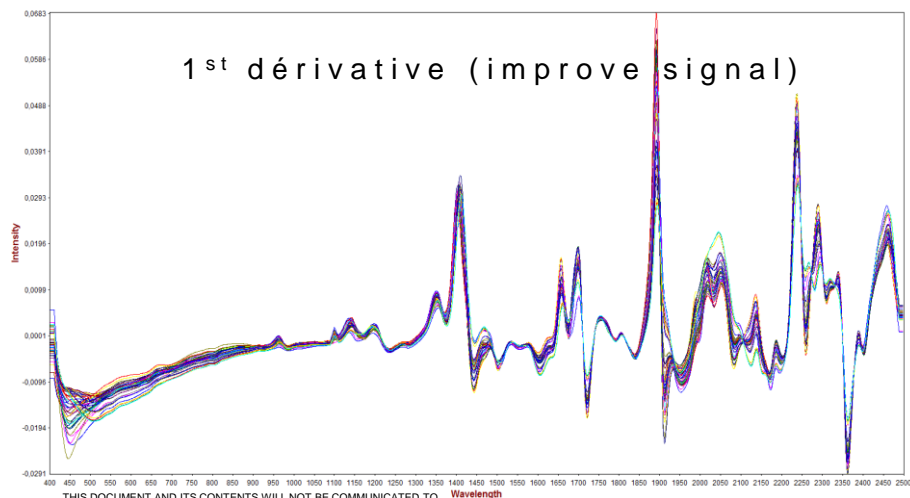
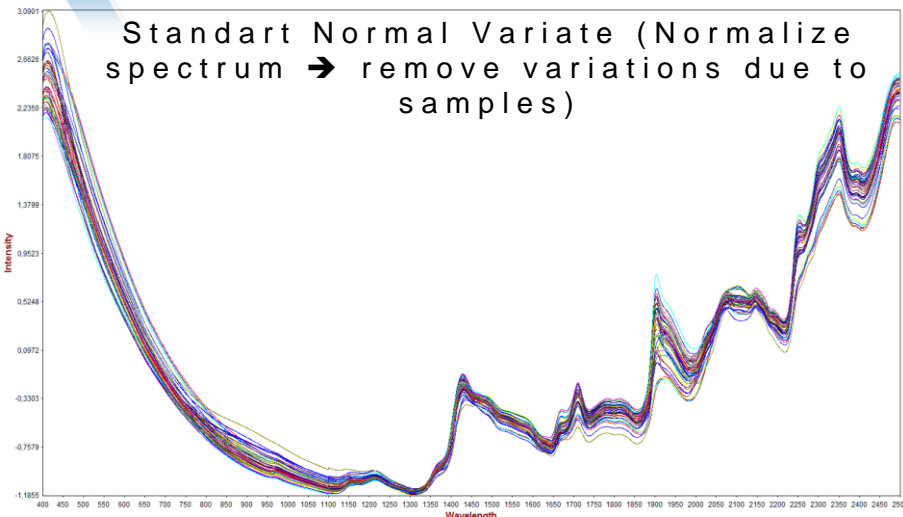
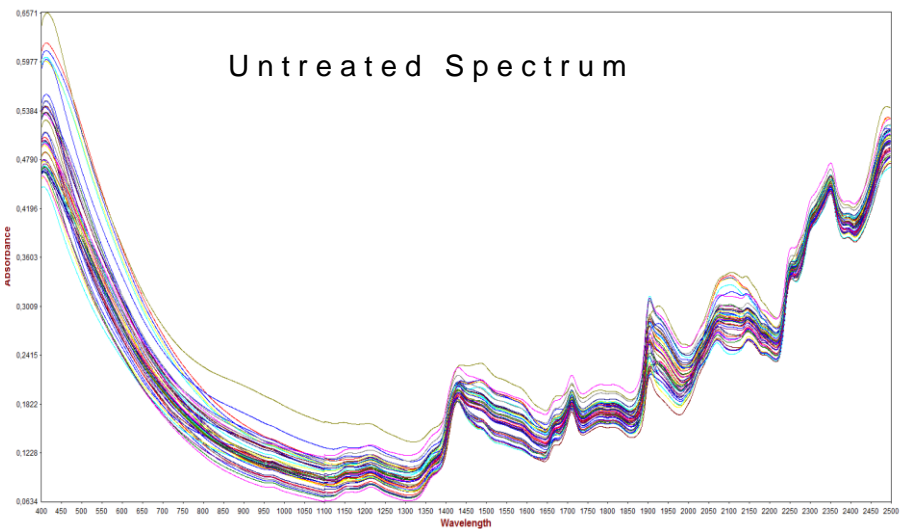
*Ex: Different lots of raw materials,
Different first pulps*



Calibration range of %N for for pulps
6% < %N pulps < 10,5%.



Chimiometric treatment



Modelling Risks :

- Loss of informations
- Non representative models fitted on curves



Quantitative models for determination of %N of final pulps

Calibration samples: 20

	Correlation coefficient (R ²)	Calibration indicaton error (SEC)	Crossed validation indicaton error (SECV)	Prédiction indication error (SEP)
Model 1	0,997	0,087	0,102	0,147
Model 2	0,997	0,079	0,094	0,107
Model 3	0,997	0,083	0,098	0,117
Model 4	0,997	0,085	0,085	0,113

Model acceptance parameters

- SECV < 1,5SEC
- SEP < SEC

SEC measures the gap between predicted values with NIR calibration and reference value of calibration

SEP measures the gap between predicted values with NIR prediction and reference value of prediction



Prediction model unsatisfactory
Need to supplement models with more samples



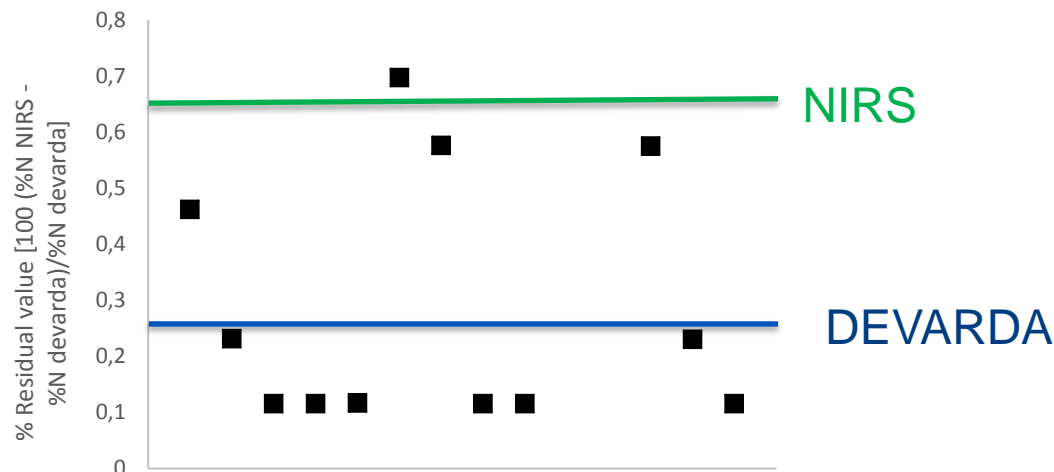
Selection of a calibration model and prediction tests

Calibration samples: 37

	Correlation coefficient (R ²)	Calibration indicator error (SEC)	Crossed validation indicator error (SECV)	Prédiction indication error (SEP)
Model 3	0,992	0,106	0,124	0,052

SEP << SEC

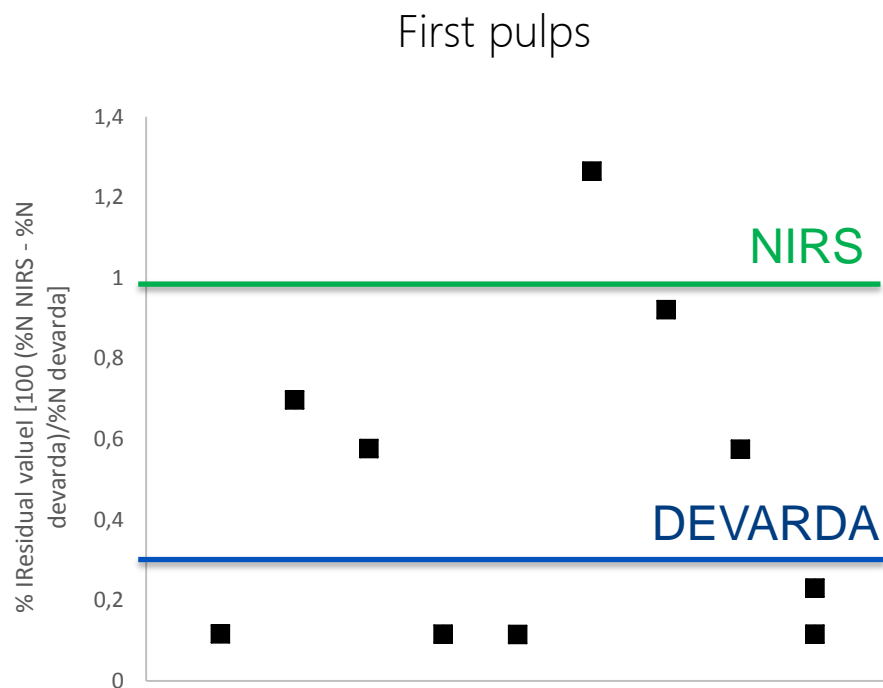
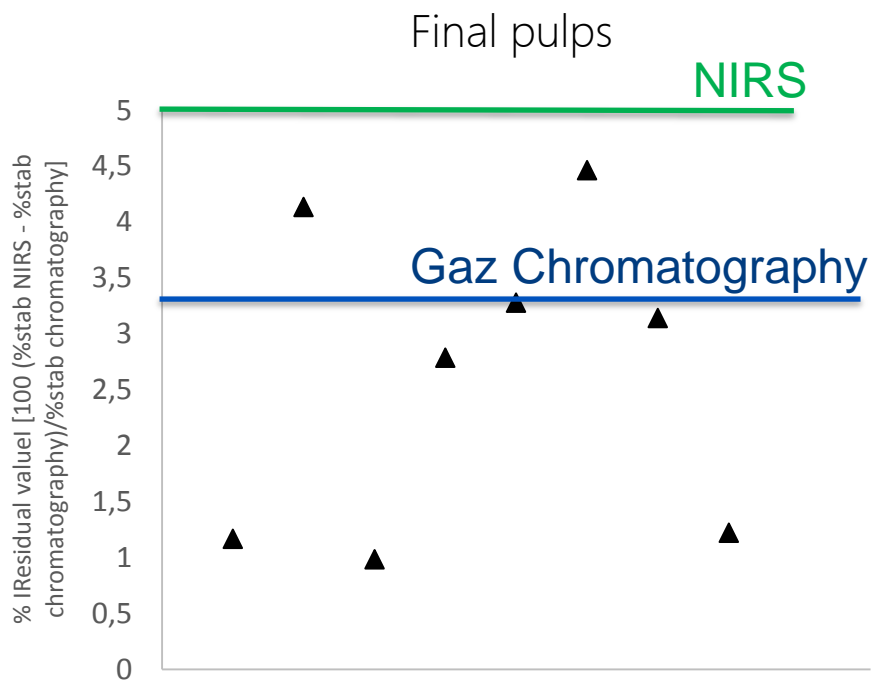
Prediction samples



Prediction model satisfactory
 Need to supplement calibration models in samples to have a better precision



Selection of calibrations models and prediction tests



Prediction model satisfactory for determination of %N and %stabilizer in pulps

Need to supplement calibration models in samples to have a better precision



Reproducibility of samples thank to a disk manufacturing machine



Calibration of %N of first and final pulps
Calibration of % stabilizer of final pulp

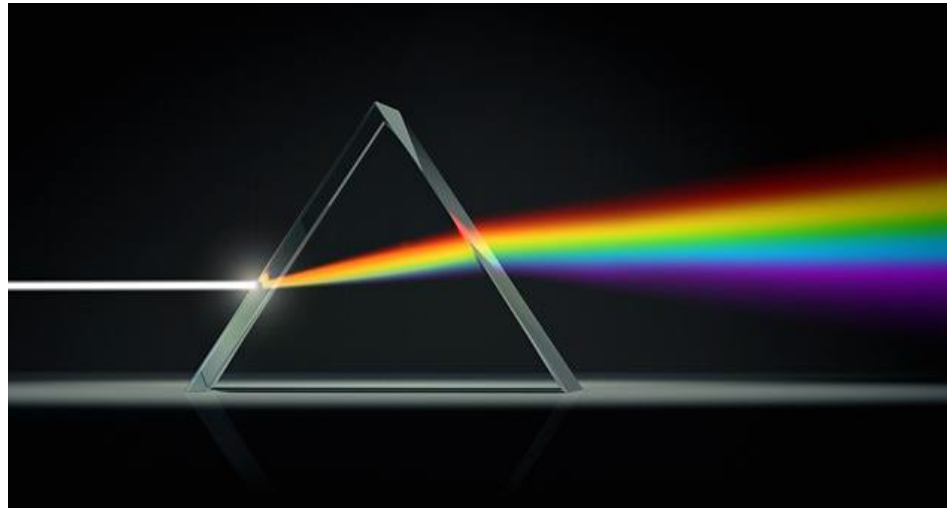


Good prediction



Supplement models in samples to improve the precision

Thanks for your attention



Raw materials



Pulping



Analysis



Nitrogen & stabilizer contents



Production process

Combustible Cartridge Cases

