Differentiation between natural and regenerated cellulose

…as part of microplastic litter in the oceans!

RESEARCH WITH PLYMOUTH UNIVERSITY

Technische Hochschule Rosenheim
Technical University of Applied Sciences

MOSER STEFAN PROCESS OPTIMIZATION

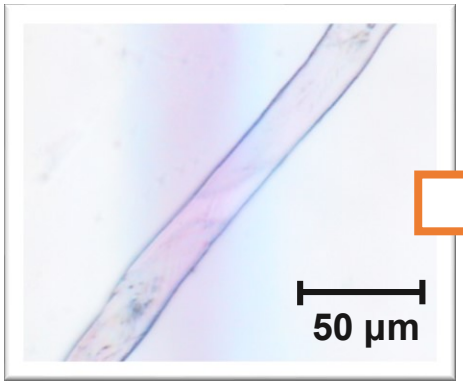
*It is considered a **top environmental problem** [UNEP, 2005]*

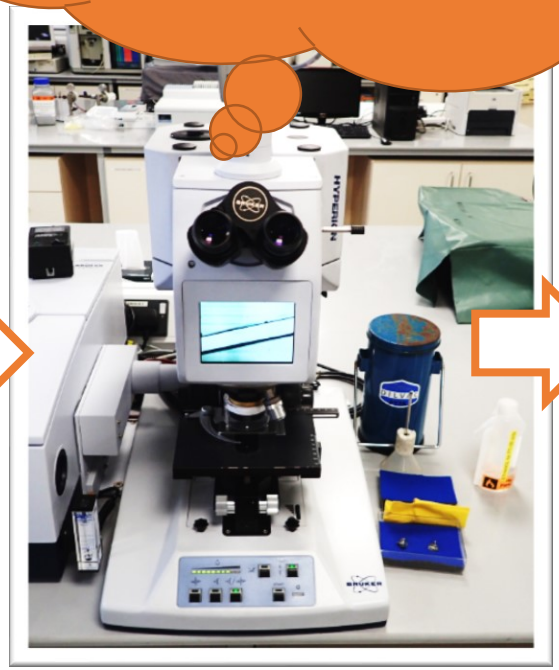
*(…) were found in remote locations such as the **arctic** and the **deep sea** [Obbard et al. 2014]*

*(…) identified as a factor **contributing to biodiversity loss** [Gall and Thompson 2015]*

*(…) a **potential threat** to **human health** and **activities** [Thompson et al. 2009]*

The analysis of microplastics is very difficult. As a consequence **questionable results** are reported. [Lendl et al. 2017]

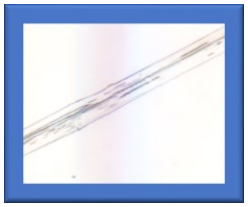




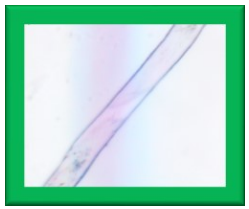
Rayon, Viscose … regenerated cellulose

The analysis of microplastics is very difficult. As a consequence **questionable results** are reported. [Lendl et al. 2017]

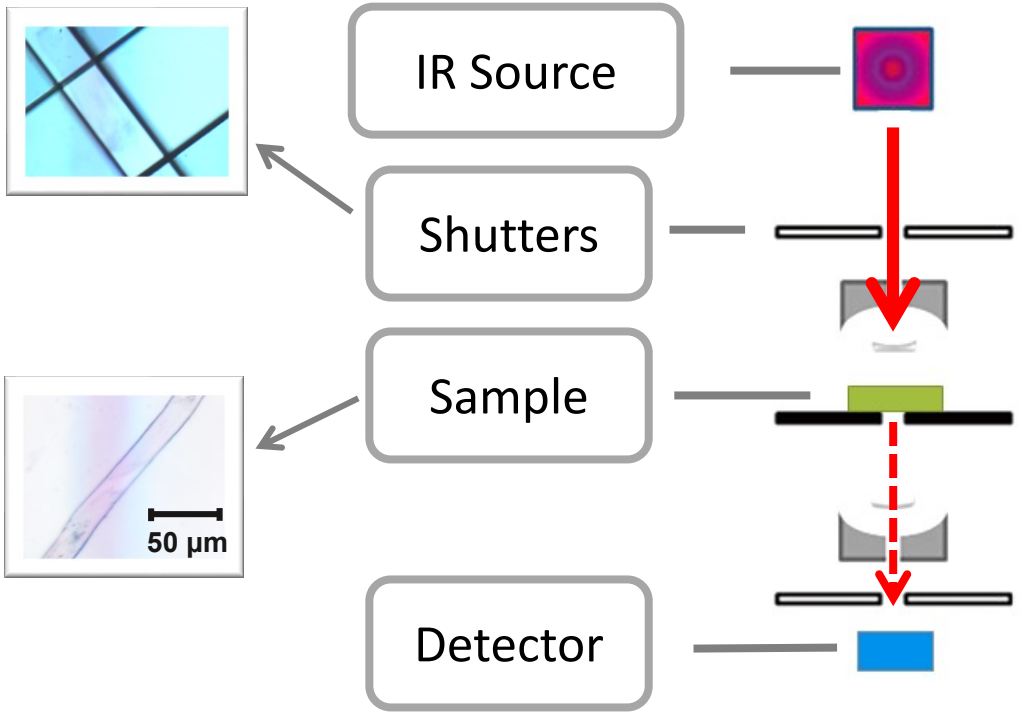
Raw Material



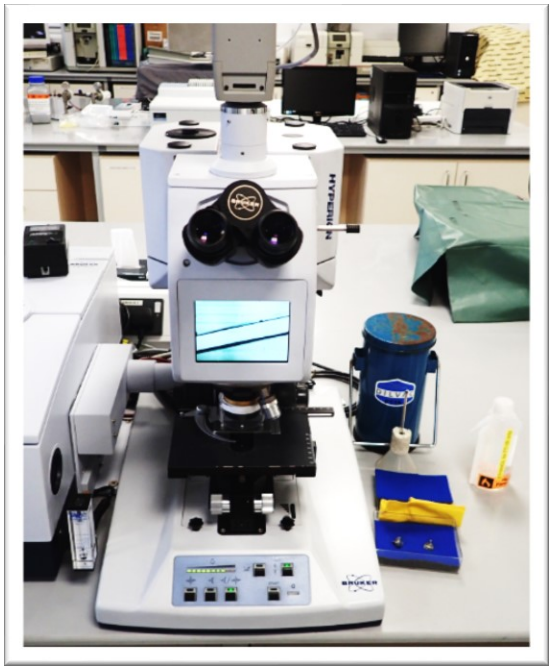
Regenerated Cellulose
(Rayon, Viscose etc.)



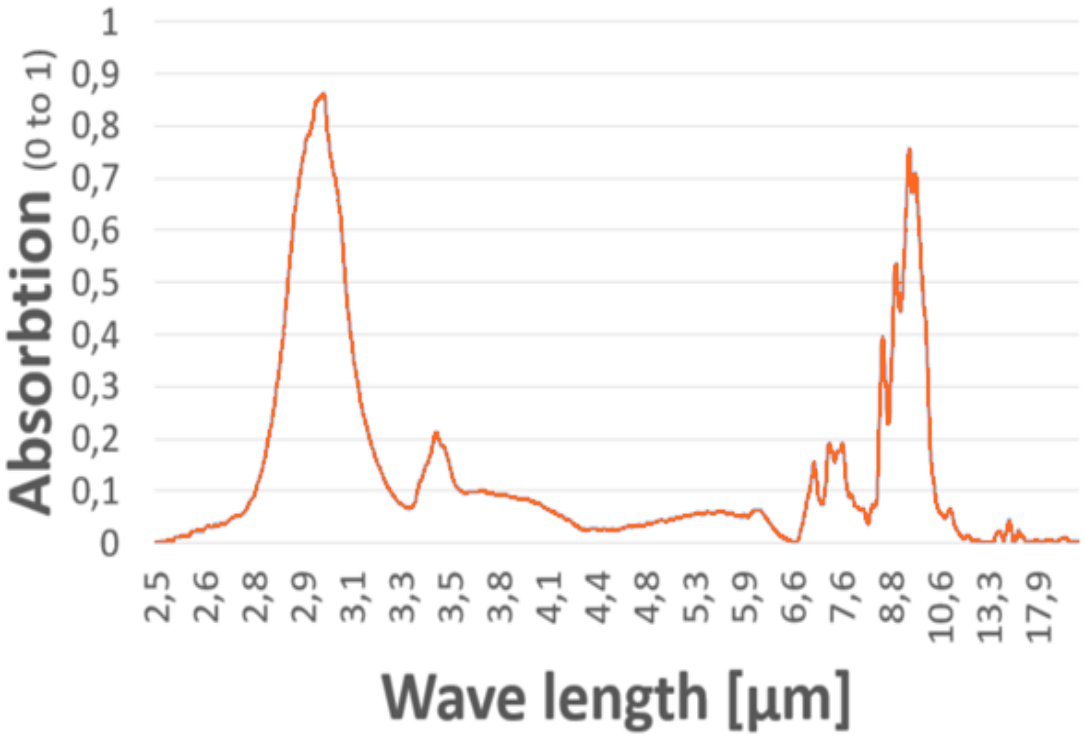
Natural Cellulose
(Cotton, Wood Pulp)



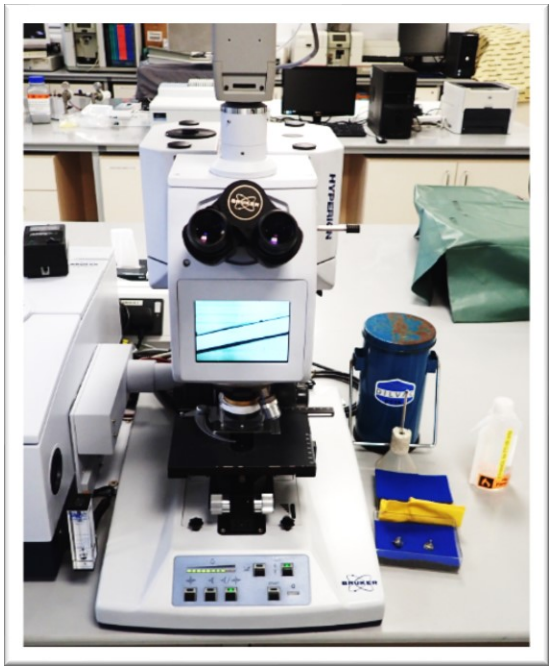
FT-IR Microscope
(Transmission, ATR)



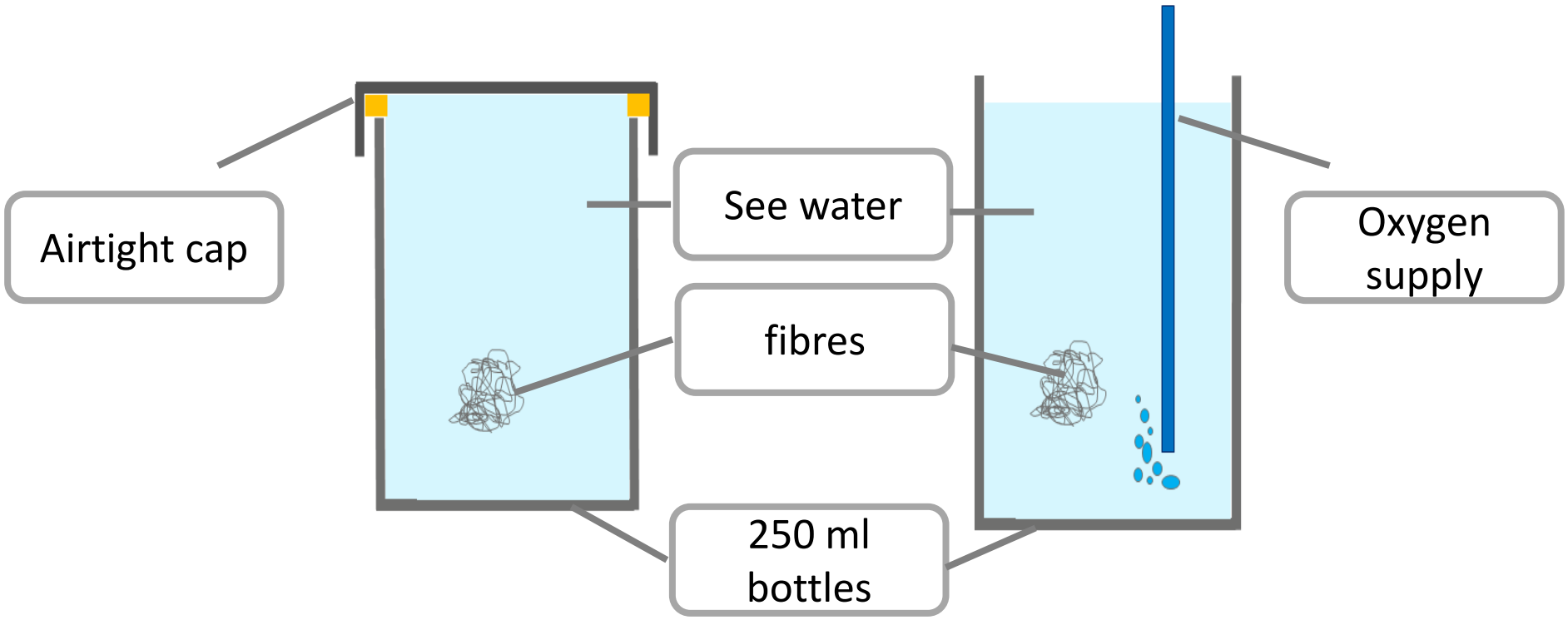
Bruker Hyperion 1000



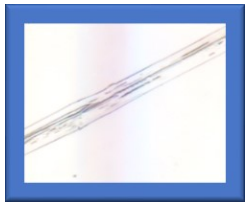
FT-IR Microscope
(Transmission, ATR)



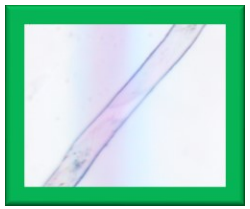
Bruker Hyperion 1000


Airtight cap
See water
fibres
250 ml bottles
Oxygen supply

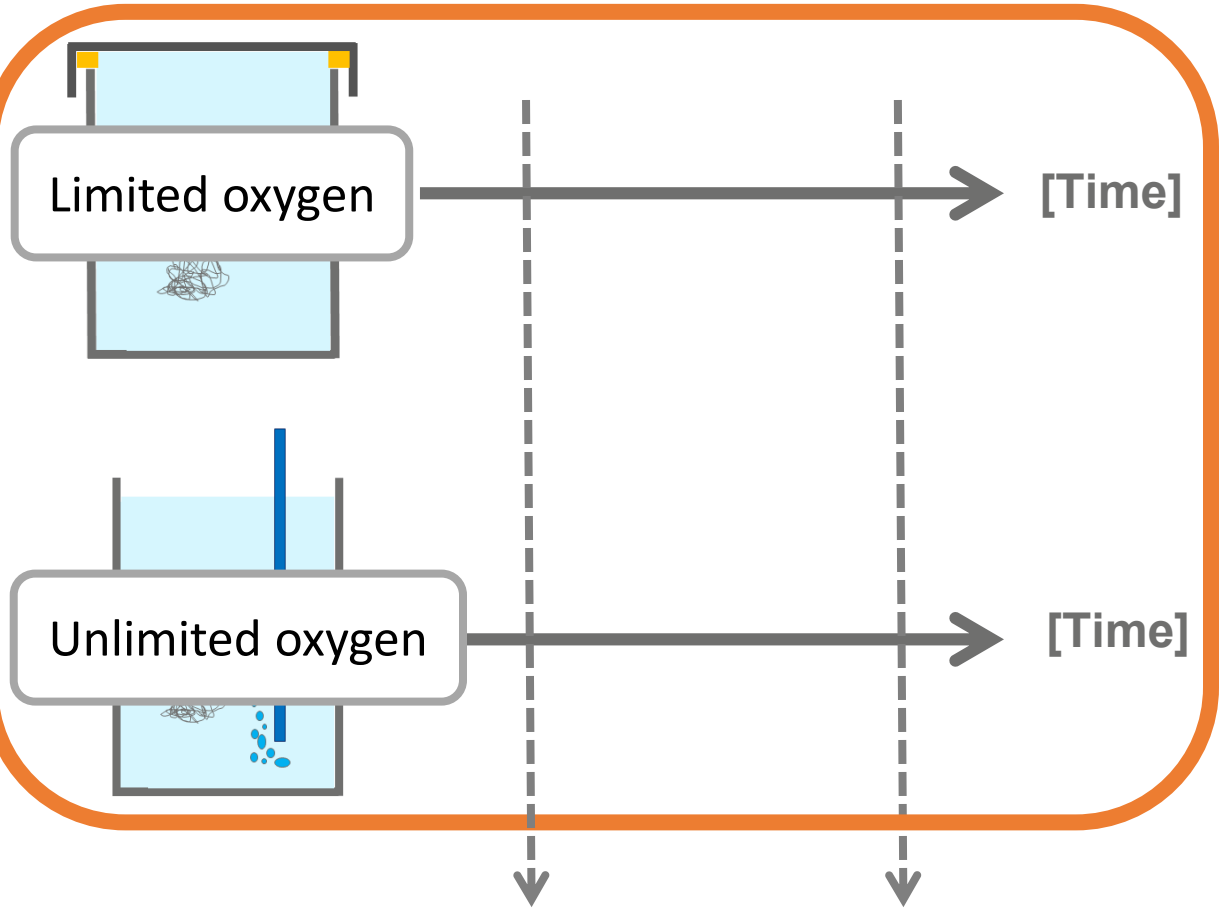
Raw Material

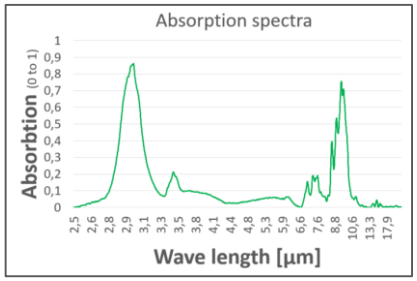


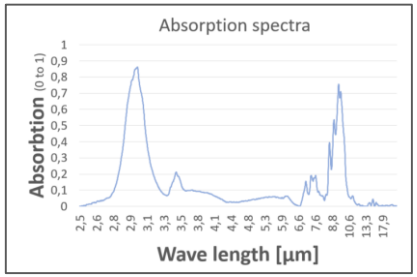
Regenerated Cellulose
(Rayon, Viscose etc.)

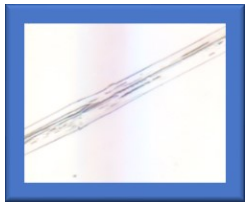


Natural Cellulose
(Cotton, Wood Pulp)

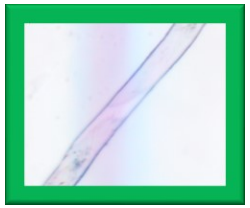




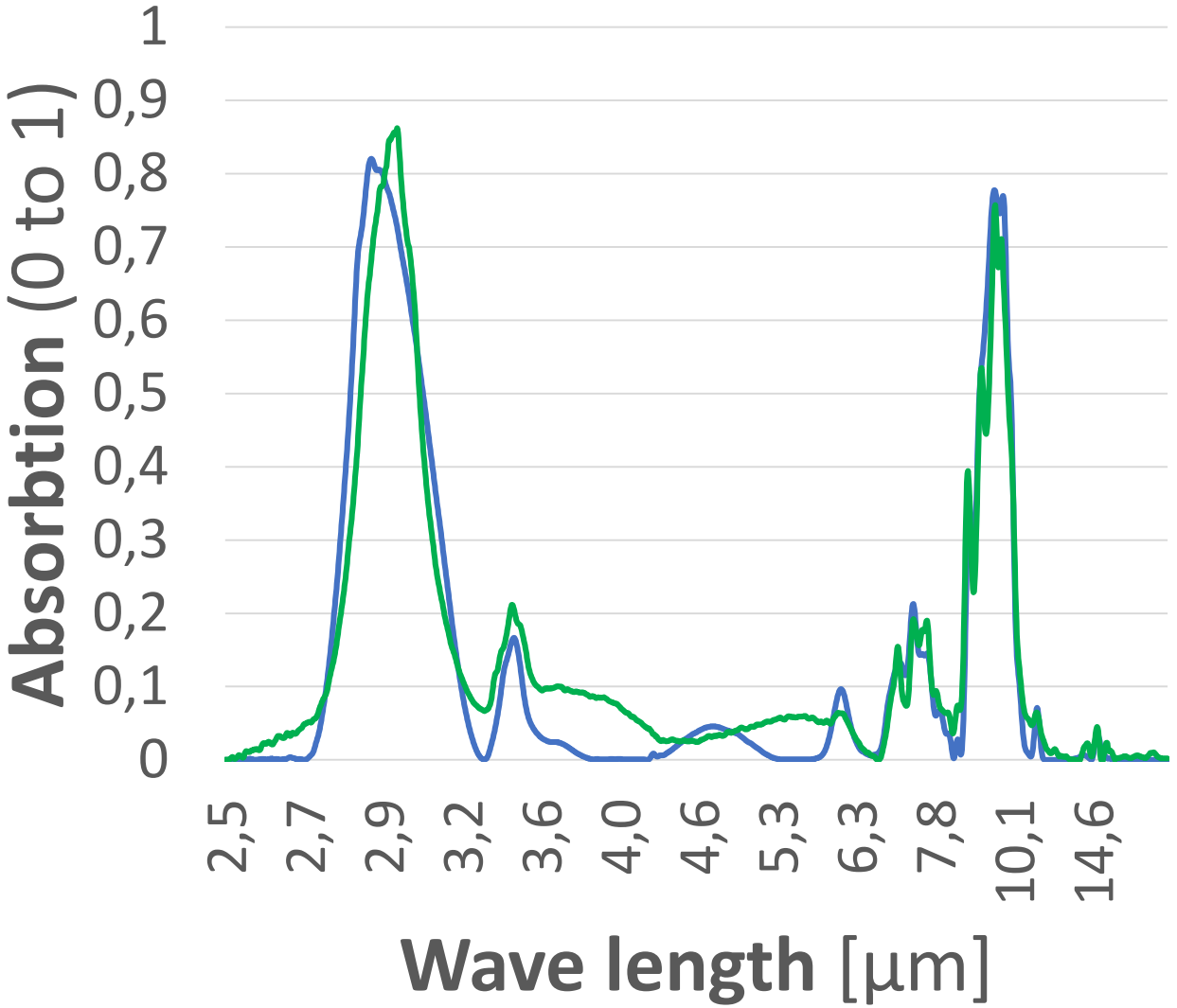


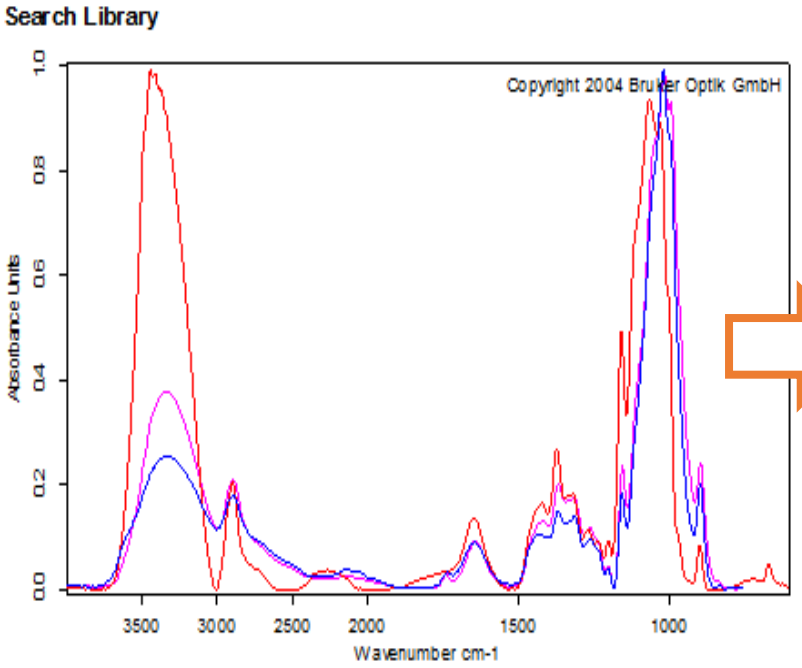


Regenerated Cellulose

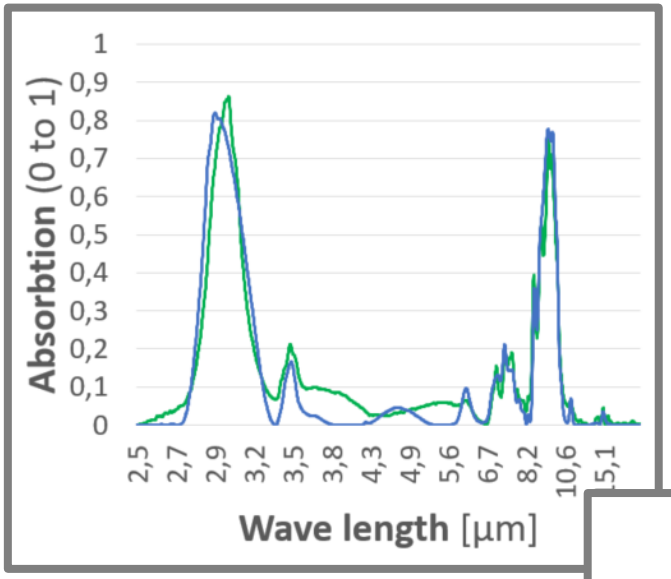


Natural Cellulose





Sample	Result
Cotton	Rayon
Wood Pulp	Rayon
…	Rayon
Rayon_1	Rayon
Rayon_2	Rayon
…	Rayon

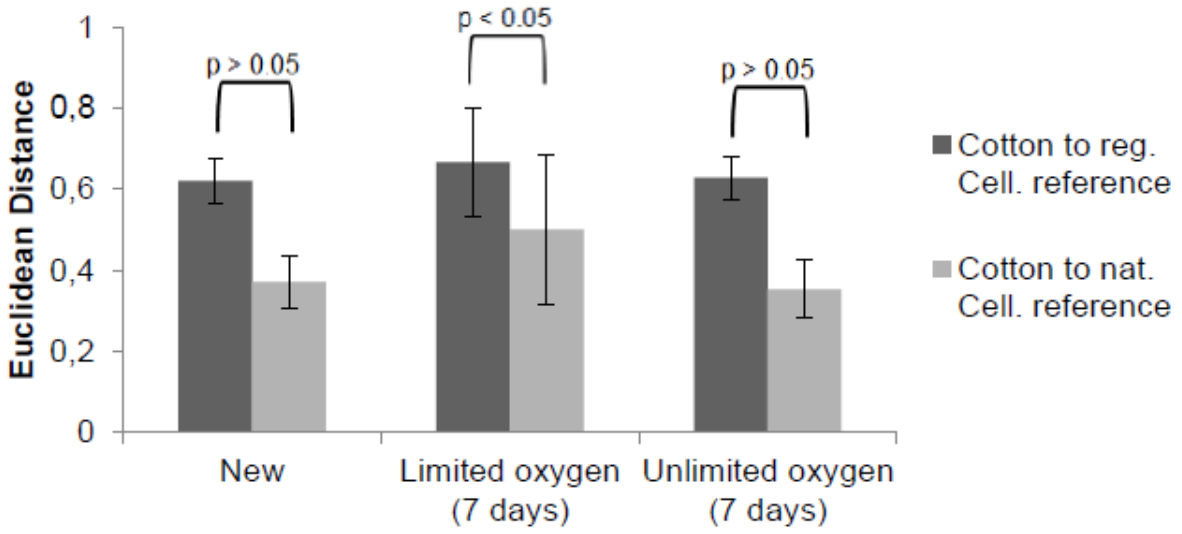


$$D = \sqrt{\sum_i \left(As(\lambda i) - Ar\ (\lambda i)\right)^2}$$

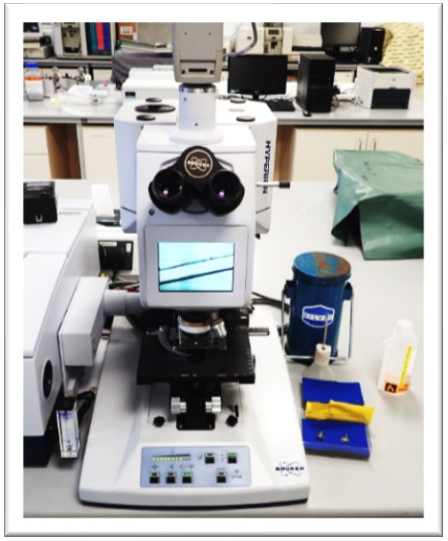
D: *Euclidean Distance*

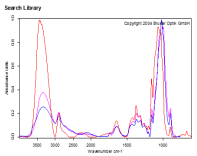
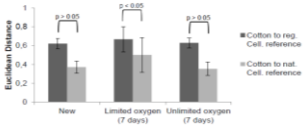
As (λi): *Absorption value of the sample spectrum at (λi)*

Ar (λi): *Absorption value of the reference spectrum at (λi)*



Summary so far …



		Transmission	ATR
Library Search		No results	 No results
Own database		64 % - 91 %	 18 % - 28 %

Problem formulation I

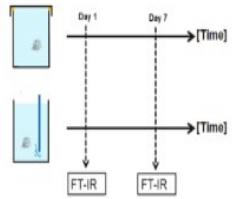


Fig. 10 Laboratory experiment exposure to the marine environment and the analysis via FT-IR

Time

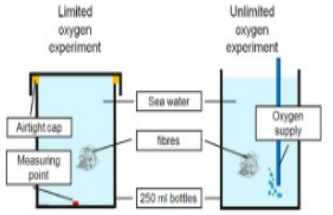
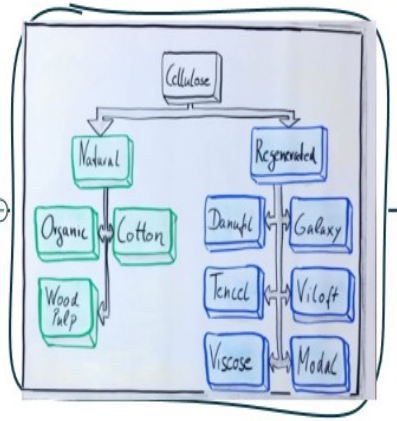


Fig. 9 Schematic representation of the limited and unlimited oxygen experiment

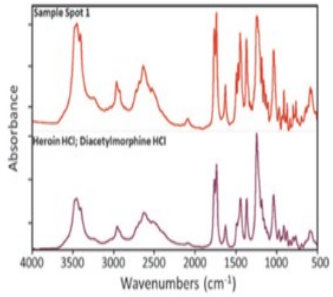
Treatment

Nature ?



Analysis

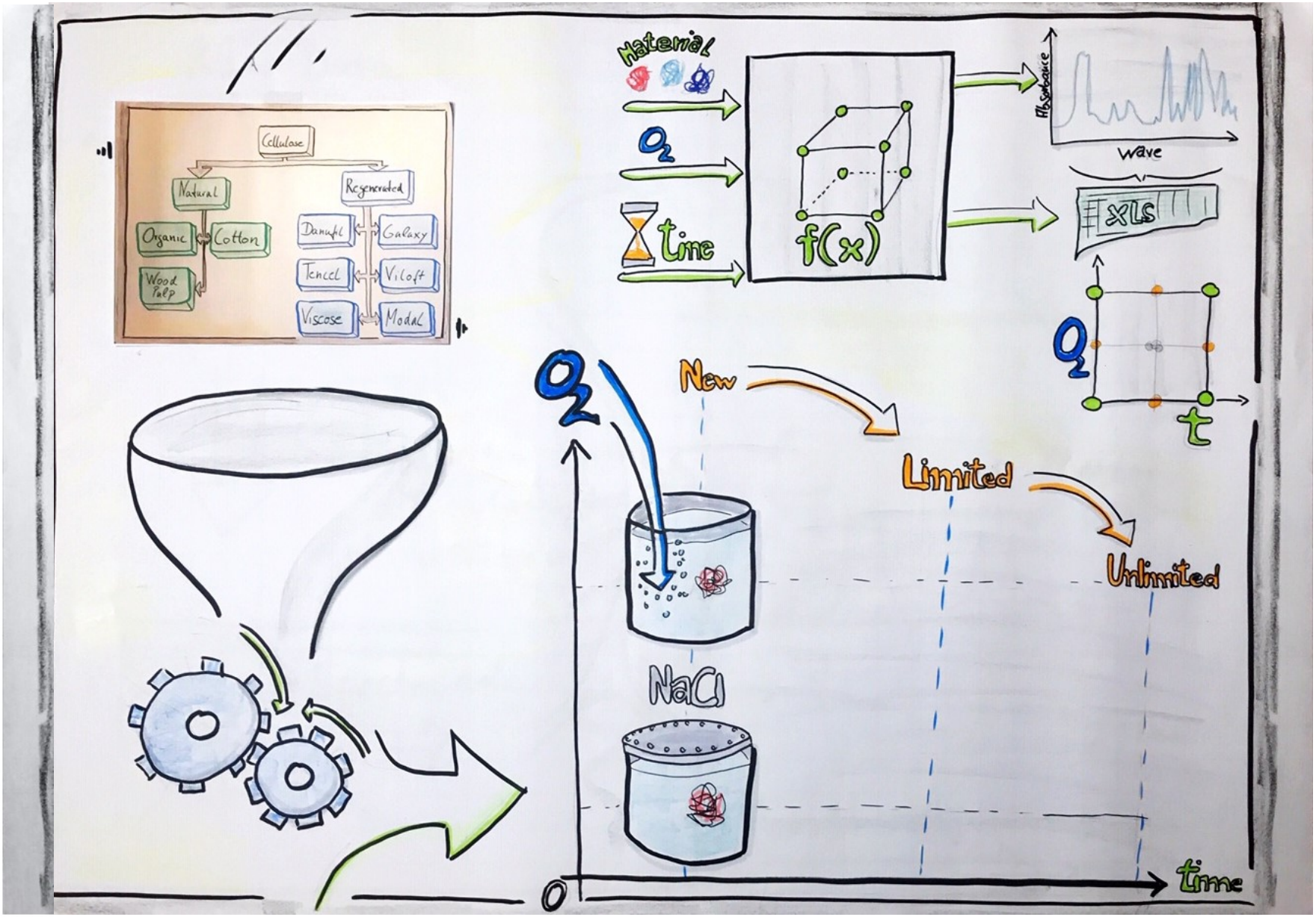
Measurement



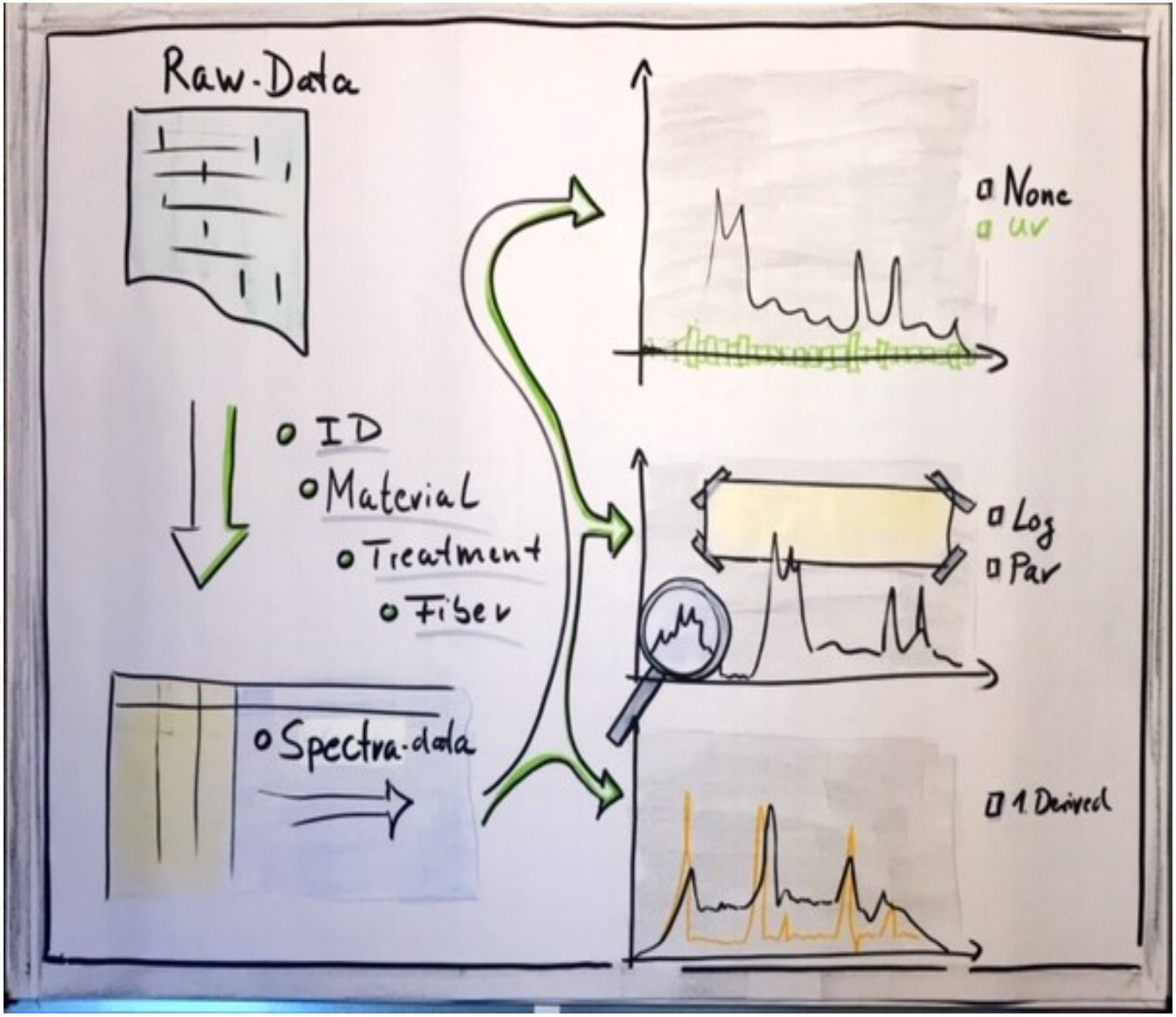
Spectra

Is it possible to differentiate between regenerated and natural cellulose?

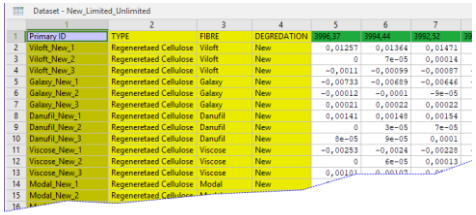
Problem formulation II

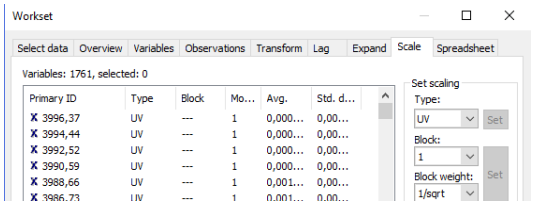


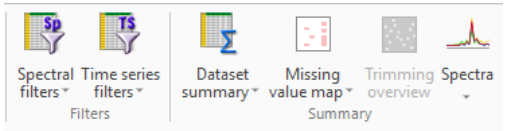
Importing the data and data preparation



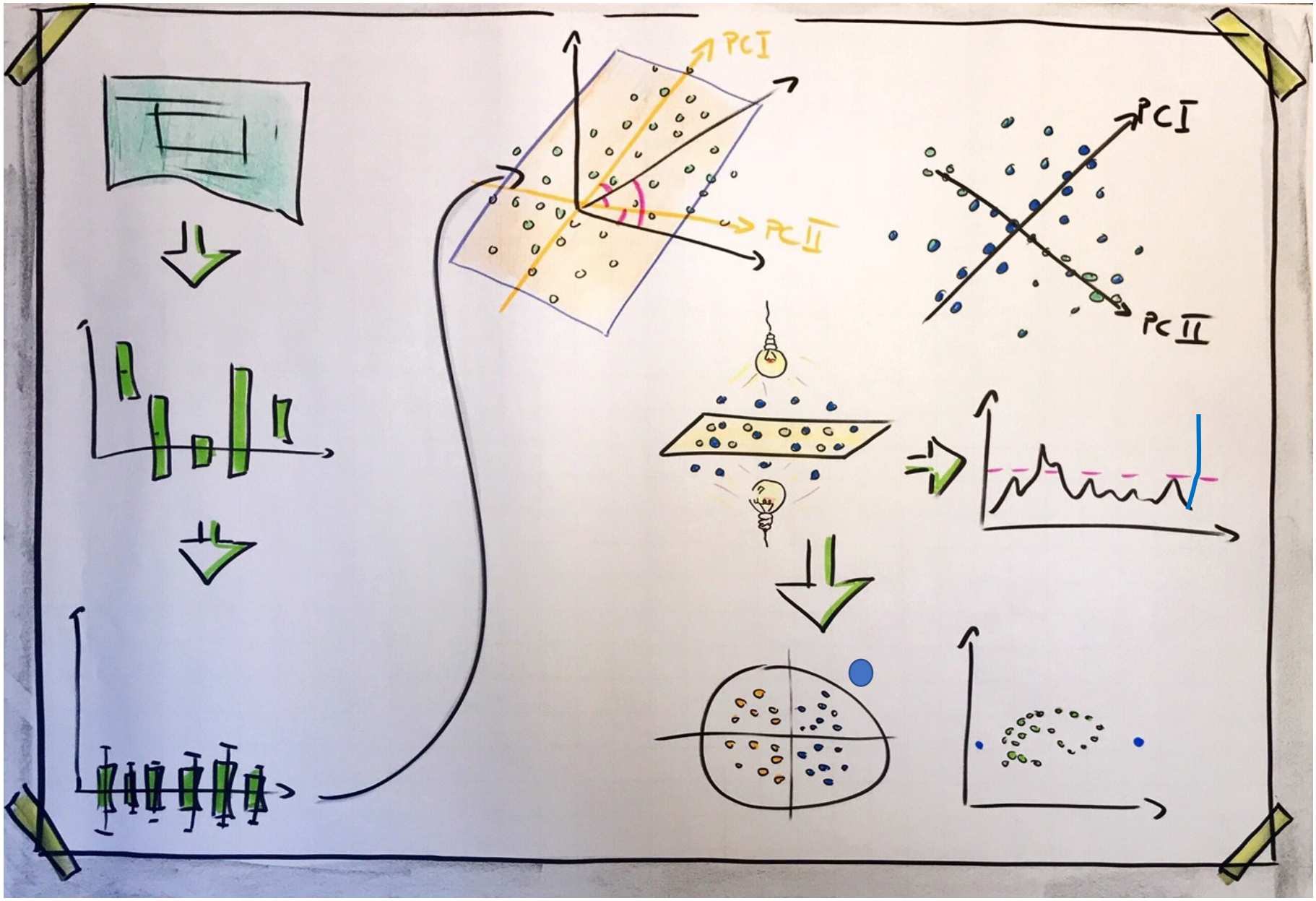
- 99 observations
- 1671 variables
- Data pre-treatment



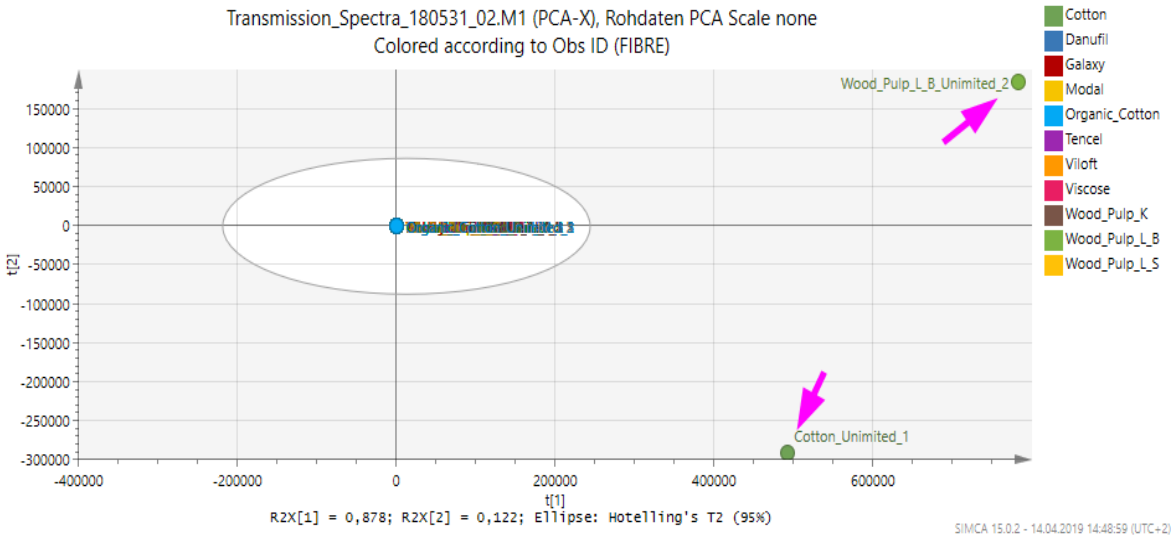




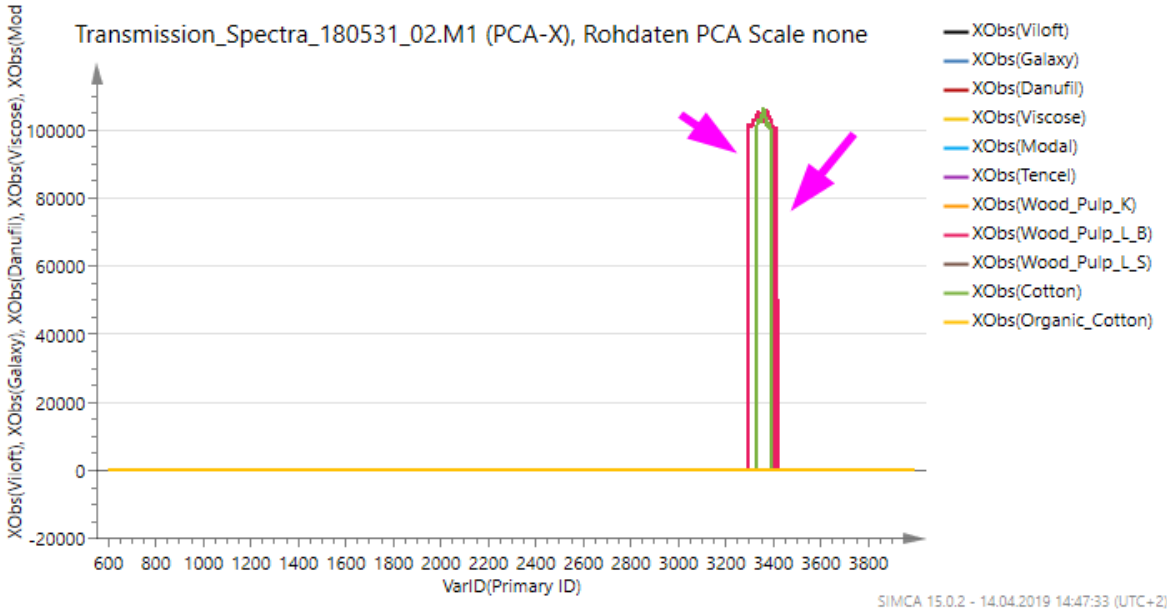
Very brief instruction to the PCA principles



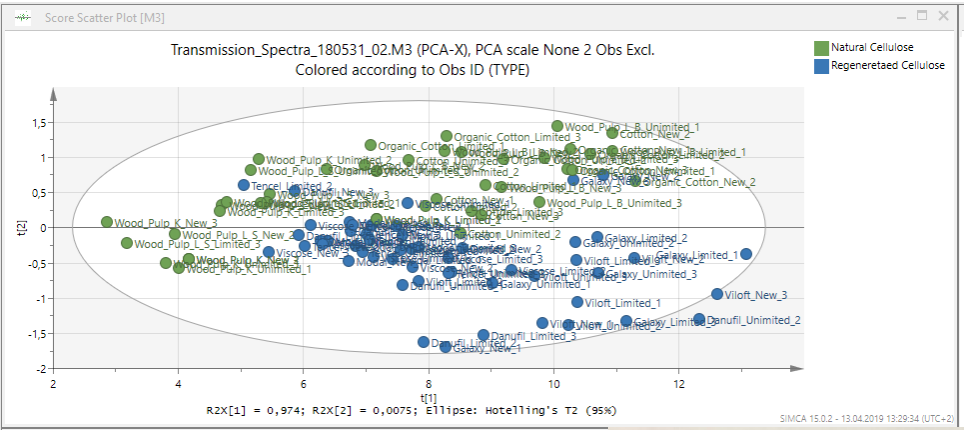
Check the data due to abnormalities with PCA-Model (no Scaling)

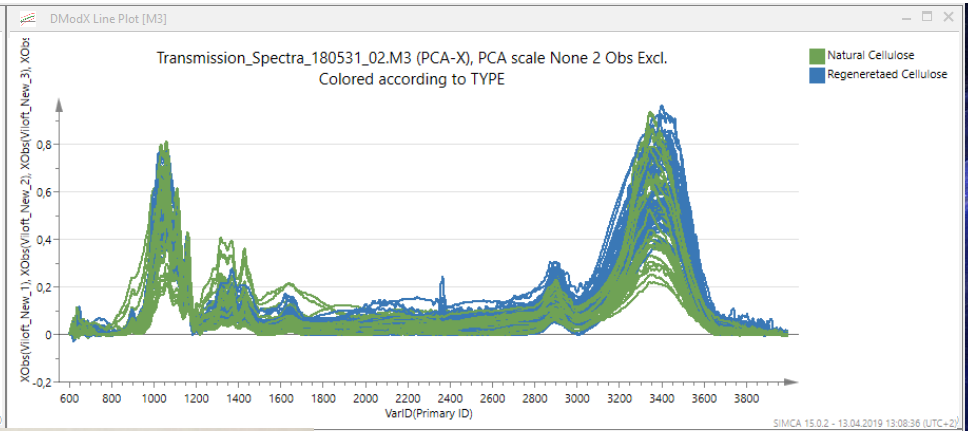


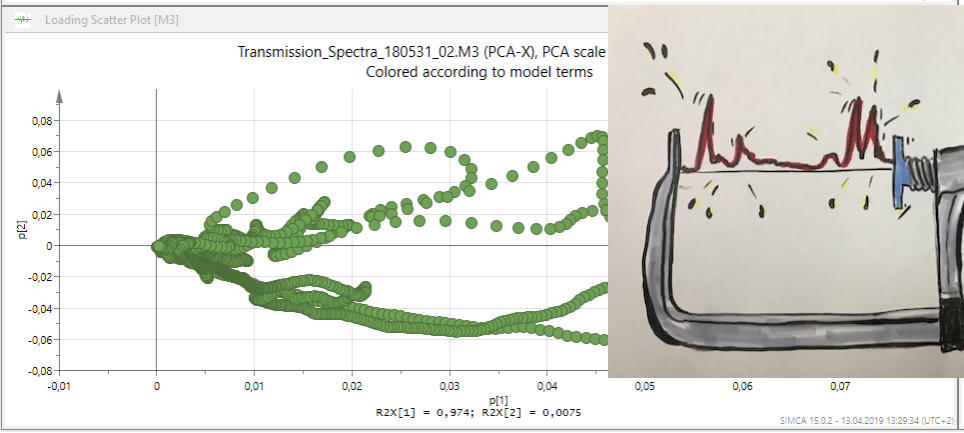
2 Observations excluded!

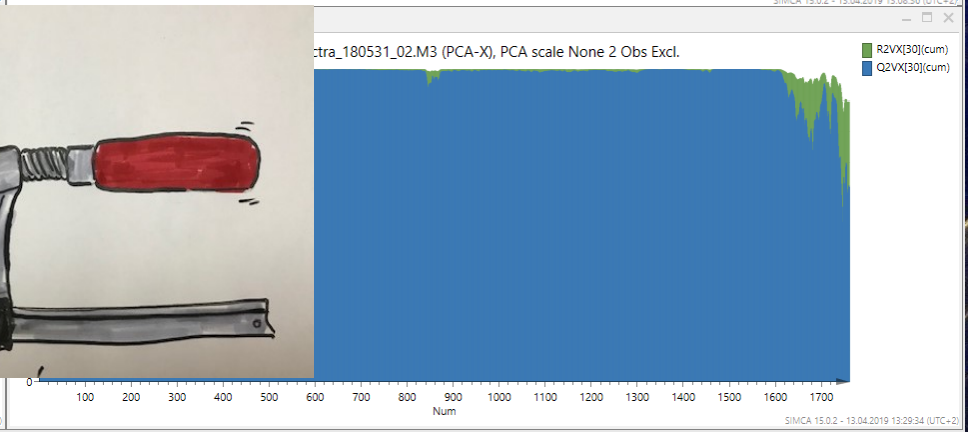


Fast overview for displaying imported data in SIMCA (without outliers)

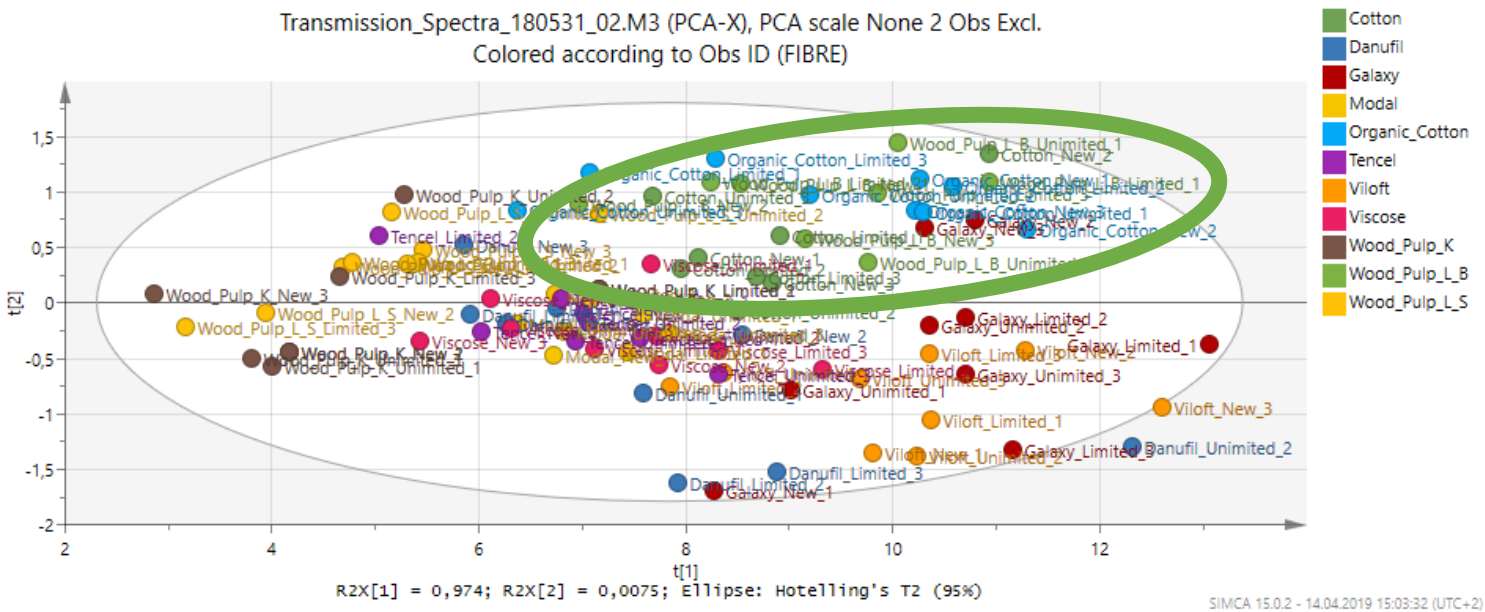


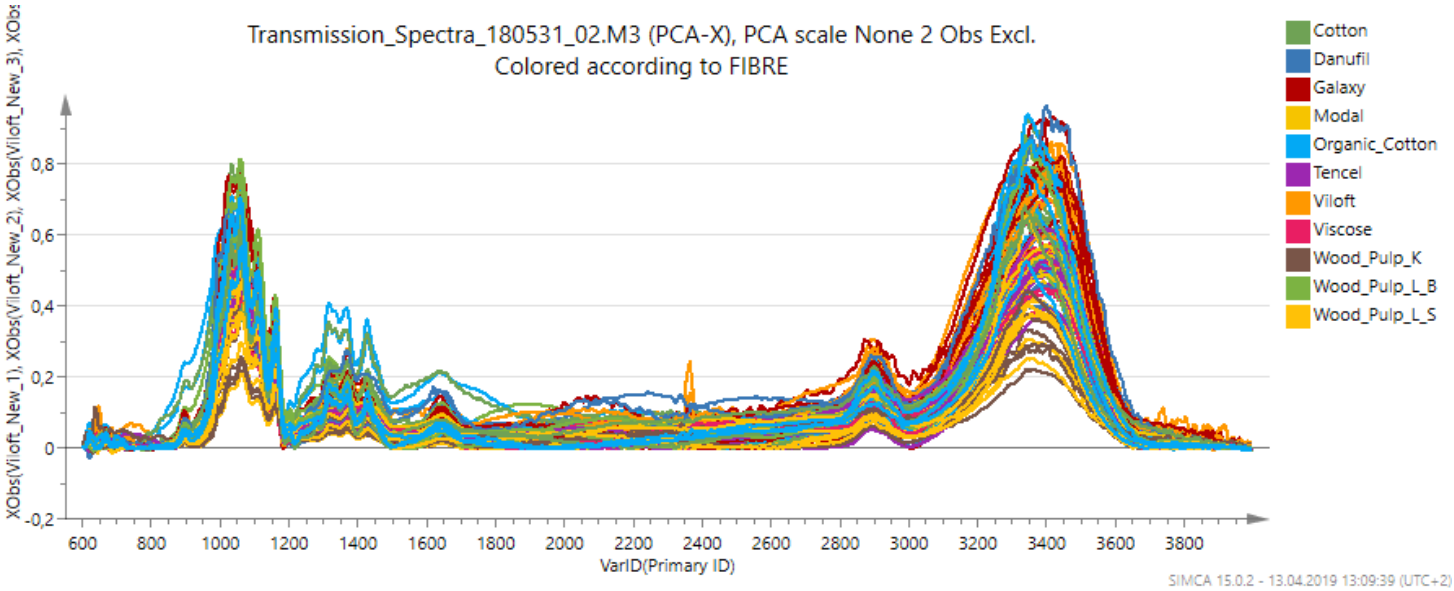




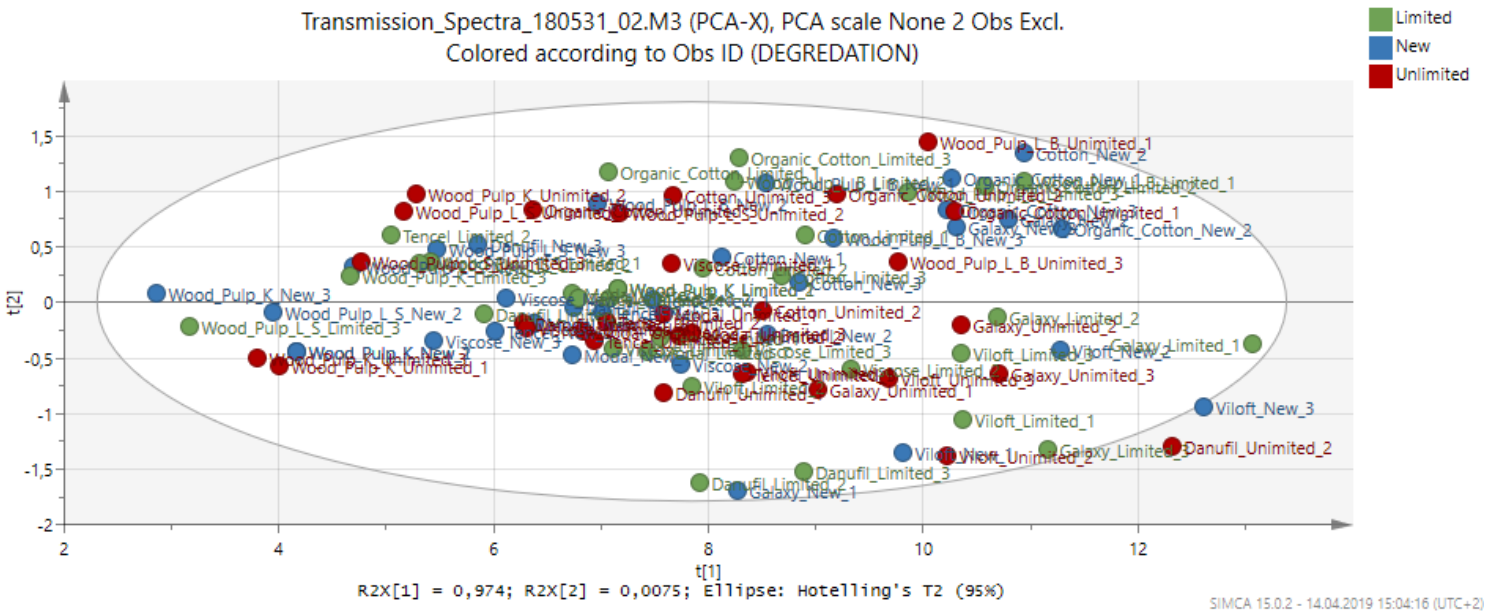


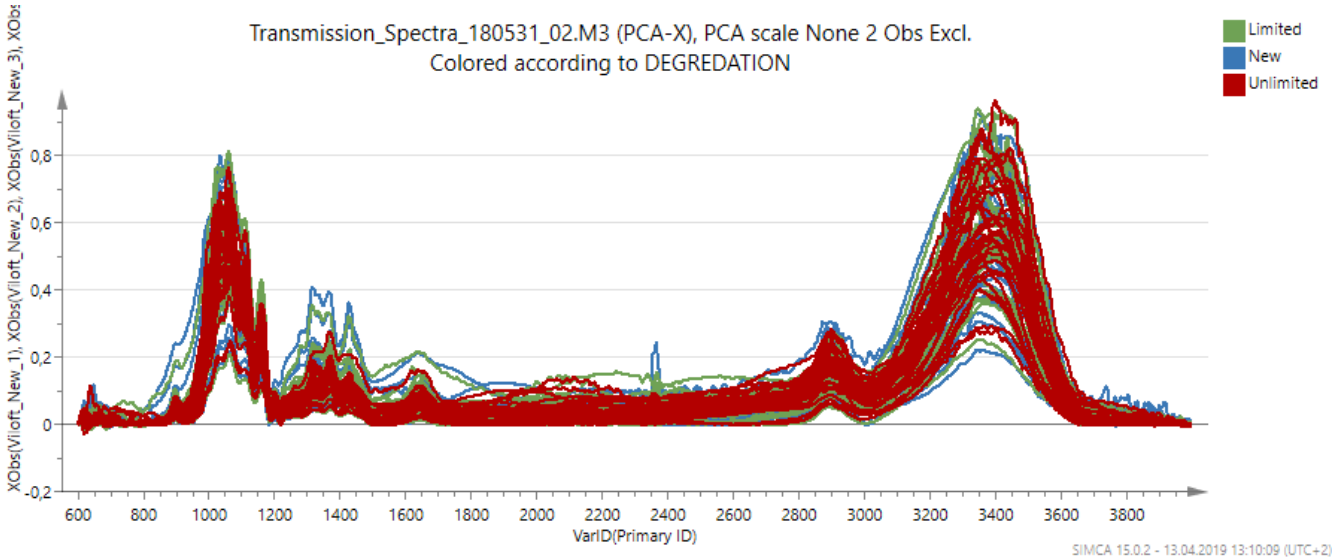
Corresponding Plots - Example data reduction I → Cellulose Type



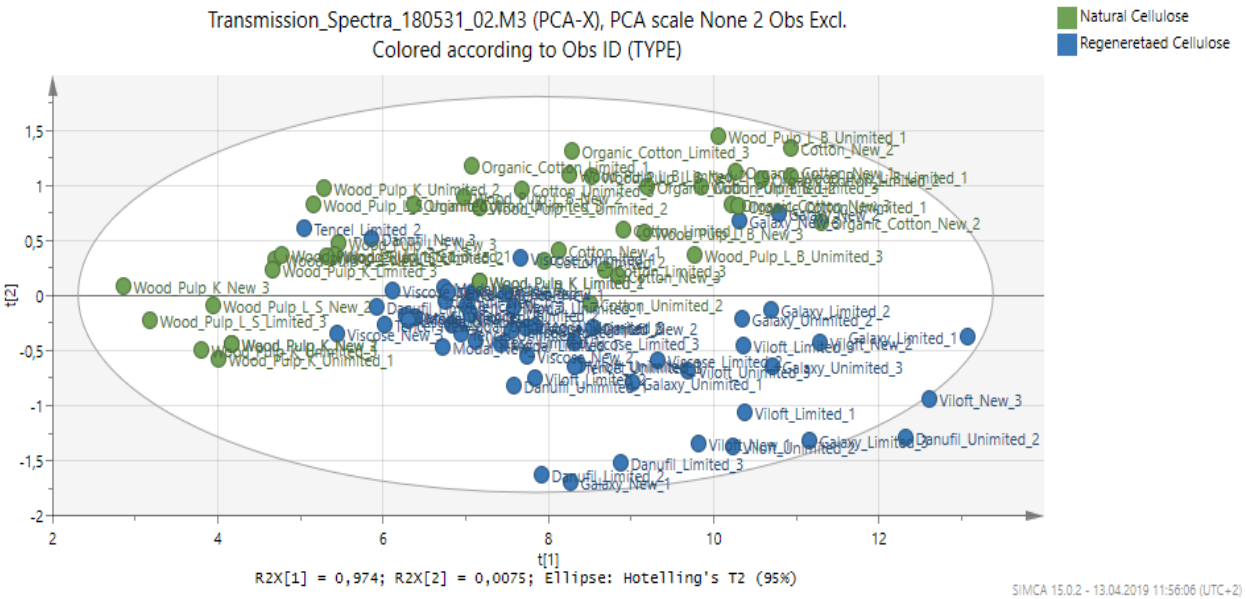


Corresponding Plots - Example data reduction II → Treatment

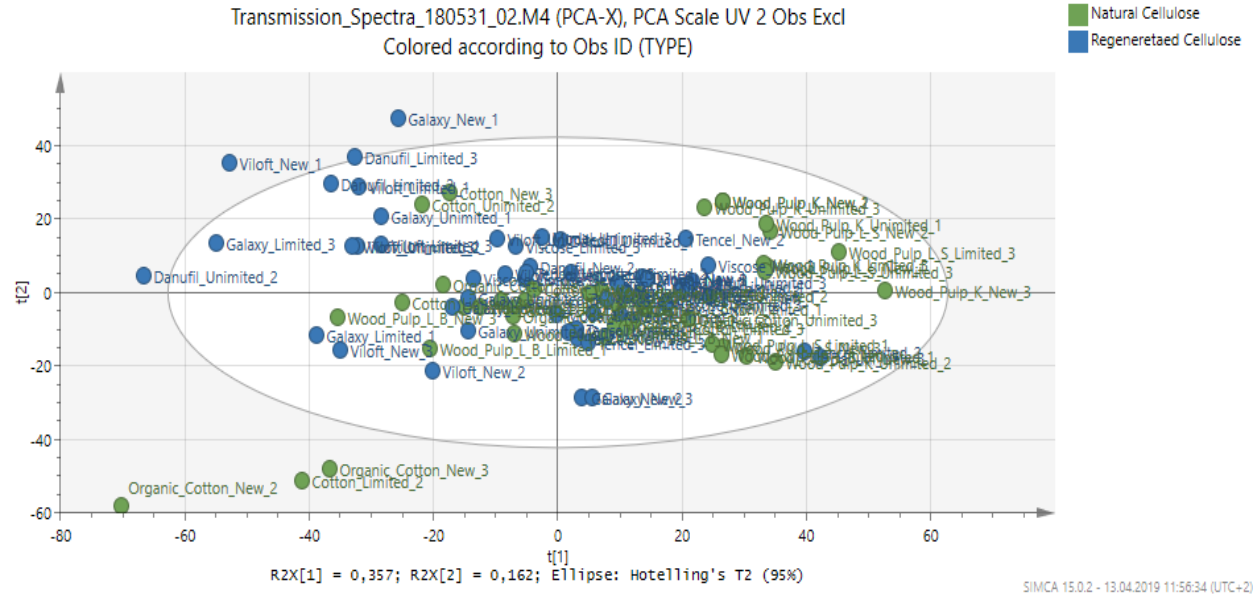




Basic Analysis

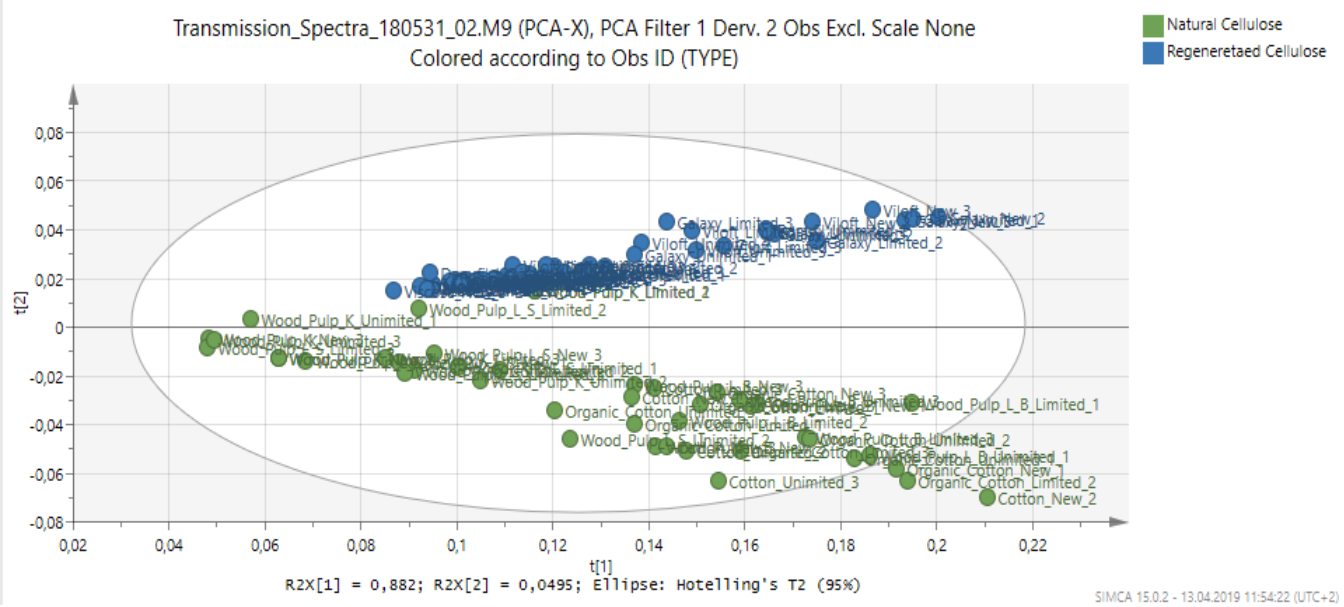


- PCA Model
- 2 Obs. excluded
- no scaling

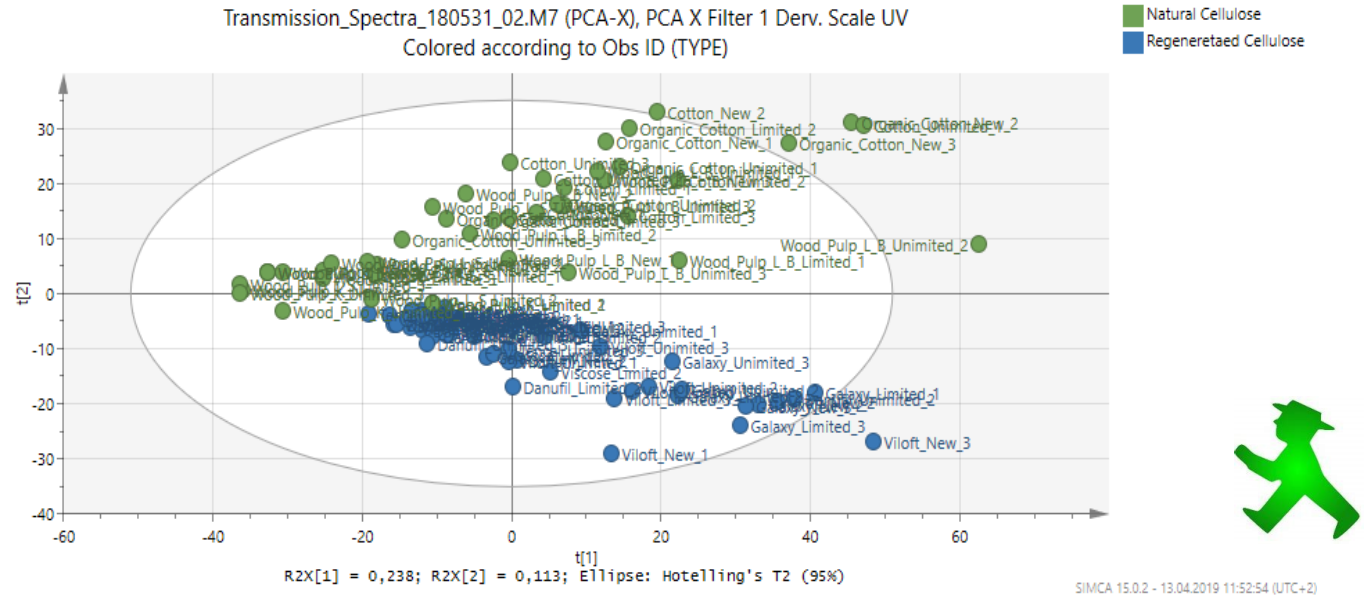


- PCA Model
- 2 Obs. excluded
- UV scaling

Basic Analysis – with filter 1st Derv.



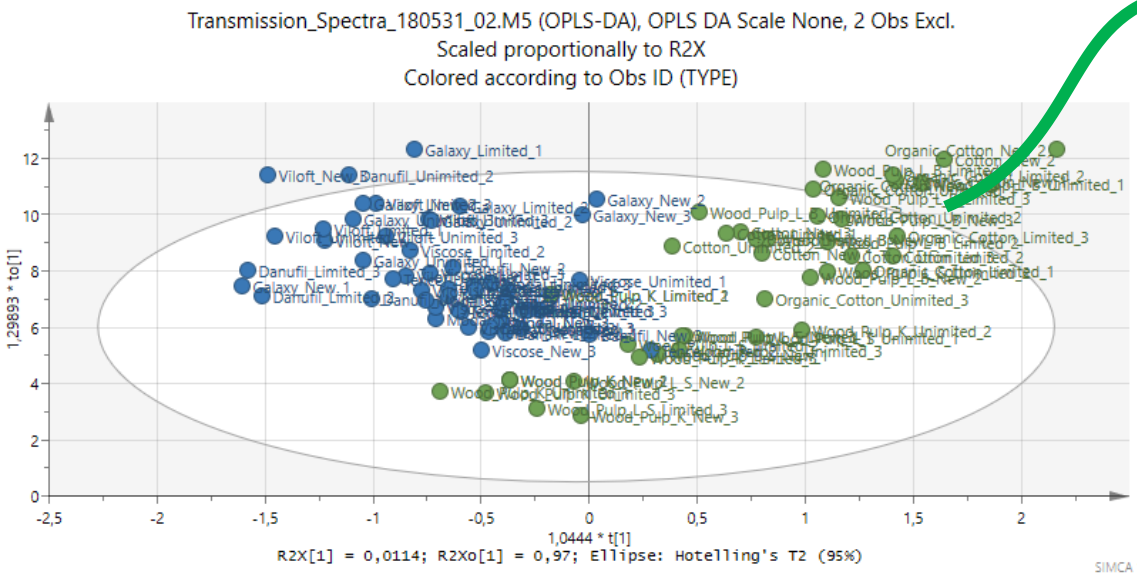
- PCA Model
- Filter 1st Derv.
- 2 Obs. excluded
- no scaling

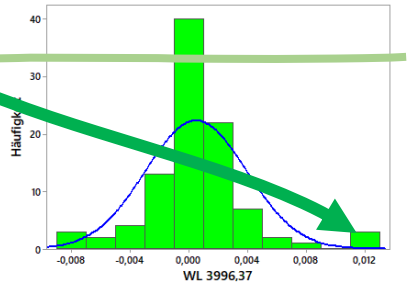


- PCA Class - Model
- 2 Obs. Excluded
- Filter 1st Derv.
- UV scaling

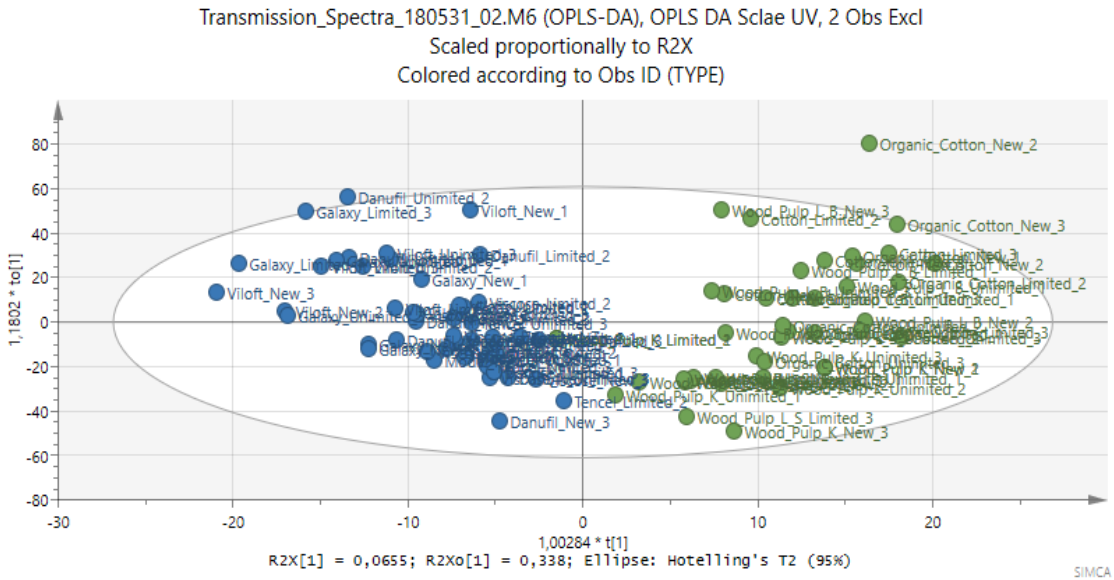
97,94 %

Advanced Analysis with OPLS-DA



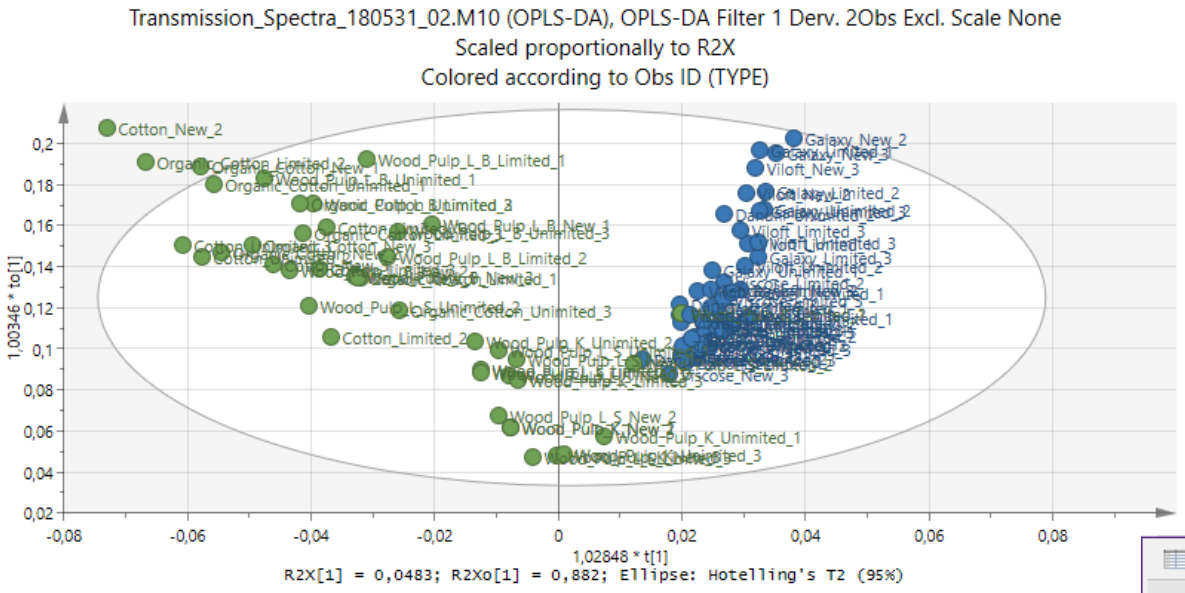


OPLS-DA Model
2 Obs. excluded
no scaling



OPLS-DA Model
2 Obs. excluded
UV scaling

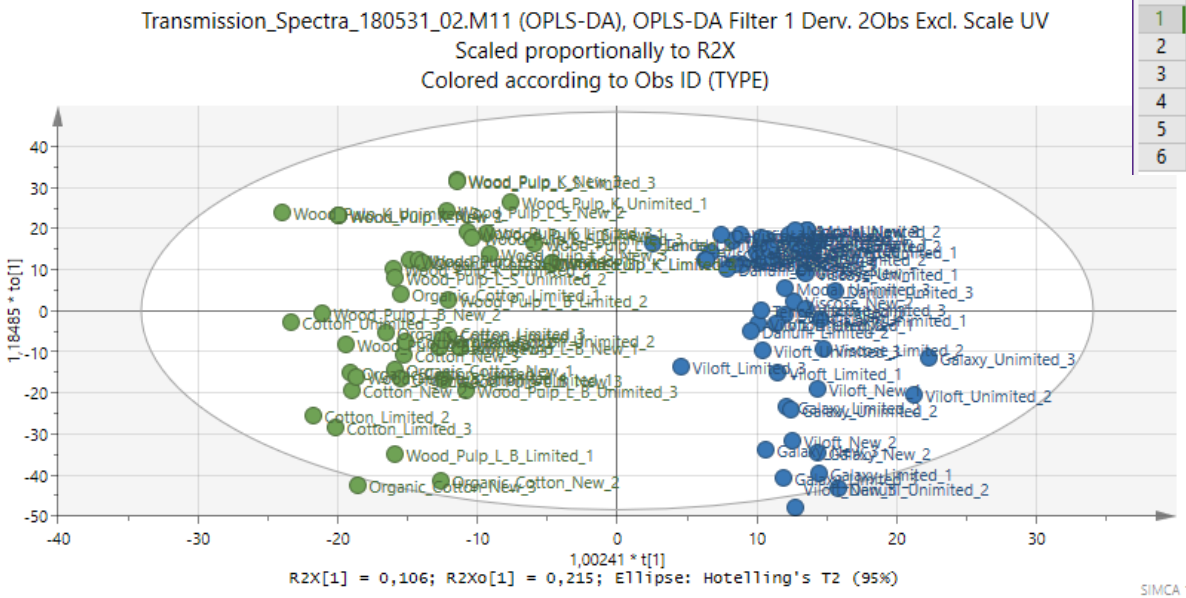
Advanced Analysis



- OPLS-DA Model
- Filter 1st Derv.
- 2 Obs. Excluded
- no scaling

Misclassification Table for Model 11

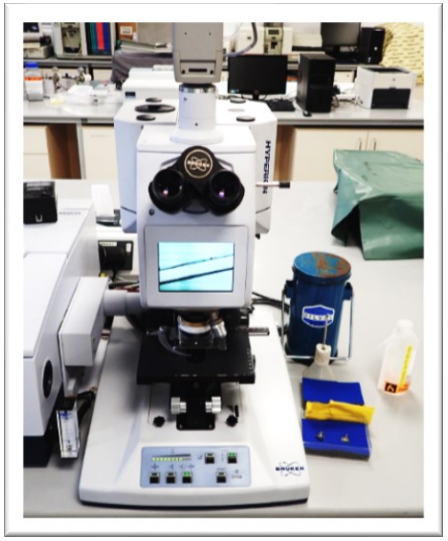
	1	2	3	4	5	6
1		Members	Correct	Nat.	Reg.	No class (YPred <= 0)
2	Nat.	43	100%	43	0	0
3	Reg.	54	100%	0	54	0
4	No class	0		0	0	0
5	Total	97	100%	43	54	0
6	Fisher's prob.	1,4e-28				

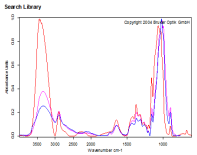
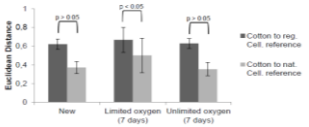
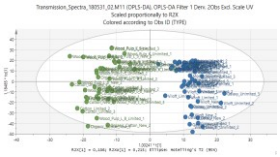


- OPLS-DA Model
- Filter 1st Derv.
- 2 Obs. Excluded
- UV scaling

100 %

SIMCA 15.0.2 - 13.04.2019 12:00:50 (UTC+2)



		Transmission		ATR
Library Search		No results		No results
Own database		64 % - 91 %		18 % - 28 %
Multivariate data analysis		100 % (except to outliers)		97 %


Outlook
Differentiation is possible
Further degredation experiments
Further materials (products, cellulose derivatives, coatings, additives ...)
Research project?


Any Questions
or comments?

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