   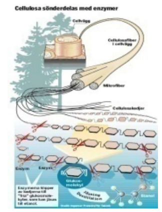  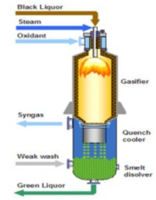 

COST-Action FP0901, Analytical Techniques for Biorefineries, WP2

Biomass characterisation by NIR techniques

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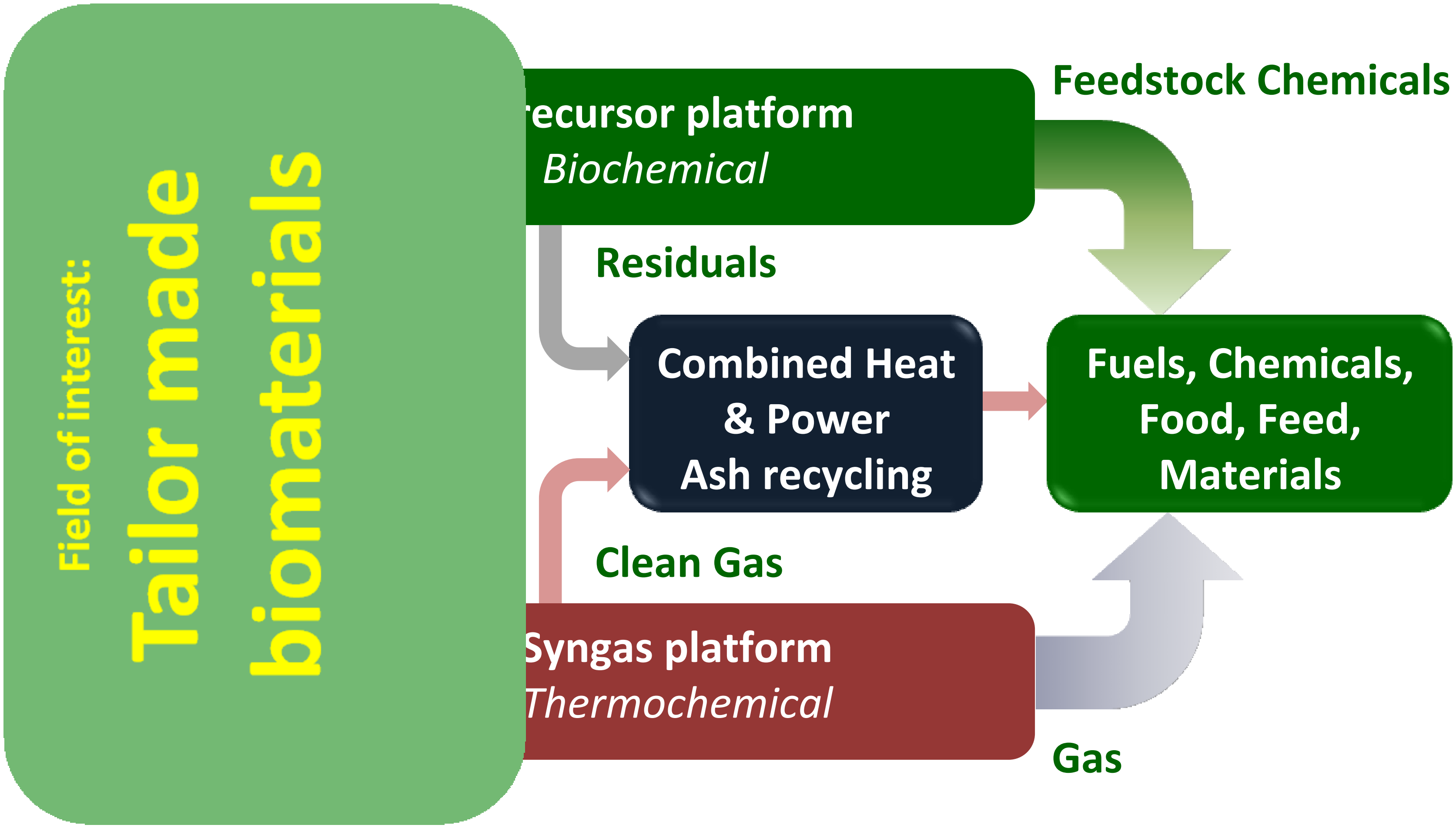
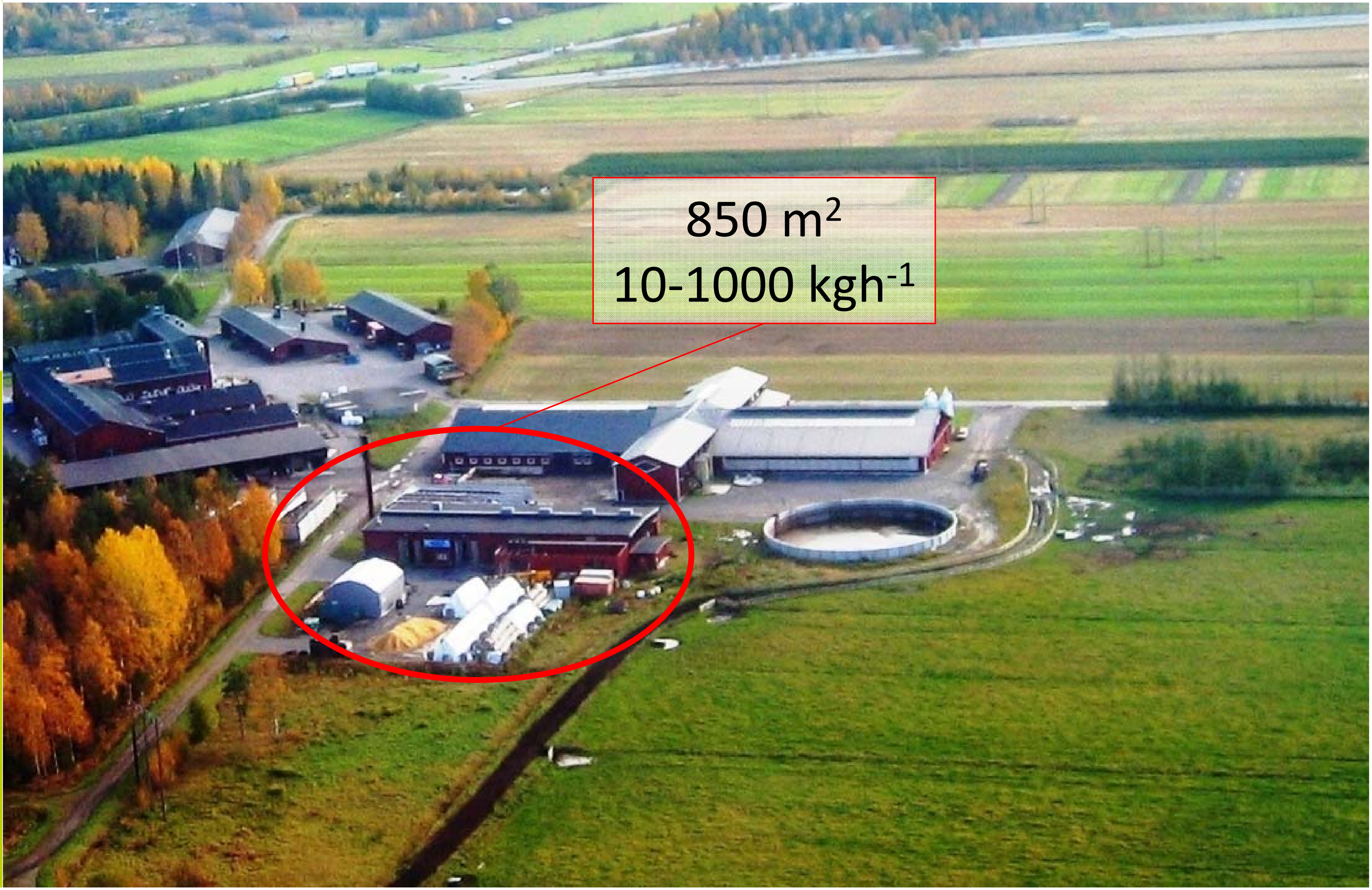
Focus for the SLU BTC research group:

Biorefinery and energy combine concept



SLU BTC Research pilot plant for solid biofuels

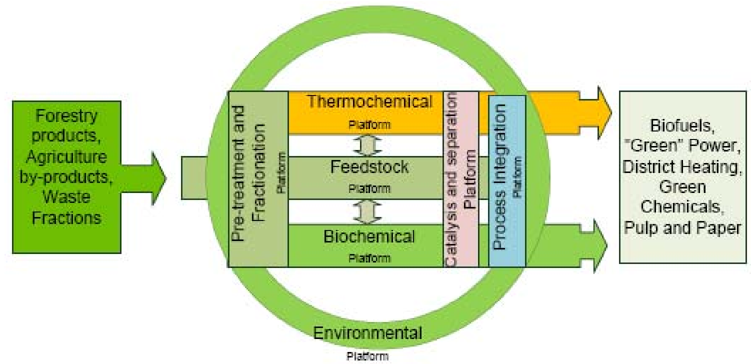


Involvement in some R&D projects

Biofuel Pellet Platform; PI; 2007-2010
- a 4.6 M€ → Objective: widening of raw materials
  and improving of the pelletizing process and biofuel pellet quality

BIO4ENERGY

Bio4Energy – co-PI, 2010-2014
- 20 M€ → Objective: find novel and innovative
  process routes and biorefinery products
- 3 universities cooperate in 8 platforms
- our research group: pretreatment

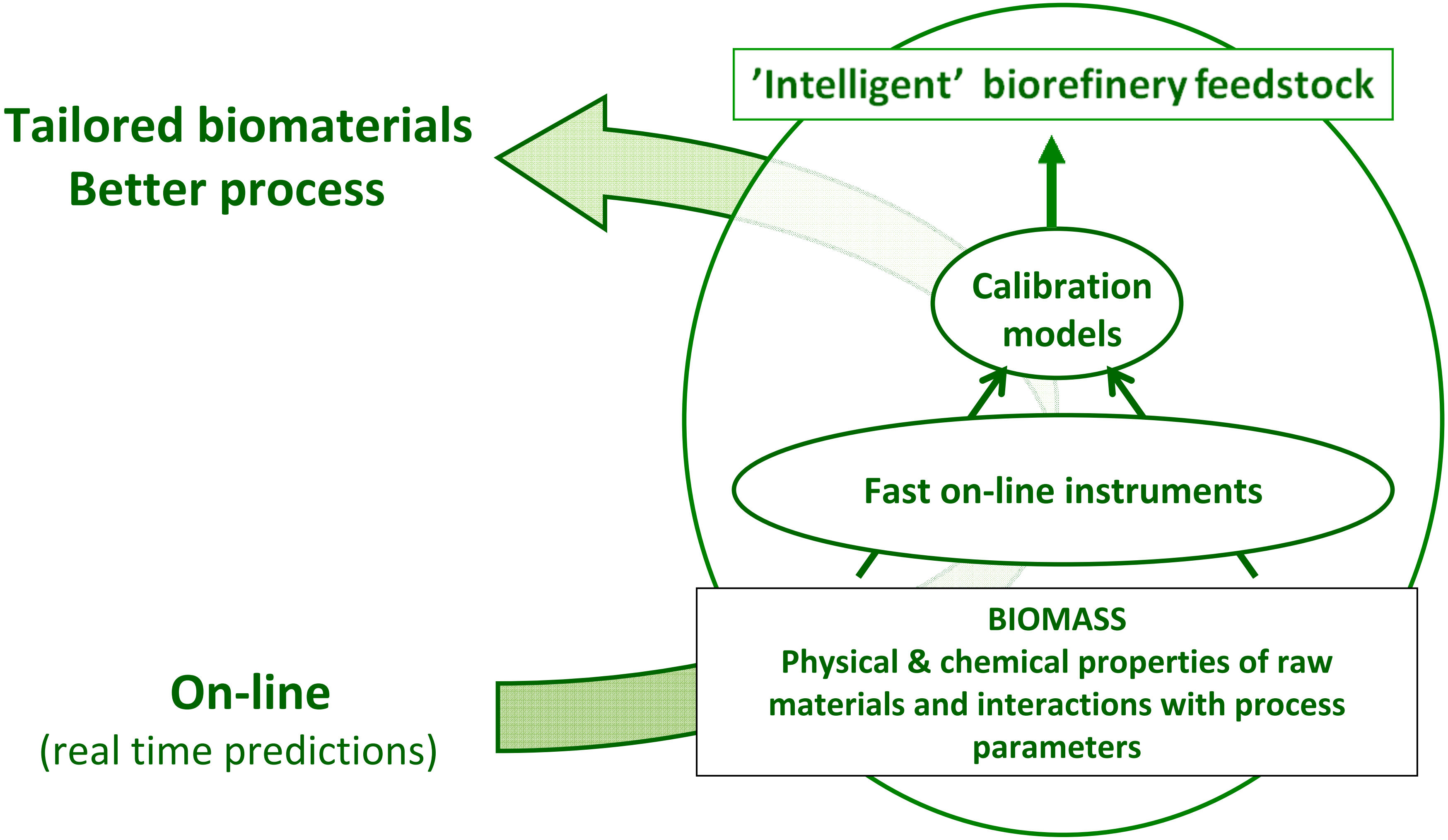


Characterization of ash elements in biofuels - PI, 2009-2012
- 0.4 M€ → Objective: use process analytical tools to predict ash
  elements in biomass.

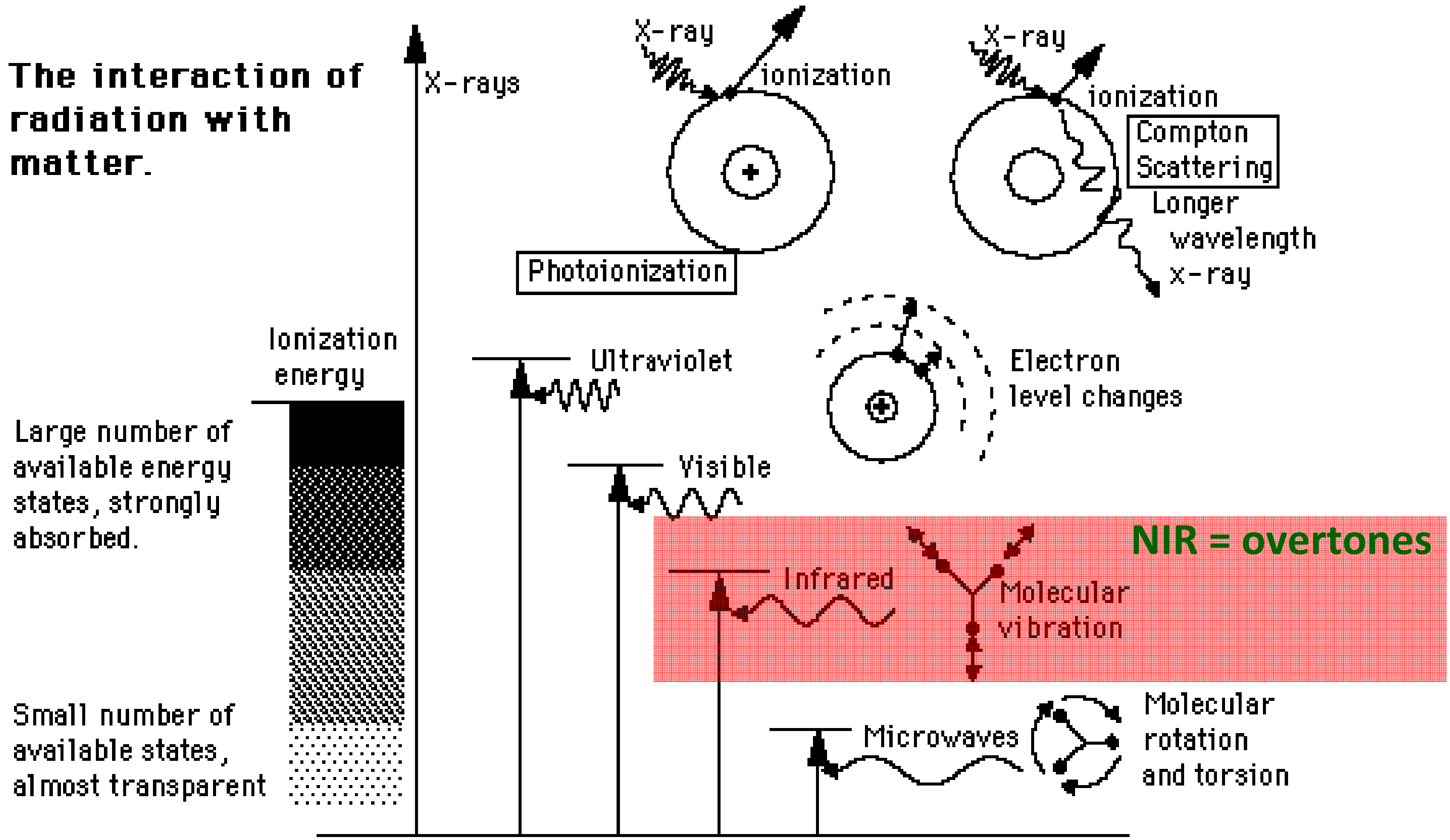
Special interest: On-line characterization of rapid biomass streams

Overall vision for my research group:

’Intelligent’ and uniform biorefinery feedstock

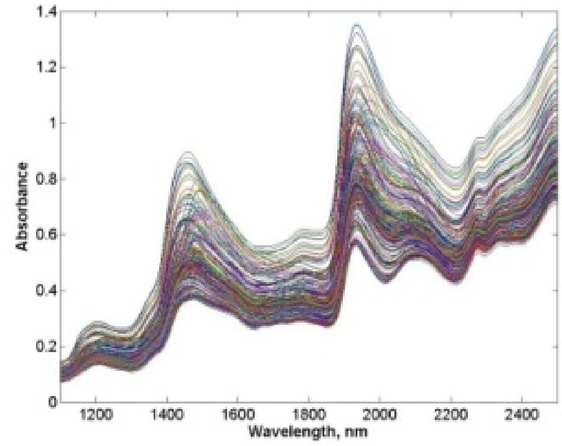


Interaction of radiation with matter → on-line

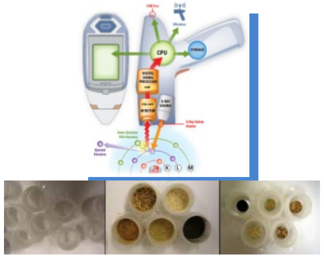


Spectroscopic characterization – inst. mod.: Near Infrared

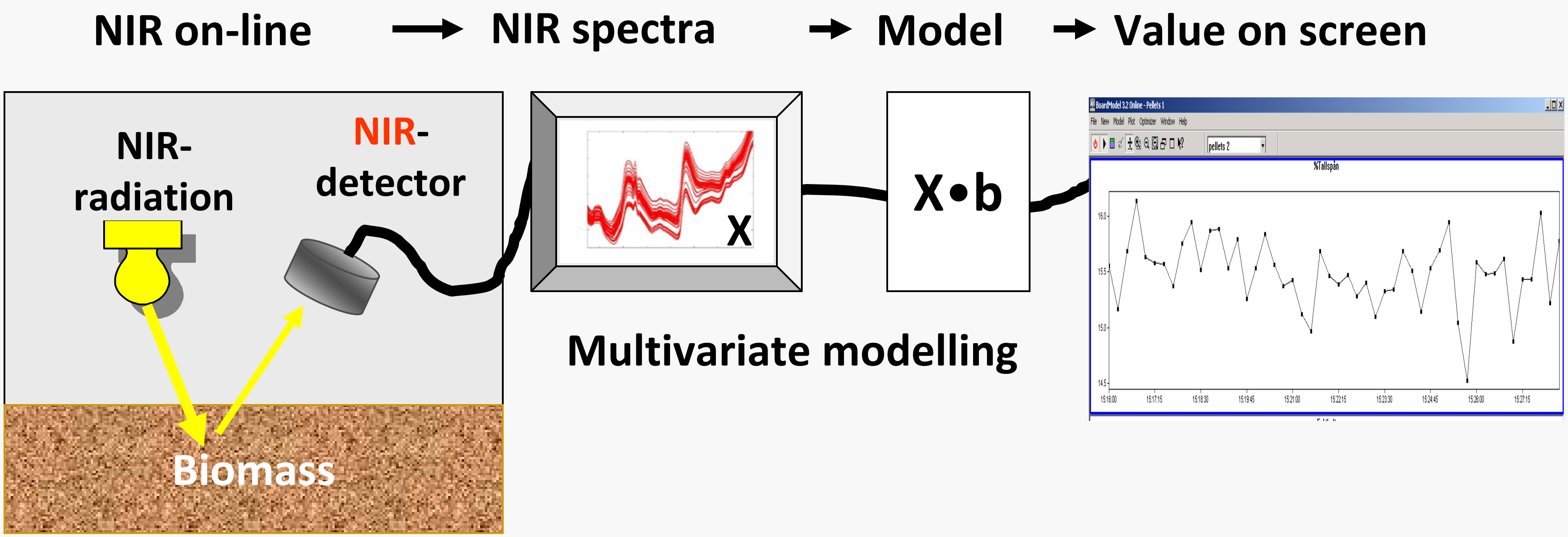
- **organic part of biomass**
- **structural groups: C-H, O-H, N-H, S-H, C=O, C=C,**
  … nearly all bonds except C-C and inorganic
- **simultaneous collection at all wavelengths**
- **256 channels at 5 nm increments 950-1700 nm**
- **100 spectra per second**

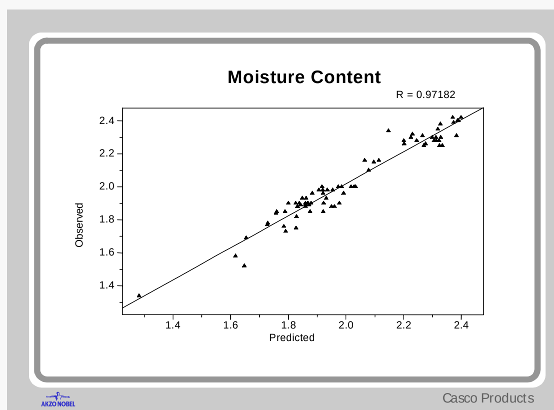


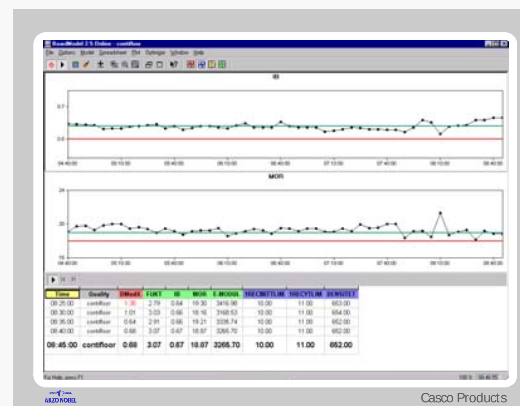
Some instruments available at
the research group:



From spectroscopic signal via screen to process operator







Prediction models based on NIR spectra – example I

Moisture content, ash content and energy content

Excellent models

Table 3 Overview of results in BPLS modelling of moisture content, ash content and gross calorific value

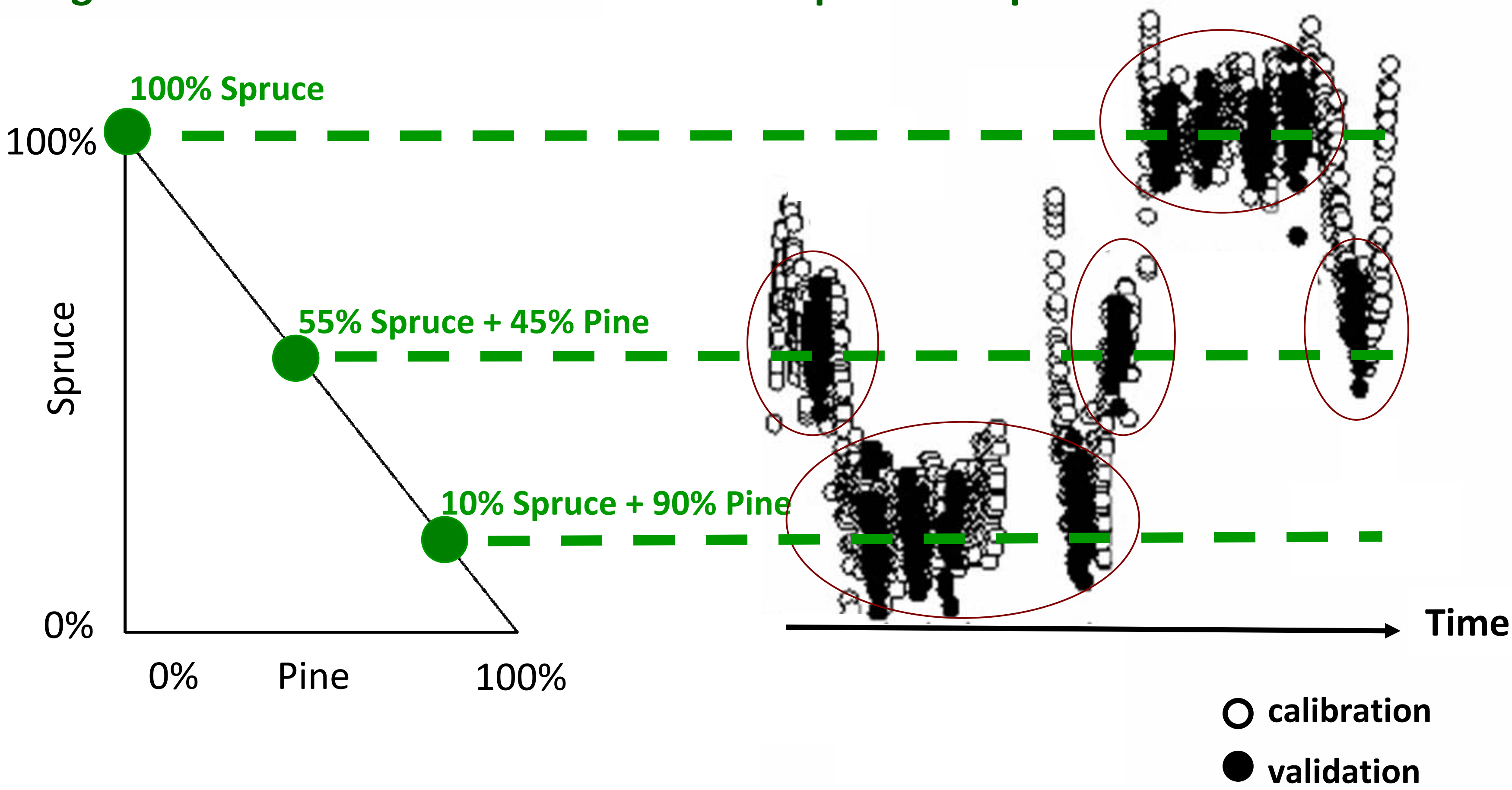
Reference variable	Model	Wavelength region/nm	Rank #	RMSEE	RMSEP	Q^2	Bias
Moisture % fw min: 9.4 max: 50.0	DIR-BPLS	780–2498	4	0.973	0.726	0.997	0.035
Moisture content	DIR-BPLS	1400–2400	4	0.980	0.827	0.997	−0.098
	OSC-BPLS	780–2498	4	0.956	0.712	0.998	0.030
	OSC-BPLS	1400–2400	4	0.968	0.793	0.997	−0.065
Ash % dw min: 0.3 max: 2.2	DIR-BPLS	780–2498	6	0.108	0.077	0.992	0.023
Ash content	DIR-BPLS	1400–2400	6	0.119	0.113	0.982	0.002
	OSC-BPLS[a]	780–2498	5	0.108	0.077	0.992	0.022
	OSC-BPLS	1400–2400	3	0.136	0.099	0.986	0.004
	DRY-BPLS	780–2498	6	0.120	0.101	0.986	0.021
	DRY-BPLS	1400–2400	8	0.167	0.108	0.983	0.000
Gross calorific value kJ g^{-1} dw min: 20.2 max: 21.7	DIR-BPLS	780–2498	5	0.090	0.100	0.971	−0.042
Energy content	DIR-BPLS	1400–2400	9	0.078	0.109	0.967	−0.020
	OSC-BPLS	780–2498	4	0.090	0.100	0.972	−0.037
	OSC-BPLS	1400–2400	3	0.091	0.112	0.961	−0.051
	DRY-BPLS	780–2498	3	0.114	0.100	0.972	−0.020
	DRY-BPLS	1400–2400	6	0.146	0.128	0.954	−0.020

[a] Unit variance scaling of reference variable.

Lestander, T.A. and Rhén, C. 2005. Multivariate NIR spectroscopy models for moisture, ash and calorific content in biofuels using bi-orthogonal partial least squares regression. ***Analyst***, 130, 1182-1189.

Prediction models based on NIR spectra – example II

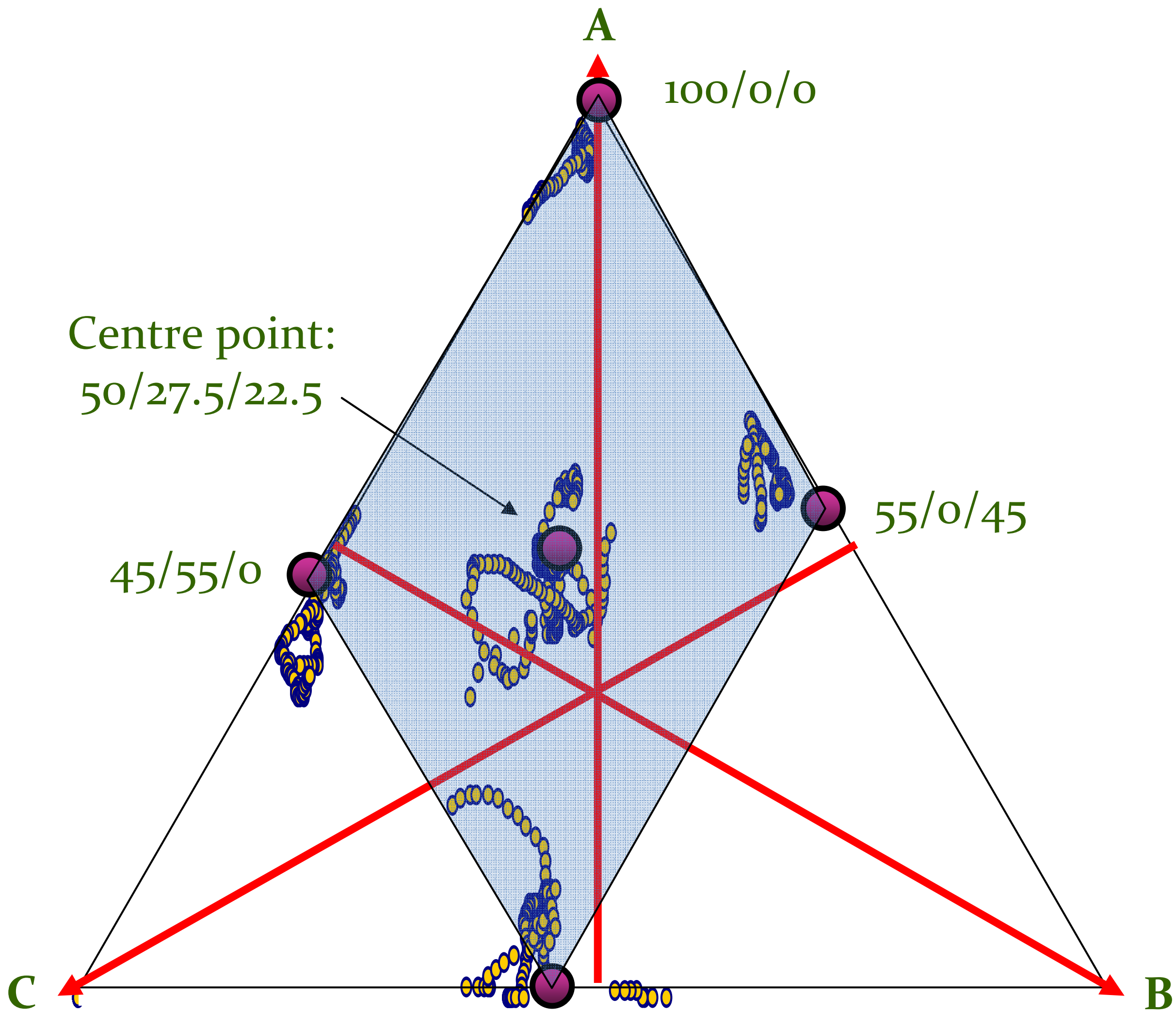
Origin of biomaterial in biomass stream – pine and spruce



Lestander T.A., Johnsson B. & Grothage M. 2009. NIR techniques create added values for the pellet and biofuel industry. ***Bioresource Technology*** 100, 1589-1594.

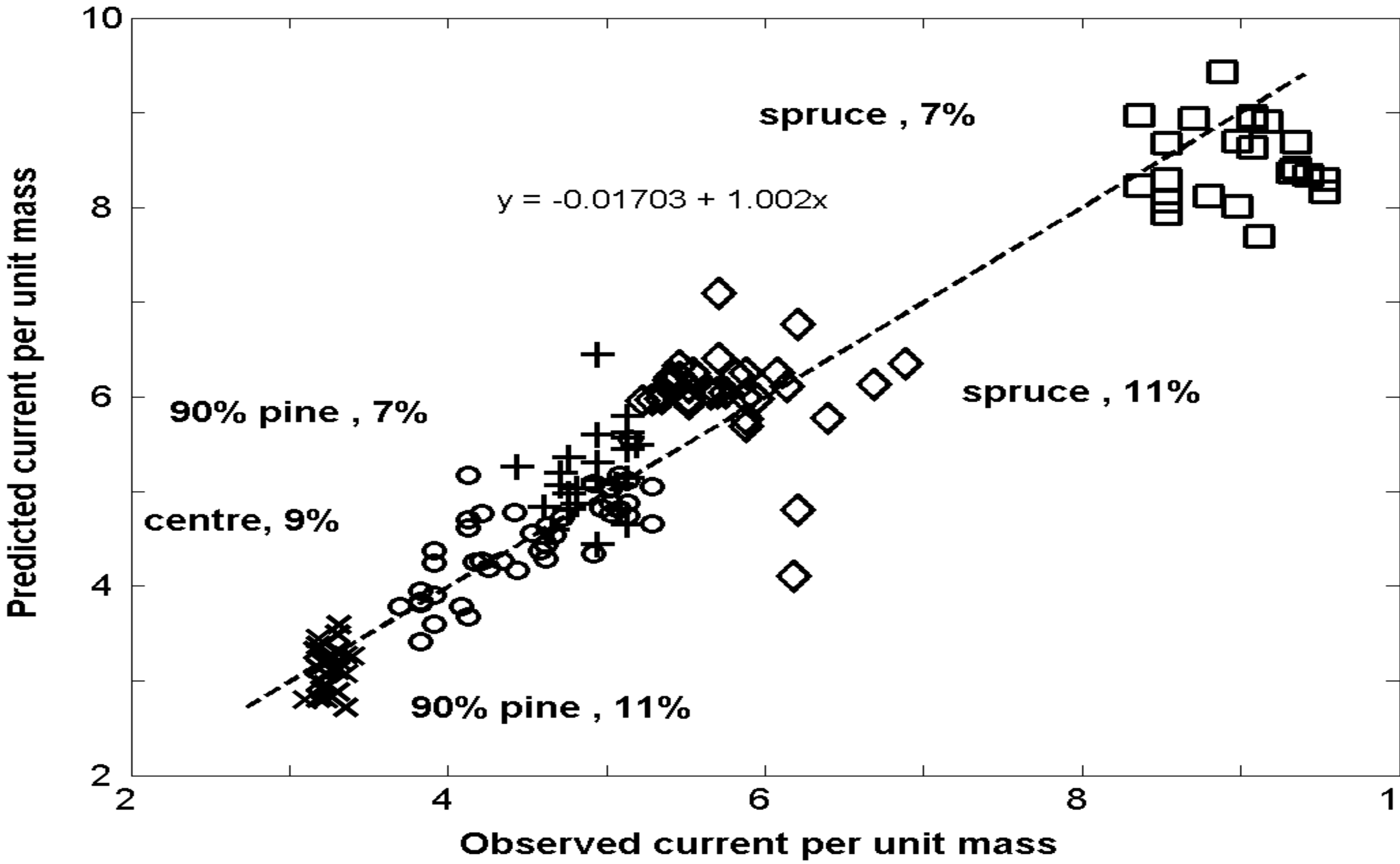
Prediction models based on NIR spectra – example III

Designed industrial experiment for blends of 3 sawdust types



Prediction models based on NIR spectra – example IV

Influence of biomass properties on electrical consumption in a process



Lestander T.A., Johnsson B. and Grothage M. 2009. NIR techniques create added values for the pellet and biofuel industry. ***Bioresource Technology*** 100, 1589–1594.

Prediction models based on NIR spectra – example V

Characterization of wood properties - softwood

Parameter	RMSEP	Q^2
Modulus of elasticity	1476.80	**0.830**
Bending strength	12.30	**0.778**
Compression strength	4.34	**0.739**

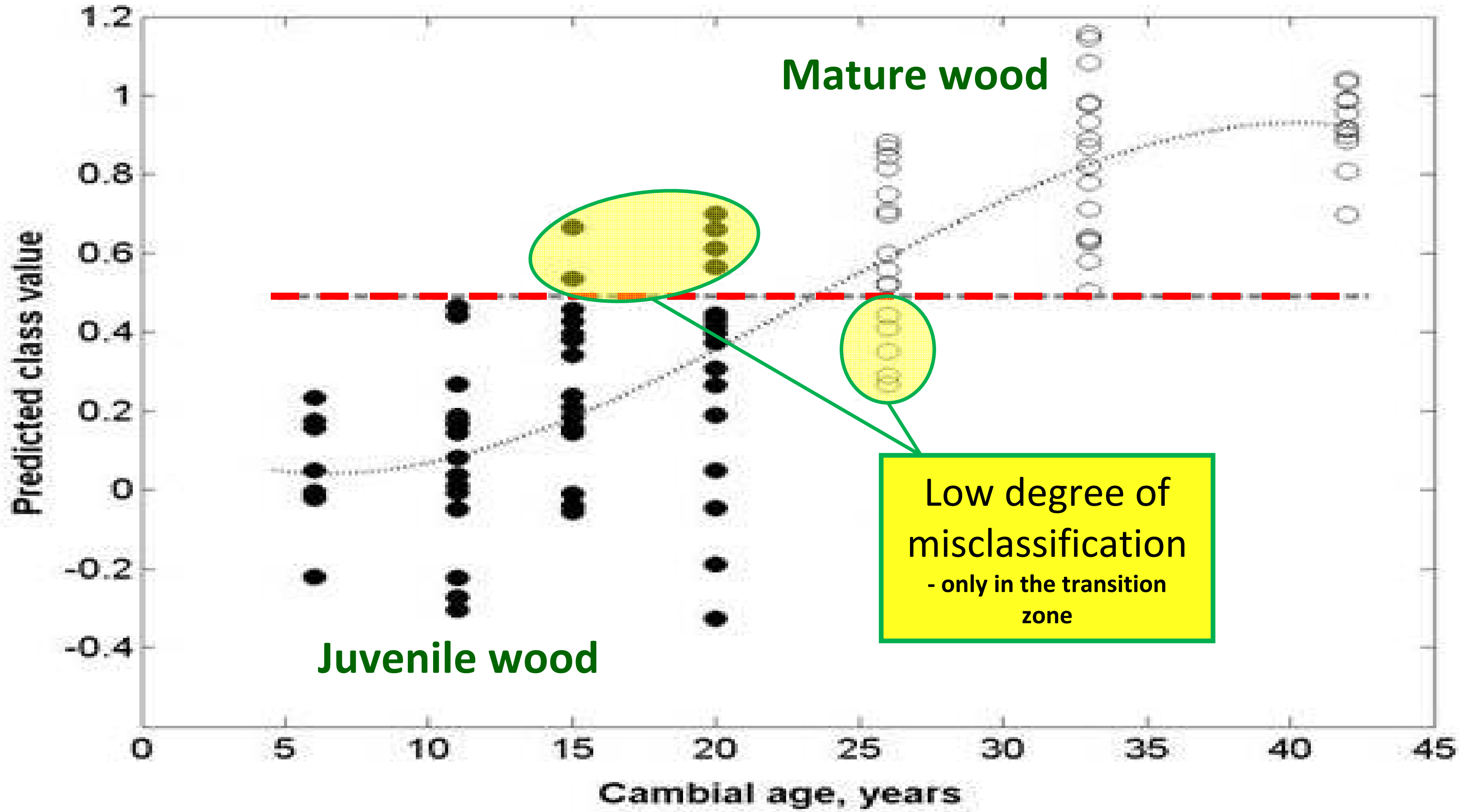
Good prediction models

RMSEP: root mean squared error; Q^2: explained variation in test set.

Lestander TA, Lindeberg J, Eriksson D and Bergsten U. 2008. NIR spectroscopy for prediction of clear-wood properties in Scots pine using bi-orthogonal partial least squares regression. ***Canadian Journal of Forest Research*** 38, 2052-2062.

Prediction models based on NIR spectra – example VI

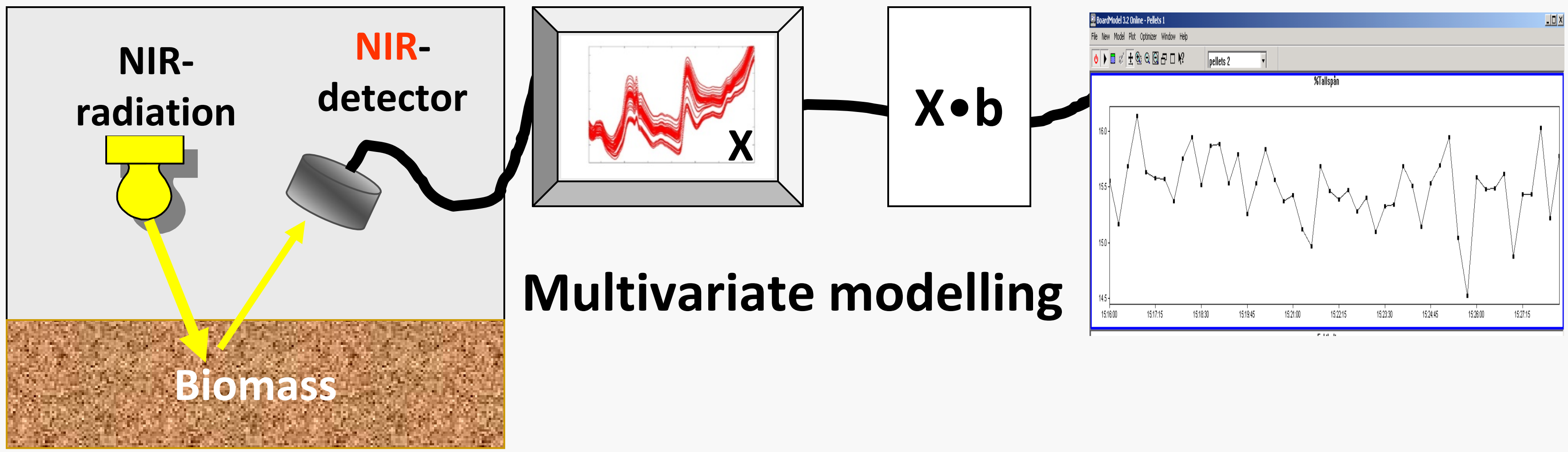
Classification of juvenile and mature wood

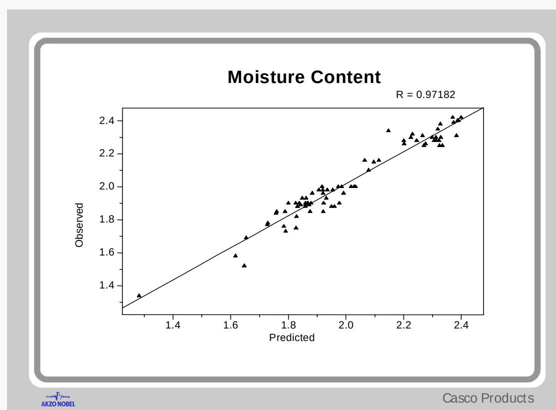


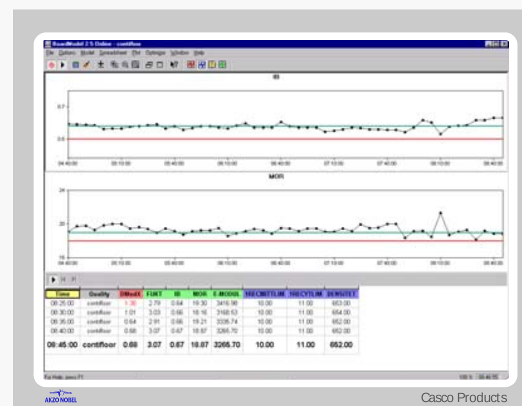
Lestander TA, Lindeberg J, Eriksson D and Bergsten U. 2008. NIR spectroscopy for prediction of clear-wood properties in Scots pine using bi-orthogonal partial least squares regression. ***Canadian Journal of Forest Research*** 38, 2052-2062.

From NIR signal via screen to process operator

NIR on-line → NIR spectra → Model → Value on screen







Multivariate modelling - PCA

- Principal component analysis (PCA)

  - only a X matrix

  -e.g. only chemical data, spectral data

  → data overview

  → interpreting of model

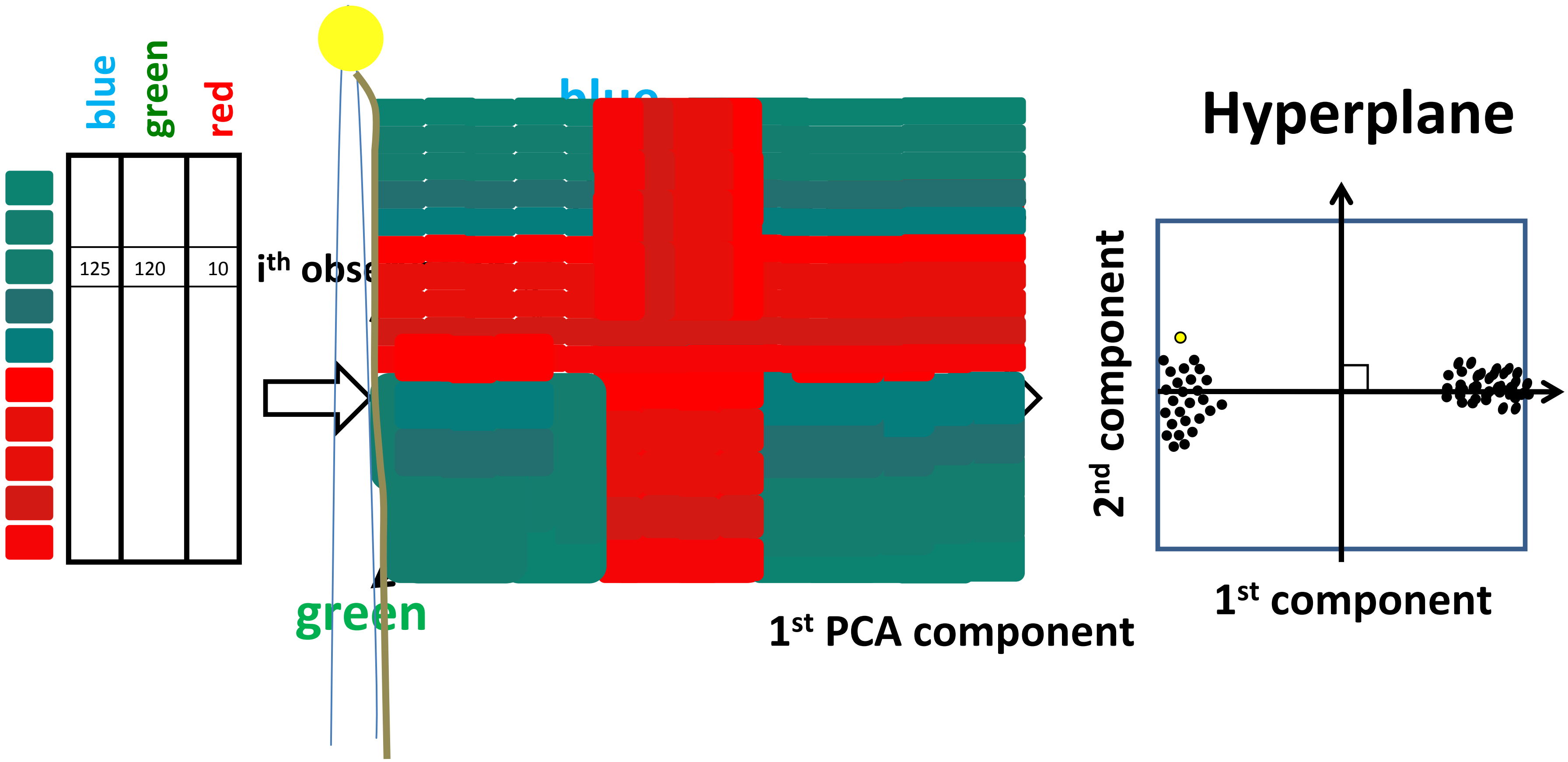
Multivariate modelling - PLS

- **Partial least squares (PLS) regression**

  - **data from both y vector and X matrix**
  - **e.g. observed product quality and chemical data**
  - **e.g. observed chemical values and NIR data**

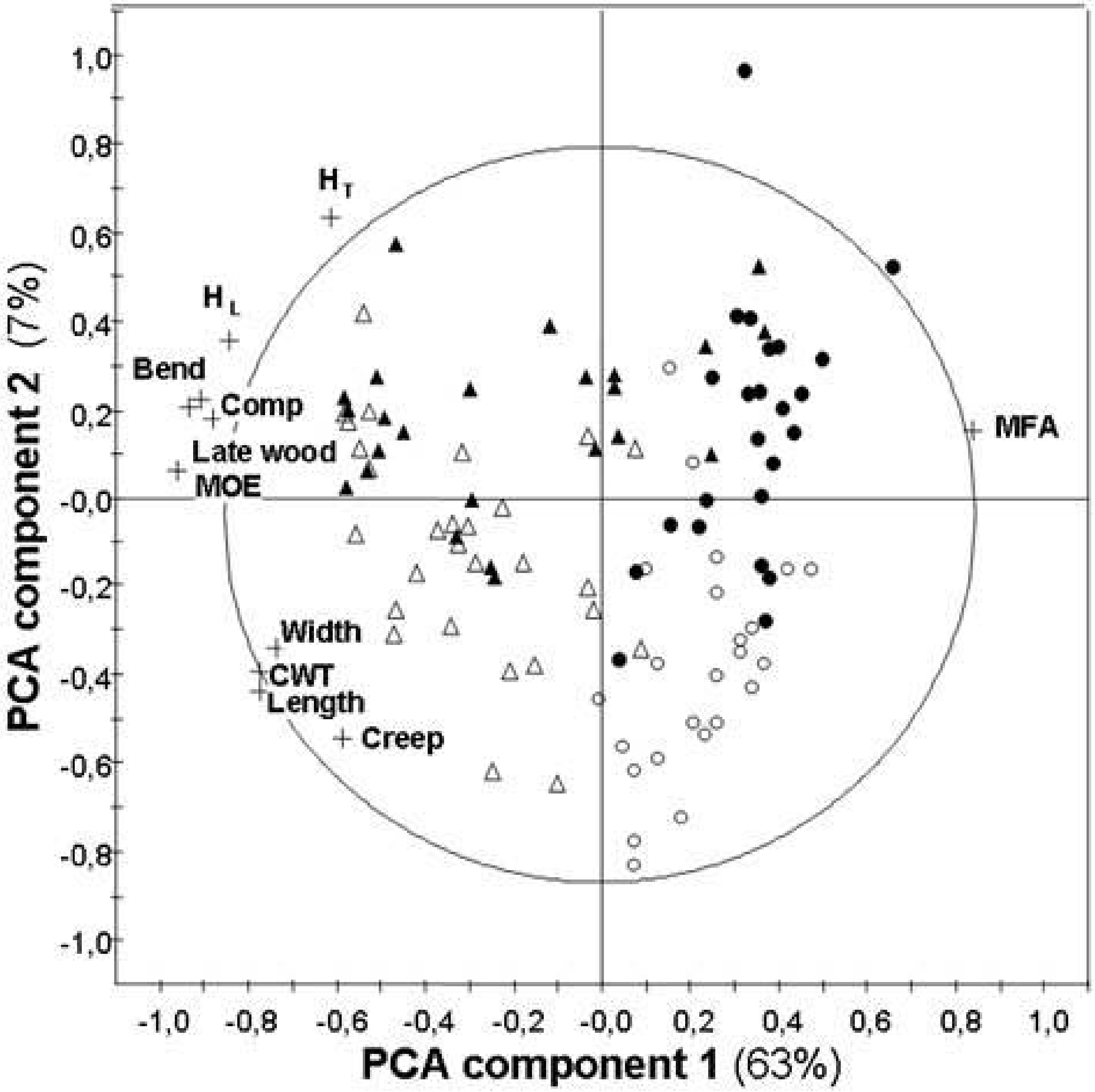
  → **calibration model (quantitative or qualitative)**
  → **interpreting of model**

The PCA algorithm – variation in X matrix



wood properties

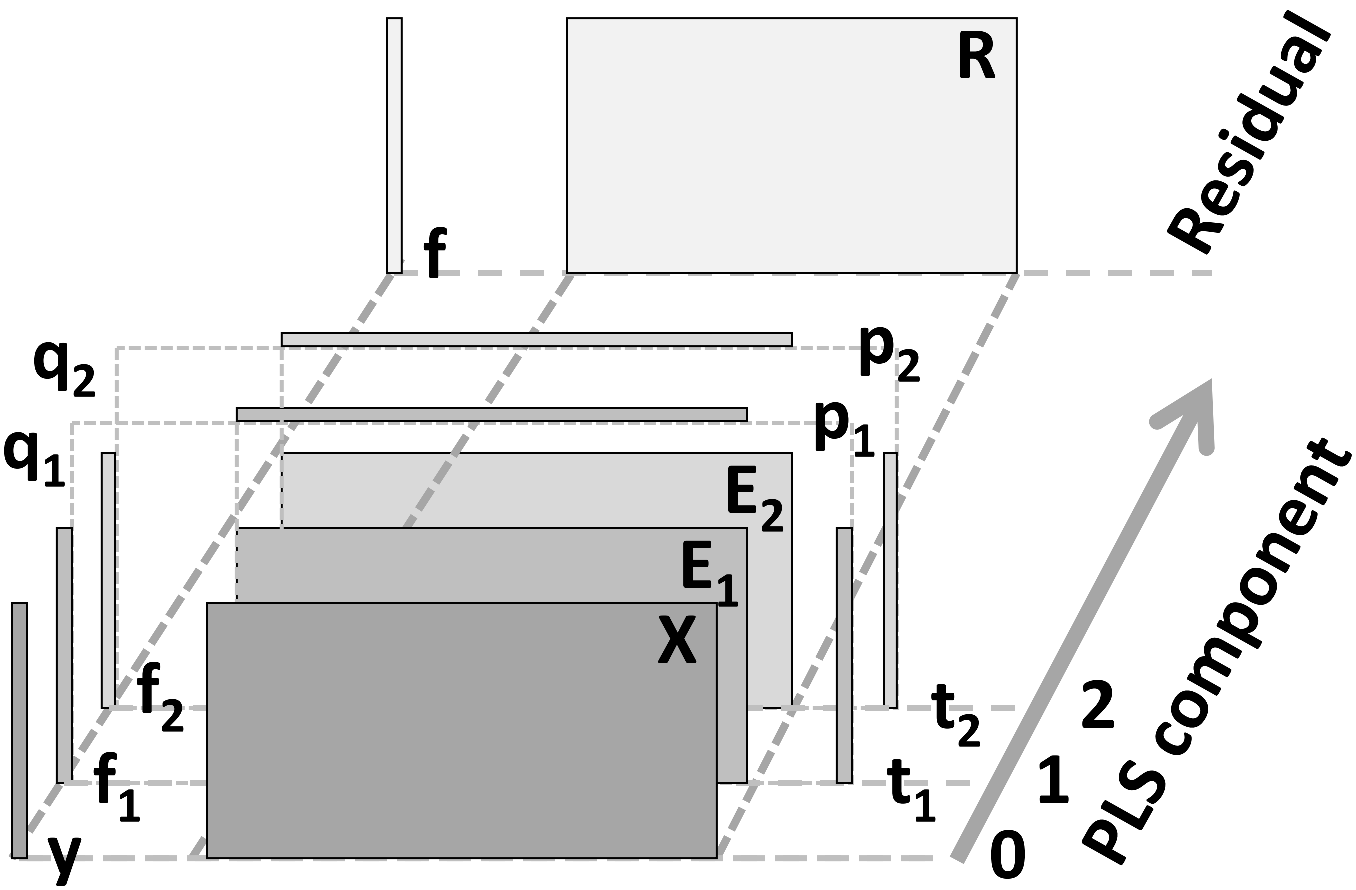
Example: PCA components (loadings and scores)



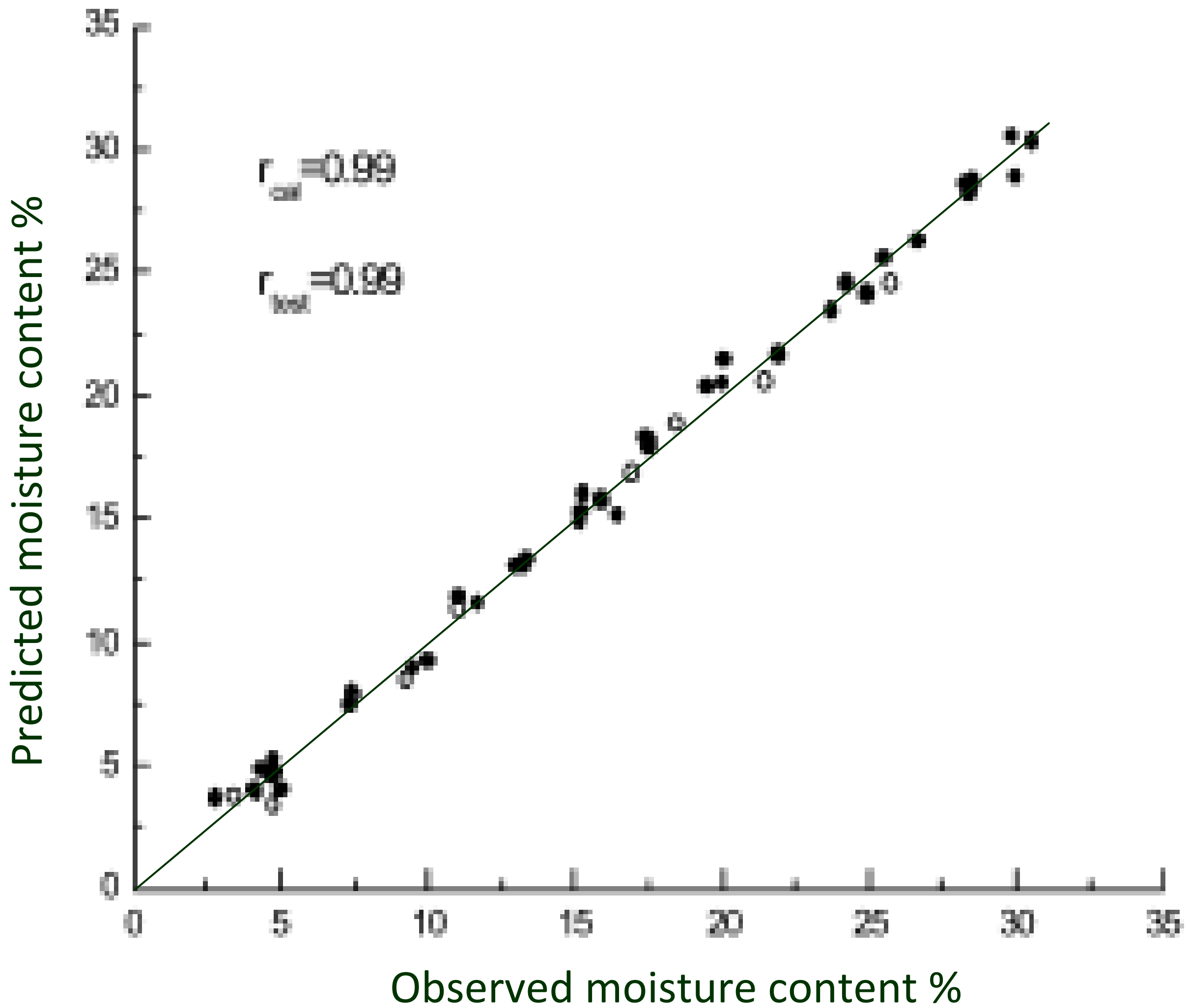
Bi-plot *of the first two loading (+) and score (1st site: ▲ base, △ top; 2nd site: ● base, ○ top) components of a PCA model explaining 70% of the variation. The percentage of explained variation is given within parenthesis. Bend: bending strength; Comp: compression strength; CWT: cell wall thickness;* H_L *and* H_T*: Brinell hardness in longitudinal and tangential direction; MFA: microfibril angle; Length: cell length; Width: cell width.*

Lestander TA, Lindeberg J, Eriksson D and Bergsten U. 2008. NIR spectroscopy for prediction of clear-wood properties in Scots pine using bi-orthogonal partial least squares regression. ***Canadian Journal of Forest Research*** 38, 2052-2062.

The PLS algorithm – variation in X and y

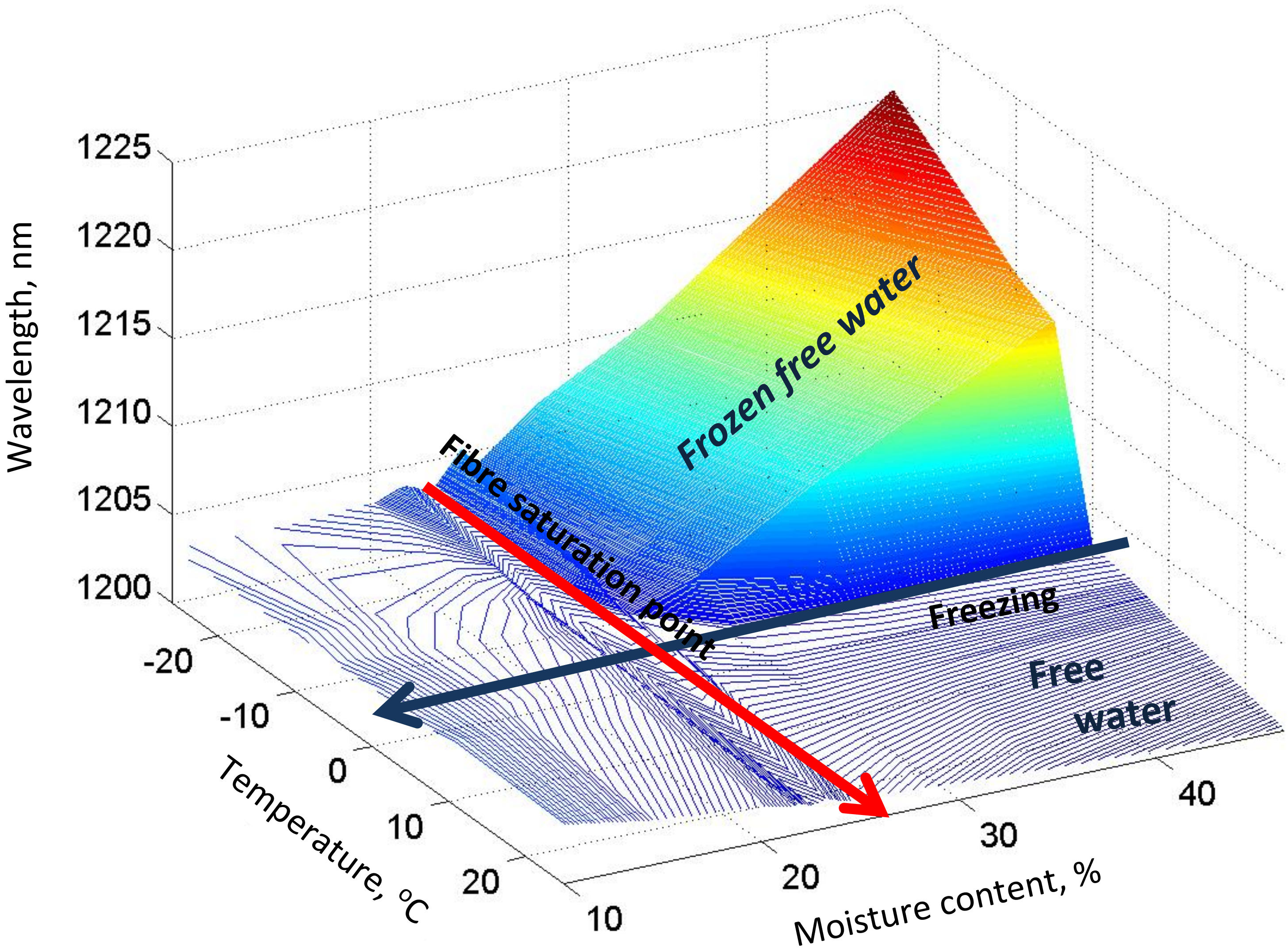


Example: PLS modelling of NIR spectra – moisture content



Lestander, T.A. and Geladi, P. 2003. *Analyst*, **128**, 389-396.

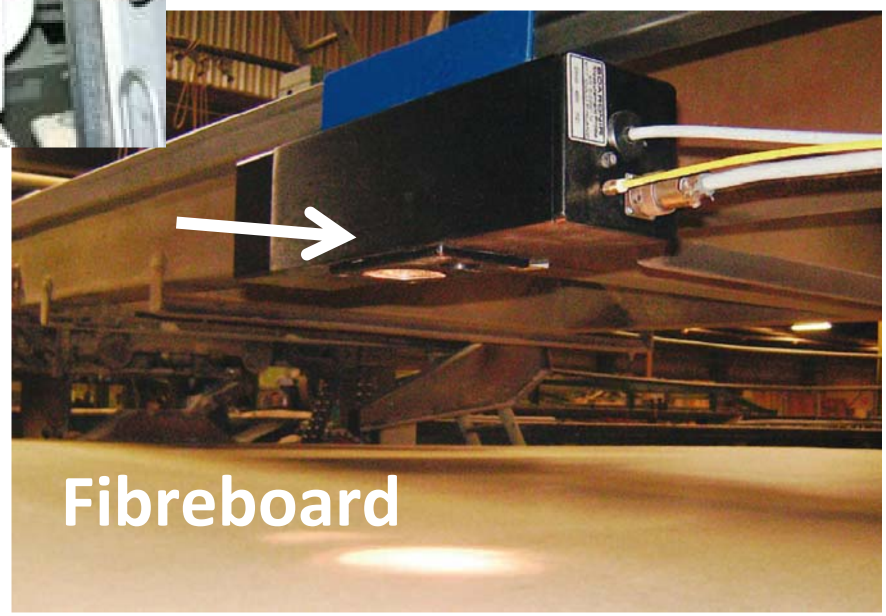
Crystalline, amorphous and free water in biomass



Lestander T.A. Hedman B., Funkquist J., Lennartsson A., Svanberg M. 2008. On-line NIR-fukthaltsmätning för styrning av panna i värmekraftverk. Värmeforsk Service AB, Stockholm. I6-605, ISSN 1653-1248, 70 pp.

NIR – industrial installations

NIR – industrial installations

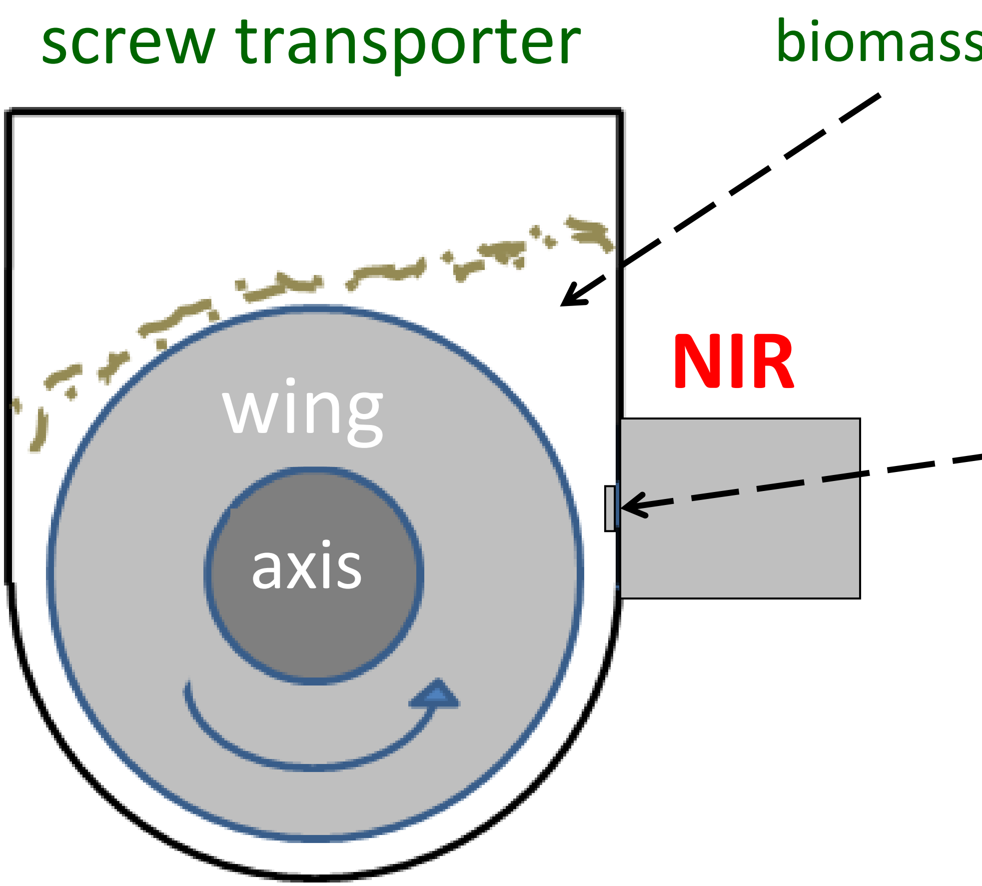


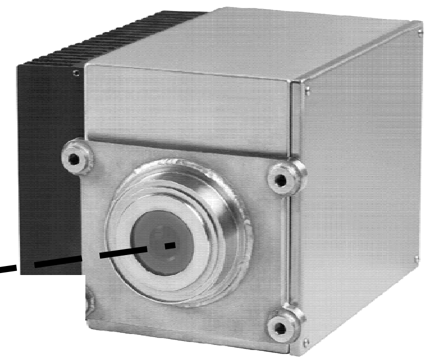

detector


detector


instrument
via by-pass

Example: Positioning of NIR instrument





the NIR instrument was placed on a screw transporter

On-line tested in industrial environment (fuel pellet industry)

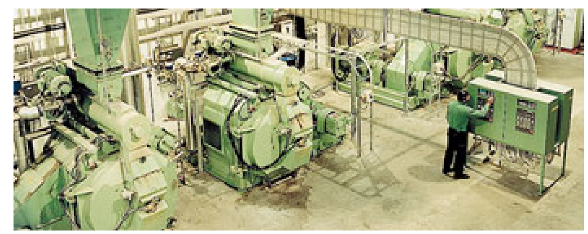
Partitioning – maintains stabile flow of biomass in the process

NIR ↓

Drying – improves storability

NIR ↓

Grinding – improves press-ability and combustion properties



NIR ↓

Pressing – increases energy density

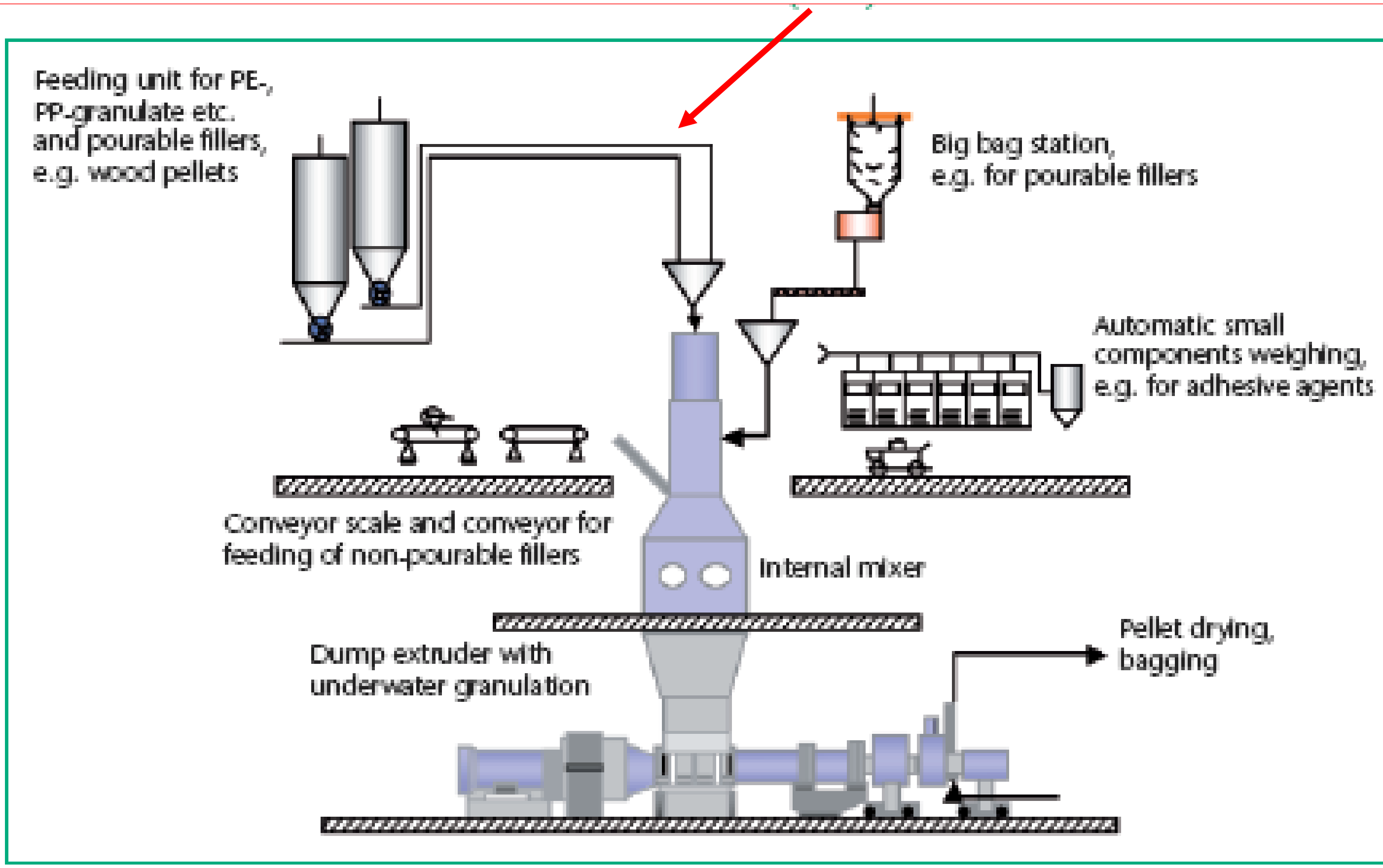
↓

Pellets – reduces costs for transport, handling and storage

NIR

Example of future application: Natural Fibre Plastics

Spectroscopic on-line characterization of e.g. pourable wood fillers



Biomass technology and NIR

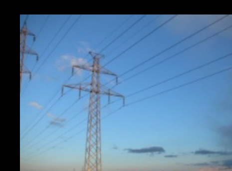




BIOMASS ➡ NIR - a tool ➡ Biofuels / Products / Electricity, heat

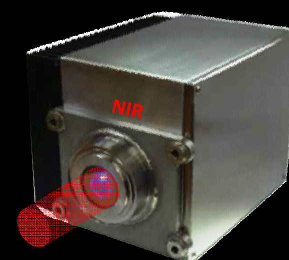




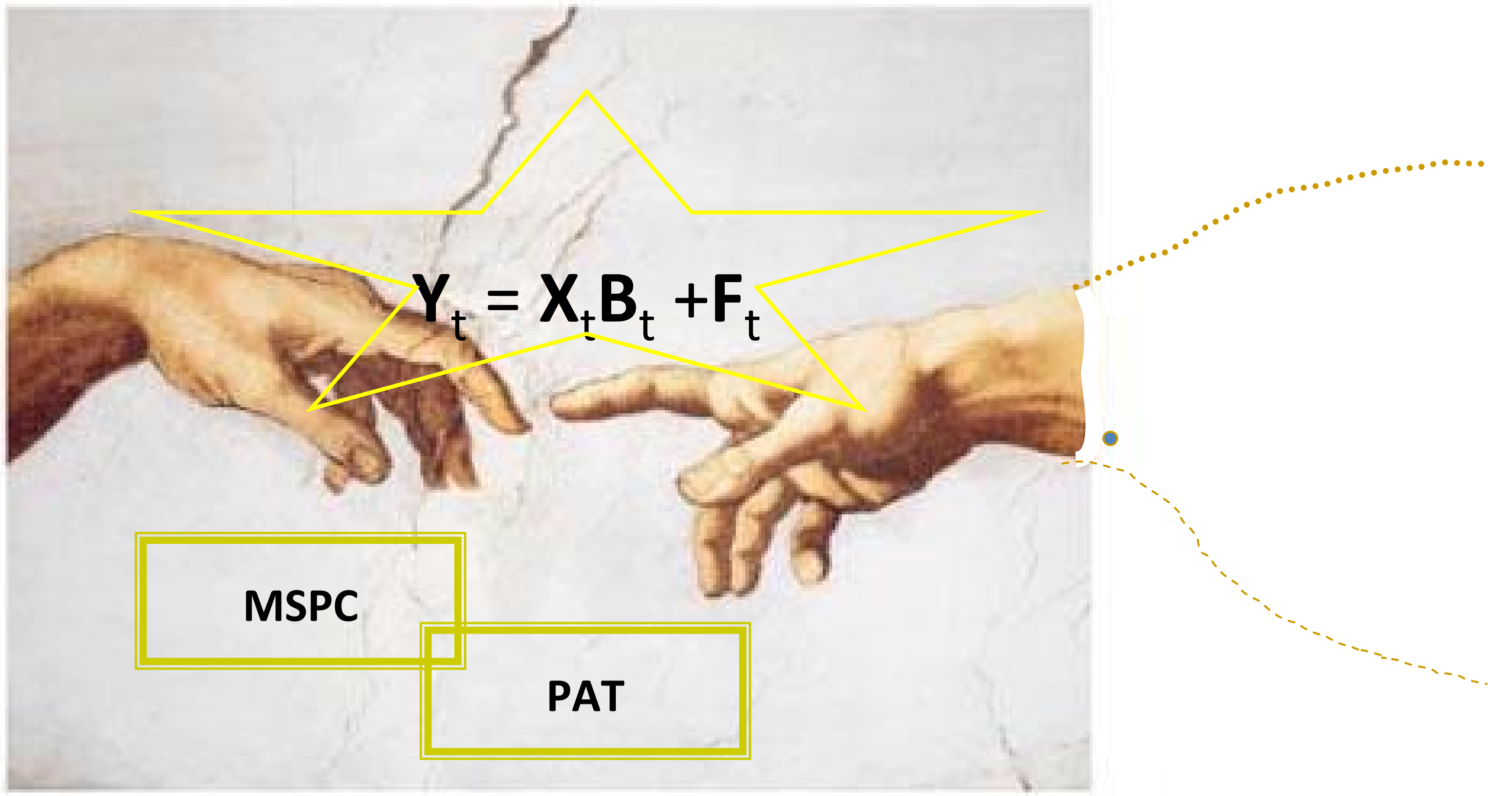


Exemples of questions in real time:

- Origin of the biomass
  - species, region, freshness, etc
- Contents in the biomass
  - moisture, calorific value, C-6 sugars, etc
- Modifications of biomass in processes
  - degree of modification, energy consumption, etc
- Process control



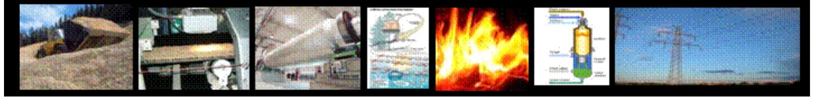
Real time measurements … reaching beyond



Thanks

… to colleges at SLU Biomass Technology and Chemistry
… Swedish Energy Agency
… Kempe Foundations
… Swedish Pellet Association

Some recent papers



On-line NIR technique for biorefinery process monitoring

Torbjörn A. Lestander[1], Robert Samuelsson[1], Michael Finell[1], Mehrdad Arshadi[1] and Bo Allvin[2]

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ELSEVIER

NIR techniques create added values for the pellet and biofuel industry

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Process analytical technology (PAT)

ABSTRACT

A 2^3-factorial experiment was carried out in an indust
feedstock. The aim was to use on-line near infrared (
of moisture content, blends of sawdust and energy
were: drying temperature and wood powder dryness
and Scots pine.
The main results were excellent NIR calibration mo
binary blends of sawdust from the two species, but
electrical energy per unit pelletized biomass can be
entering the pellet press. This power consumption i
that NIR data contained information of the compressi
The moisture content model was validated using a ru
shown that the adjusted prediction error was 0.41% m
12% moisture content. Further, although used drying
drying temperature resulted in low prediction accura
The results show that on-line NIR can be used as an
pelletizing process and that the use of NIR technique
meet customer specifications, and therefore create a

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BIORESOURCE TECHNOLOGY

High quality biofuel pellet production from pre-compacted low density raw materials

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Water absorption thermodynamics in single wood pellets modelled by multivariate near-infrared spectroscopy

Torbjörn A. Lestander*

Unit of Biomass Technology and Chemistry, Swedish

system as a result of changes in interacting electronic clouds and of hydrogen bonding.

Prediction of *Pinus sylvestris* clear-wood properties using NIR spectroscopy and biorthogonal partial least squares regression

Torbjörn A. Lestander, Johan Lindeberg, Daniel Eriksson, and Urban Bergsten

Abstract: Thirteen wood parameters were predicted using near infrared (NIR) spectra in the range 780–2380 nm modelled by biorthogonal partial least squares regression. The analysis of parameters and NIR measurements was done on clear-wood samples from the base and midstem of Scots pine (*Pinus sylvestris* L.) from trees at two sites. Calibrations based on the measured parameters at seven growth rings (cambial age ranging between 6 and 42 years) could be divided into three groups: (*i*) the best accuracy was found for longitudinal modulus of elasticity ($r > 0.9$) followed by bending, compression, and cell length ($0.8 < r < 0.9$); (*ii*) microfibril angle, longitudinal hardness, proportion of latewood, and creep with correlations in the range of 0.7–0.8; and (*iii*) tangential hardness, cell diameter, and cell wall thickness with $0.4 < r < 0.7$. It was also shown that juvenile (cambial age ≤20 years) and mature wood can be classified using NIR techniques.

tion are described by Springer and Hajny (1970) and

Ocean transport of wood pellets

Ahead … biomass properties to modify

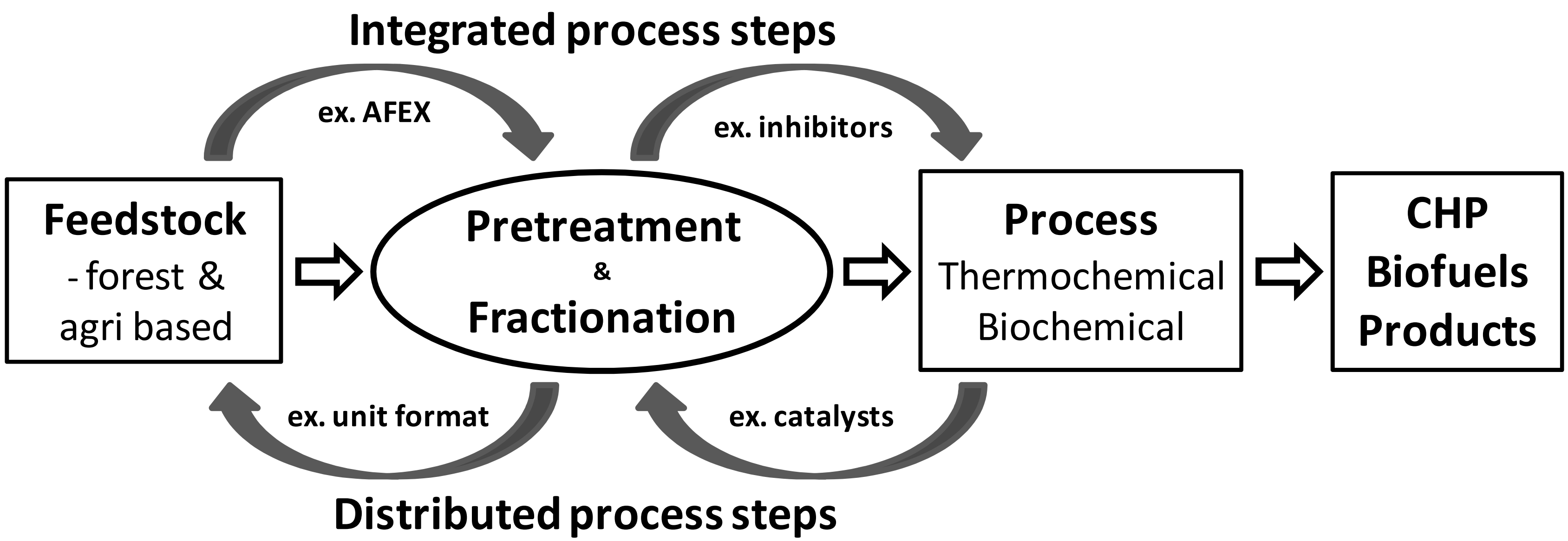
- bulk density – from harvest to industry
- crude fractionation of biomass (e.g. stem wood, stumps, branches, bark, needles)

- size (e.g. chips, sawdust, powders, fibre bundles)
- size distribution (e.g. gravity tables, air screens, wet sorting etc)
- fine fractionation (e.g. gravity tables, air screens, wet sorting etc)

- moisture content - drying
- contents of extractives
- other chemical contents (e.g. C-H-O ratios, ash elements, inhibitors, catalysts)
- reactivity (e.g. microwaves, electron beams, corona)
- rheology – reduce feeding problems in processes
- tribology – friction properties (e.g. at high pressure etc)
- density (cell wall collapse and relaxation)

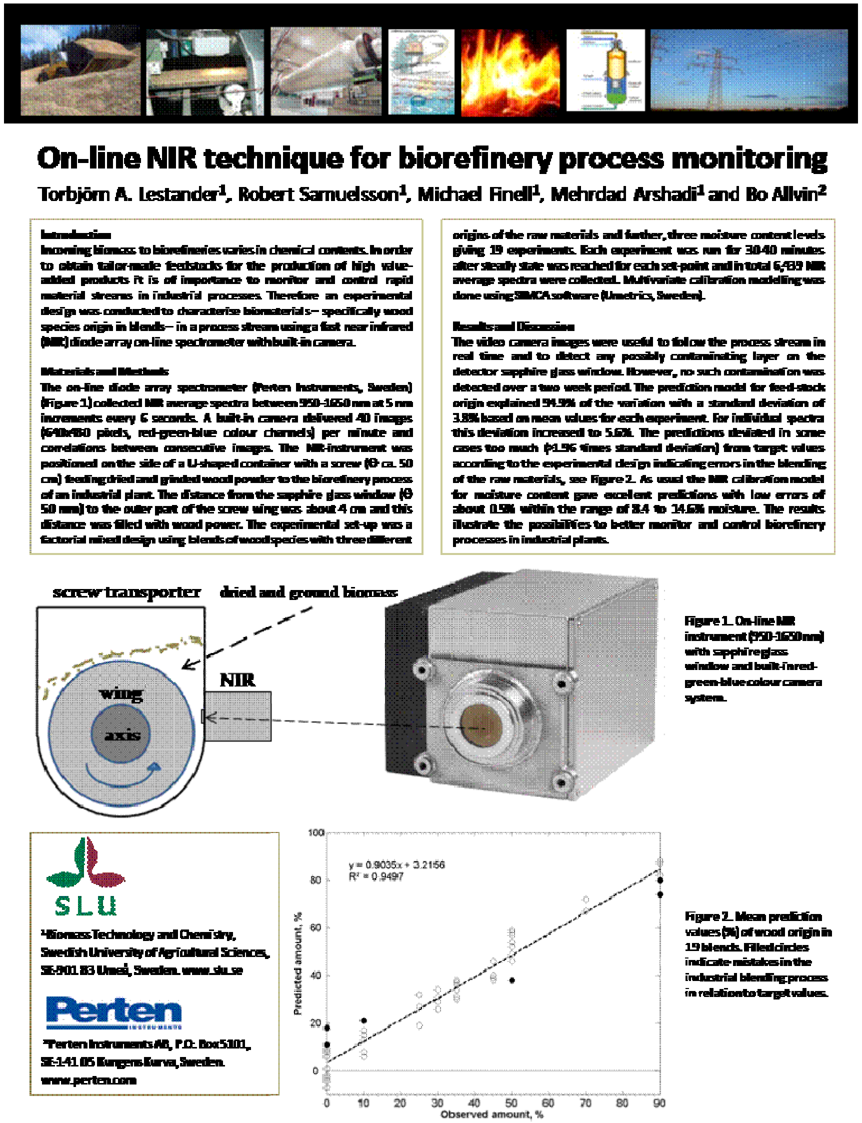
… more (what – up to discussion)

Integrated and distributed partial process steps



Objective: ***increase value added early in the supply chain*** and integrate different actors and avoid suboptimal linkage.

One objective in the Bio4Energy



On-line NIR technique for biorefinery process monitoring

Torbjörn A. Lestander[1], Robert Samuelsson[1], Michael Finell[1], Mehrdad Arshadi[1] and Bo Allvin[2]

Introduction
Incoming biomass to biorefineries varies in chemical contents. In order to obtain tailor-made feedstocks for the production of high value-added products it is of importance to monitor and control rapid material streams in industrial processes. Therefore an experimental design was conducted to characterise biomaterials – specifically wood species origin in blends – in a process stream using a fast near infrared (NIR) diode array on-line spectrometer with built-in camera.

Materials and Methods
The on-line diode array spectrometer (Perten Instruments, Sweden) (Figure 1) collected NIR average spectra between 950-1650 nm at 5 nm increments every 6 seconds. A built-in camera delivered 40 images (640x480 pixels, red-green-blue colour channels) per minute and correlations between consecutive images. The NIR-instrument was positioned on the side of a U-shaped container with a screw (Ø ca. 50 cm) feeding dried and grinded wood powder to the biorefinery process of an industrial plant. The distance from the sapphire glass window (Ø 50 mm) to the outer part of the screw wing was about 4 cm and this distance was filled with wood power. The experimental set-up was a factorial mixed design using blends of wood species with three different origins of the raw materials and further, three moisture content levels giving 19 experiments. Each experiment was run for 30-40 minutes after steady state was reached for each set-point and in total 6,439 NIR average spectra were collected. Multivariate calibration modelling was done using SIMCA software (Umetrics, Sweden).

Results and Discussion
The video camera images were useful to follow the process stream in real time and to detect any possibly contaminating layer on the detector sapphire glass window. However, no such contamination was detected over a two week period. The prediction model for feed-stock origin explained 94.9% of the variation with a standard deviation of 3.8% based on mean values for each experiment. For individual spectra this deviation increased to 5.6%. The predictions deviated in some cases too much (>1.96 times standard deviation) from target values according to the experimental design indicating errors in the blending of the raw materials, see Figure 2. As usual the NIR calibration model for moisture content gave excellent predictions with low errors of about 0.5% within the range of 8.4 to 14.6% moisture. The results illustrate the possibilities to better monitor and control biorefinery processes in industrial plants.

Figure 1. On-line NIR instrument (950-1650 nm) with sapphire glass window and built-in red-green-blue colour camera system.

Figure 2. Mean prediction values (%) of wood origin in 19 blends. Filled circles indicate mistakes in the industrial blending process in relation to target values.

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[2]Perten Instruments AB, P.O. Box 5101, SE-141 05 Kungens Kurva, Sweden. www.perten.com

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