

Differentiation between natural and regenerated cellulose

...as part of microplastic litter in the oceans!



*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]*

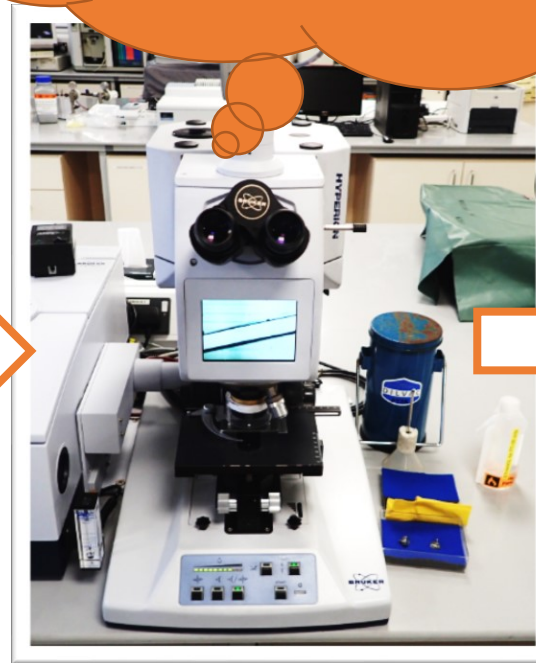
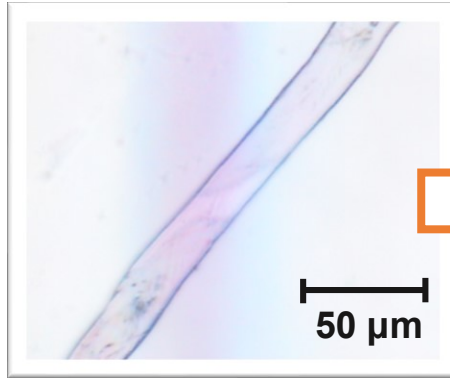


*(...) identified as a factor **contributing to biodiversity loss** [Gall and Thompson 2015]*

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

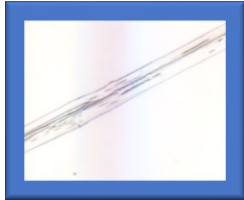
Microplastics in the oceans – Cellulosic fibres part of the problem?

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



Rayon,
Viscose ...
regenerated
cellulose

Raw Material



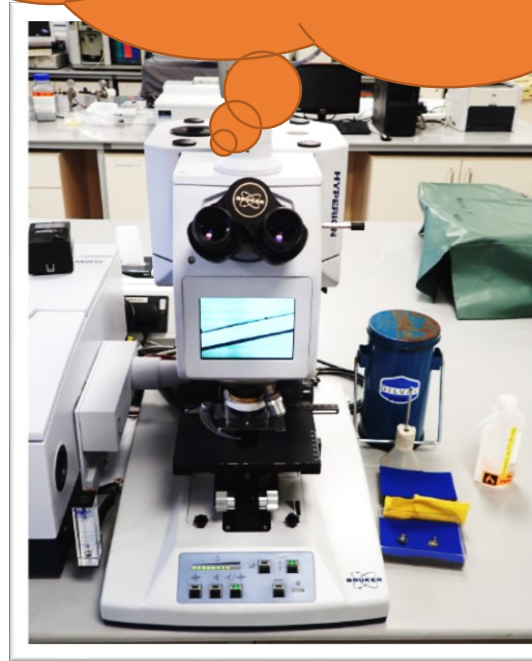
Regenerated Cellulose
(Rayon, Viscose etc.)



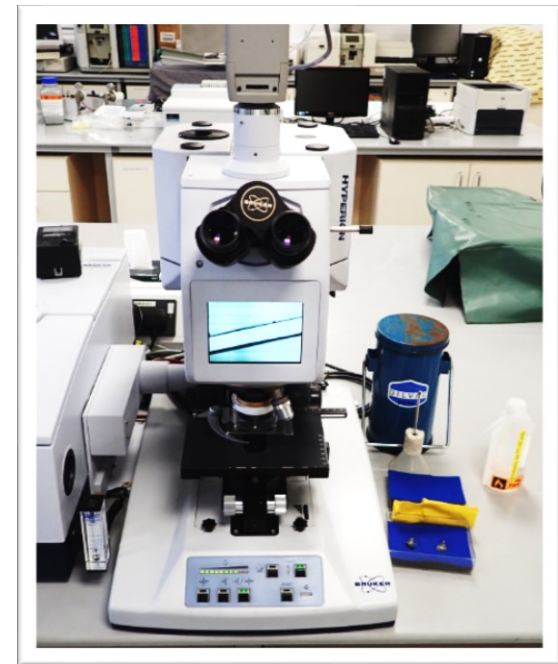
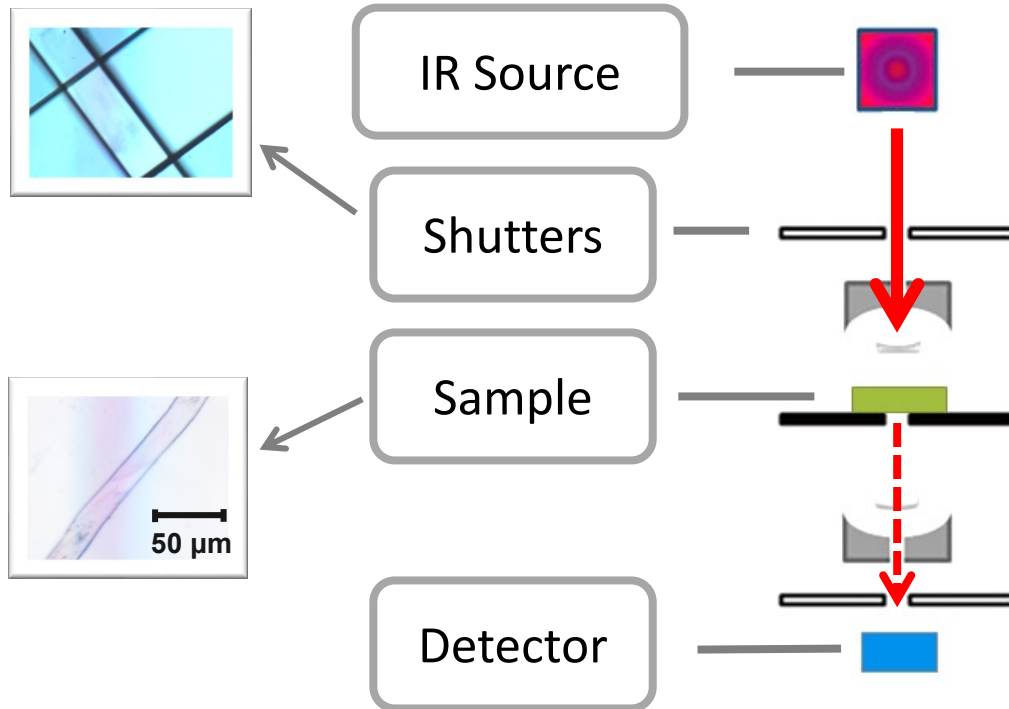
Natural Cellulose
(Cotton, Wood Pulp)



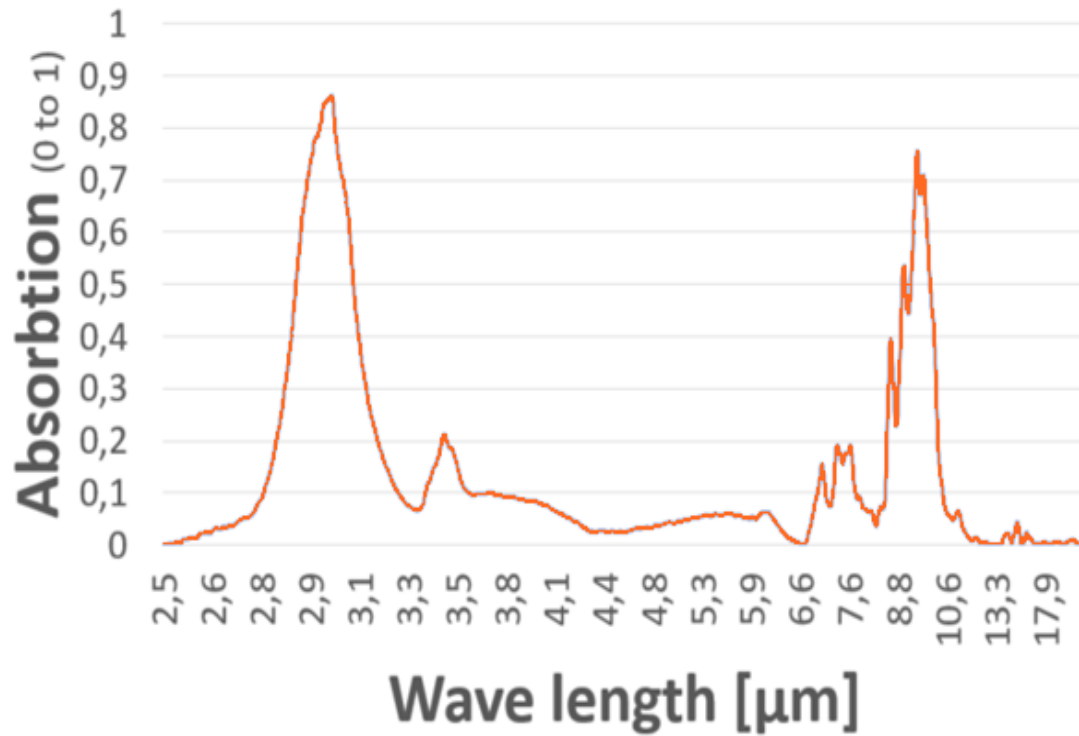
The analysis of microplastics is very difficult. As a consequence **questionable results** are reported. [Lendl et al. 2017]



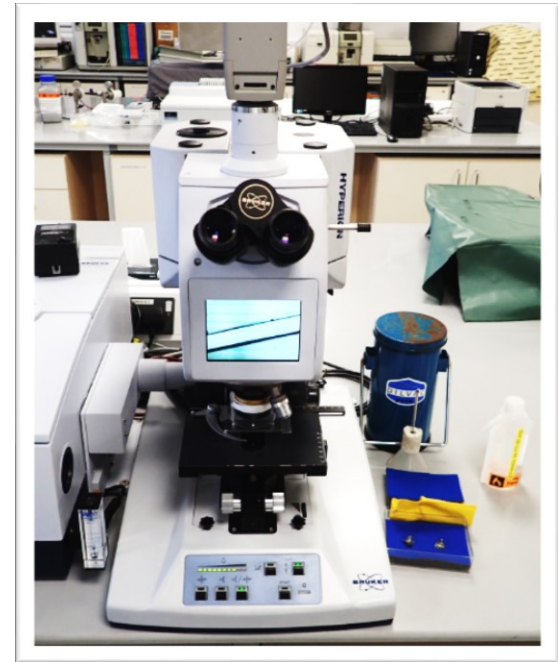
FT-IR Microscope (Transmission, ATR)



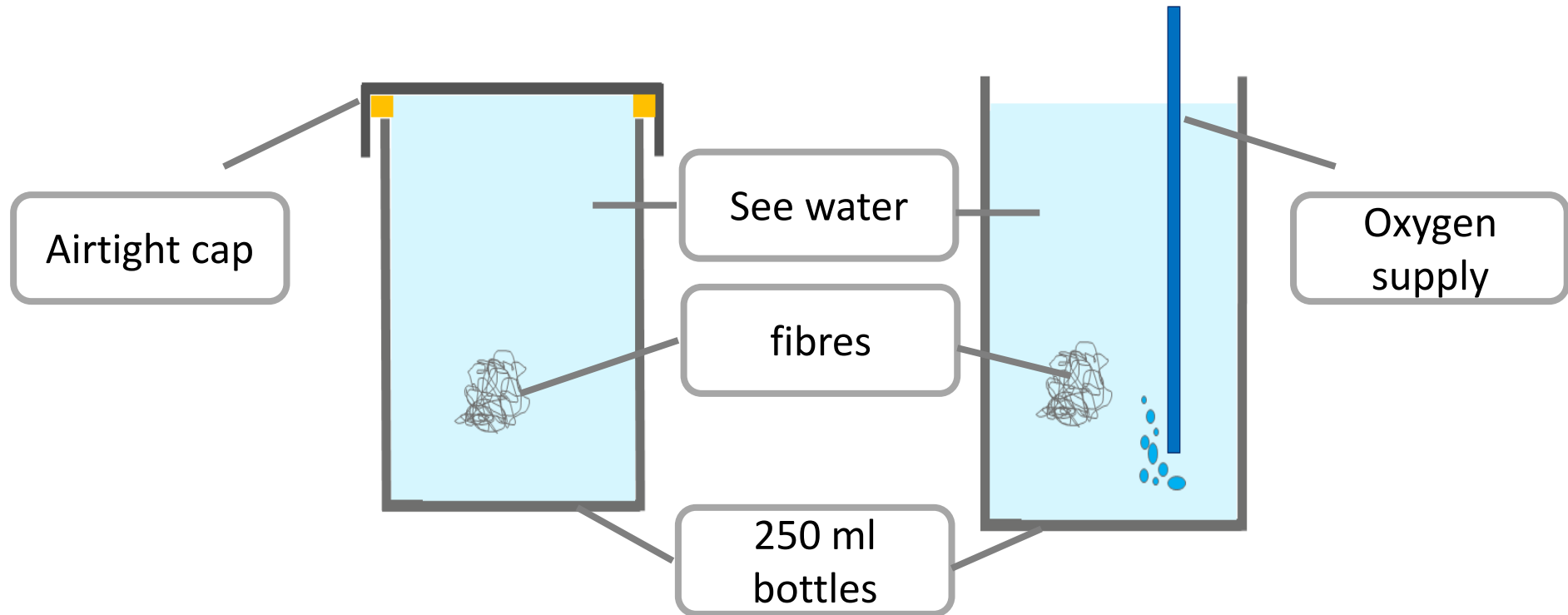
Bruker Hyperion 1000



FT-IR Microscope (Transmission, ATR)

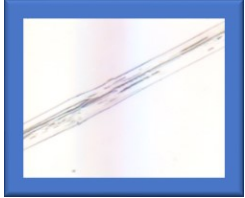


Bruker Hyperion 1000



Research question – Experimental design

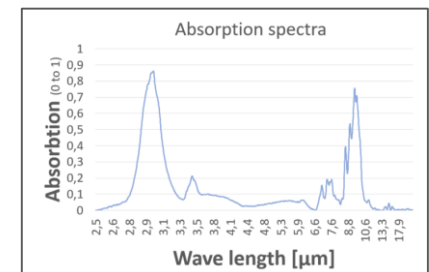
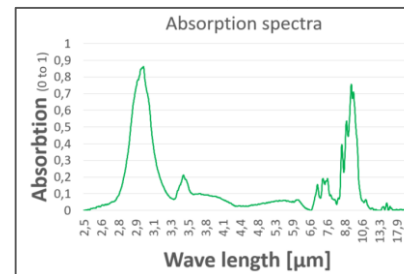
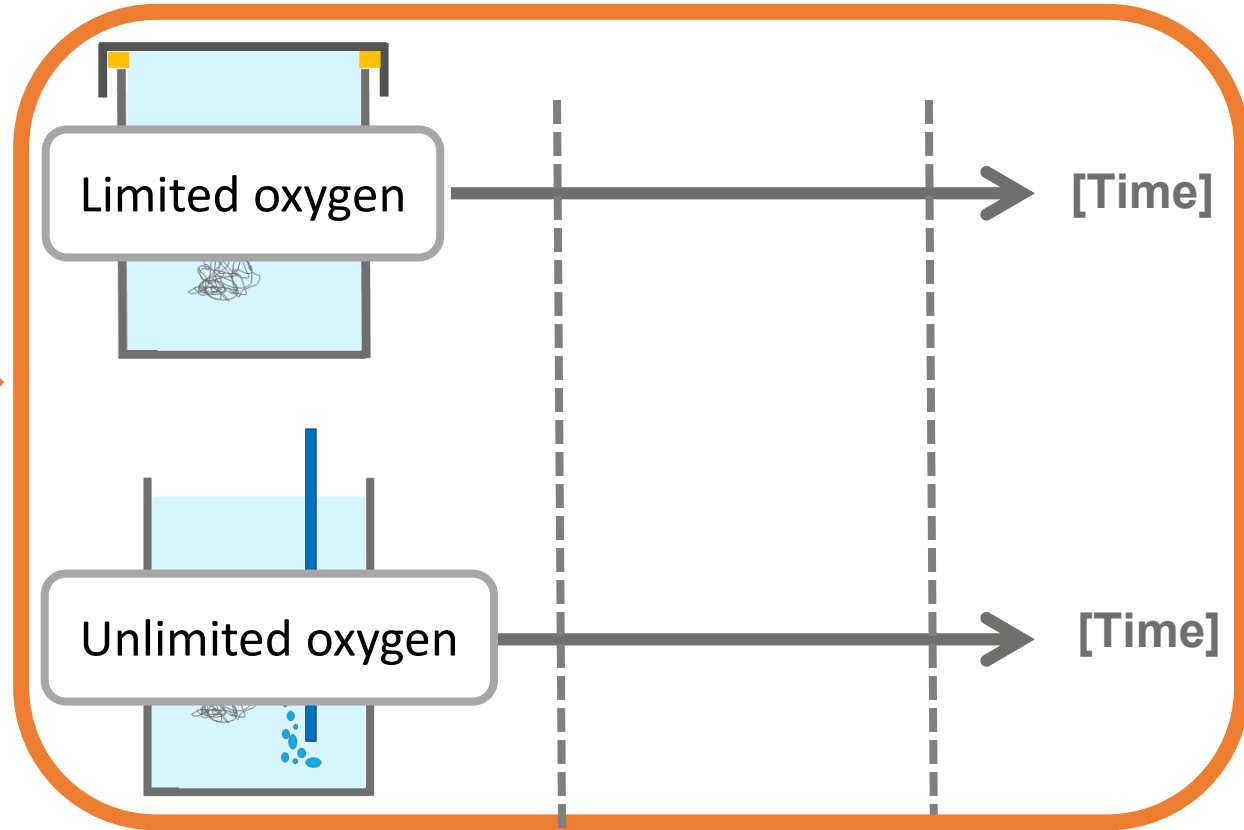
Raw Material

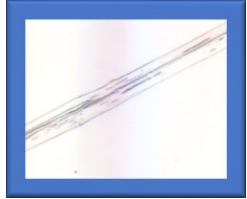


Regenerated Cellulose
(Rayon, Viscose etc.)



Natural Cellulose
(Cotton, Wood Pulp)

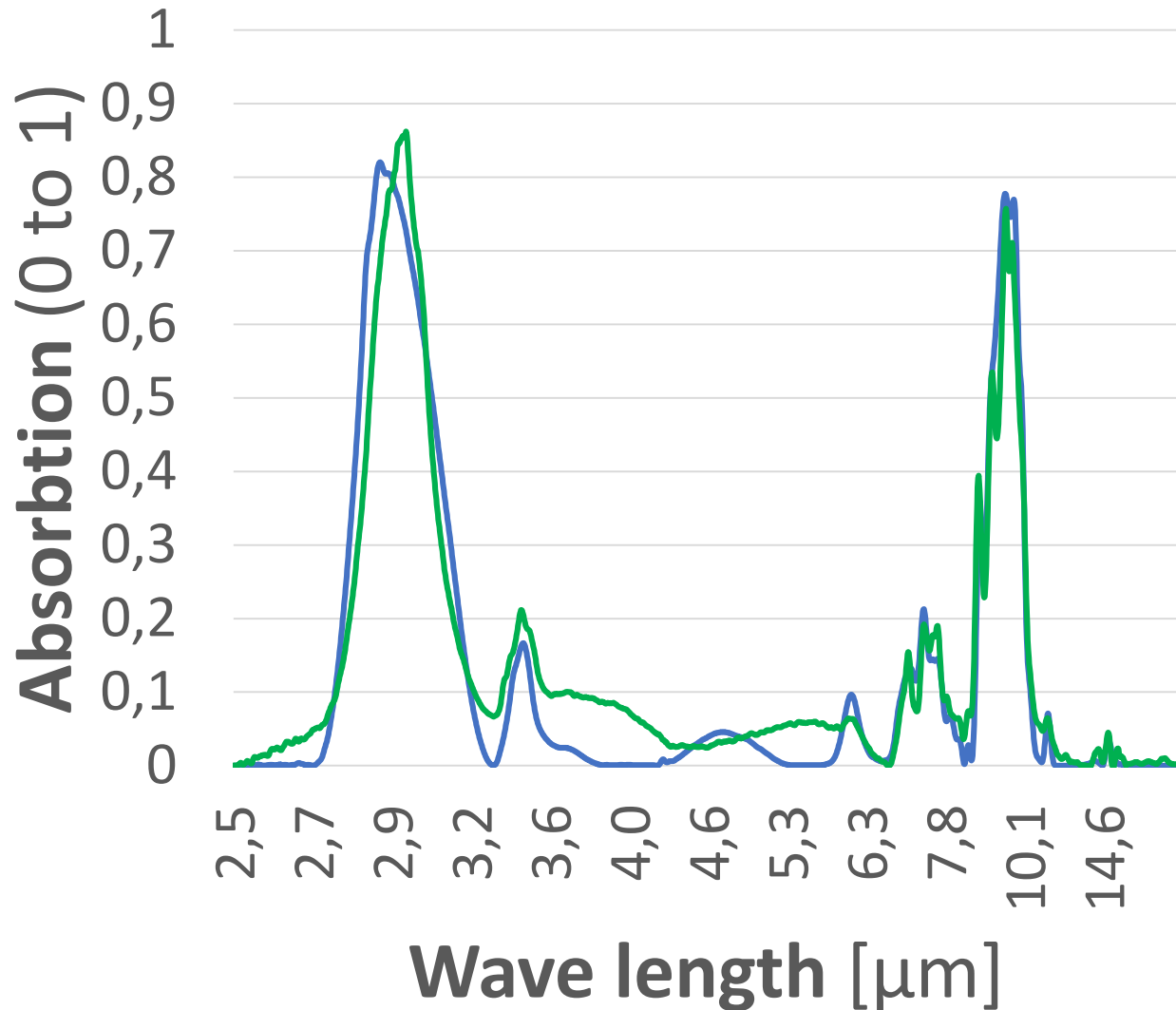




— Regenerated
Cellulose

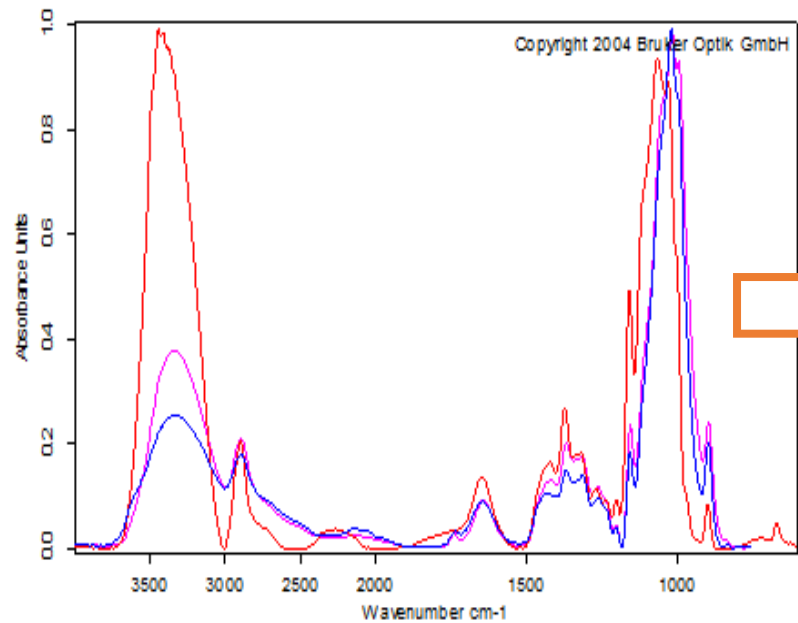


— Natural
Cellulose



Results – Library Search

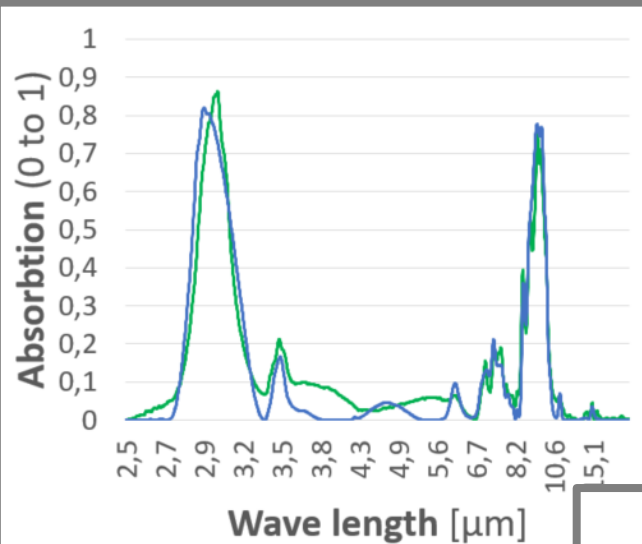
Search Library



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



Results – own database

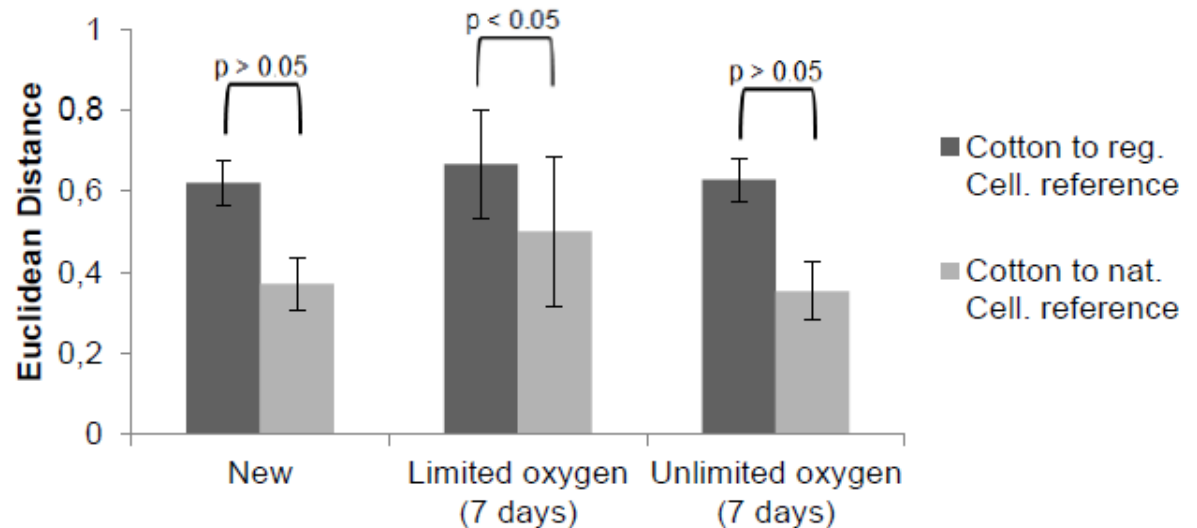


$$D = \sqrt{\sum_i (As(\lambda_i) - Ar(\lambda_i))^2}$$

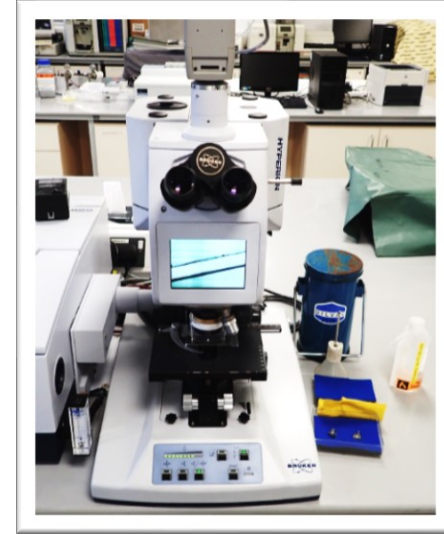
D: Euclidean Distance

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

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



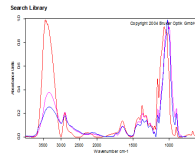
Summary so far ...



Transmission

ATR

Library Search

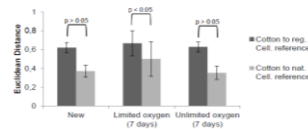


No results



No results

Own database



64 % - 91 %



18 % - 28 %

Problem formulation I

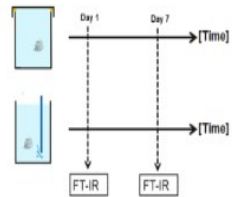


Fig. 10 Laboratory experiment exposure to the marine environment and the analysis via FT-IR

Time

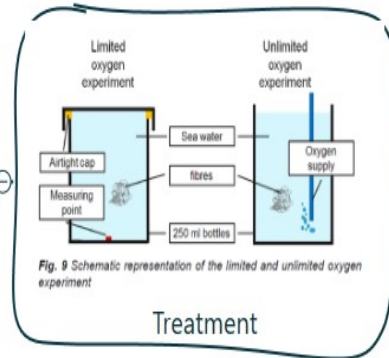
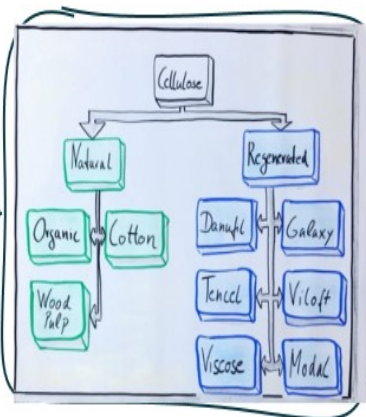


Fig. 9 Schematic representation of the limited and unlimited oxygen experiment

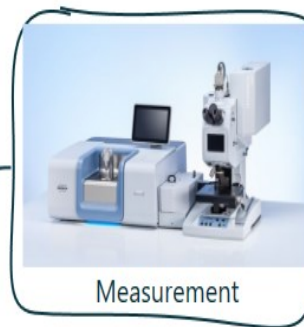
Treatment



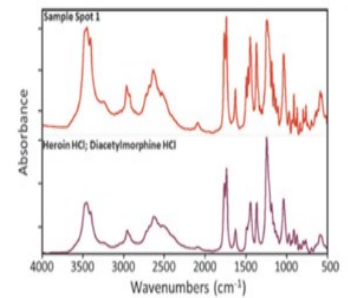
Nature ?



Analysis



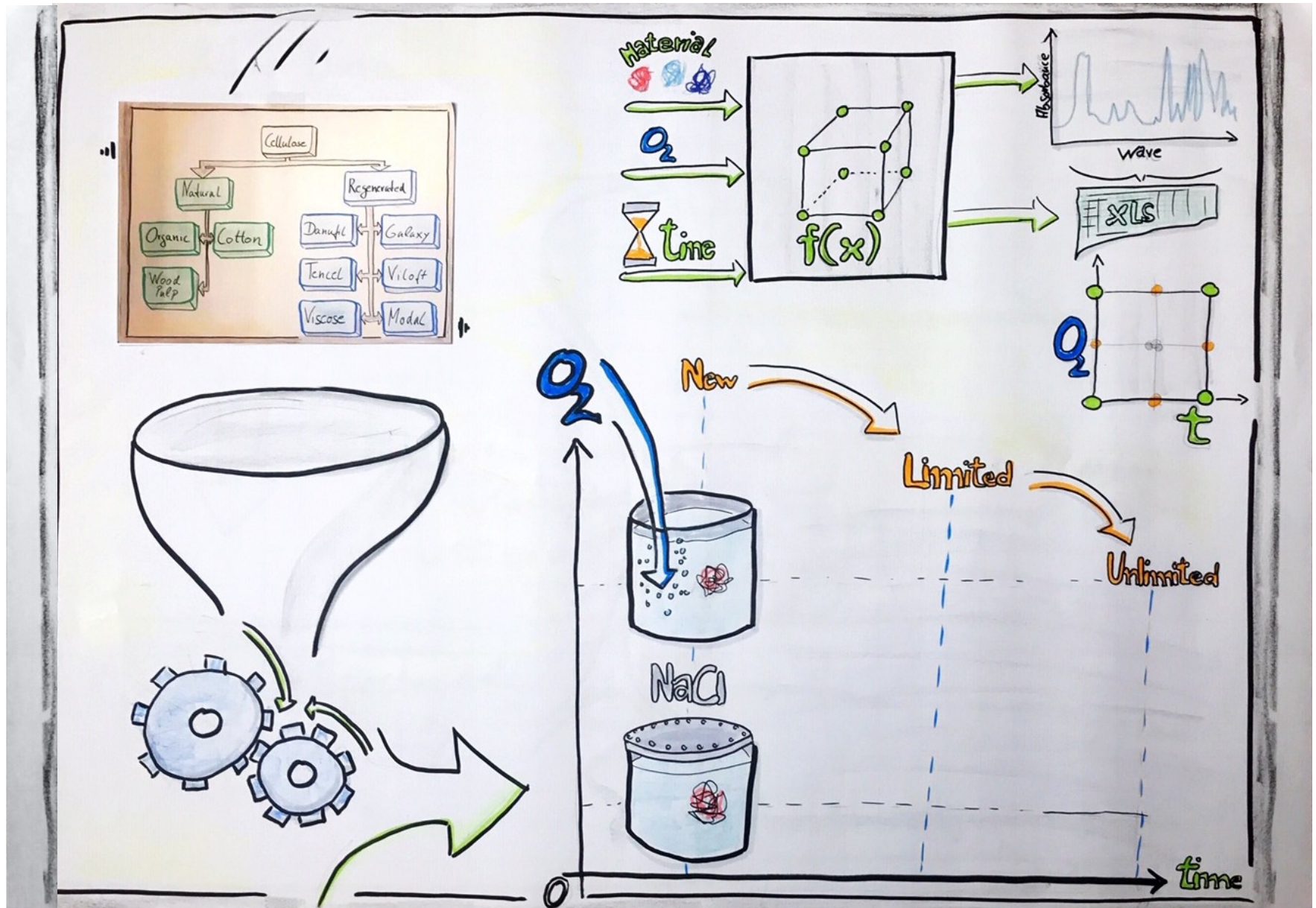
Measurement



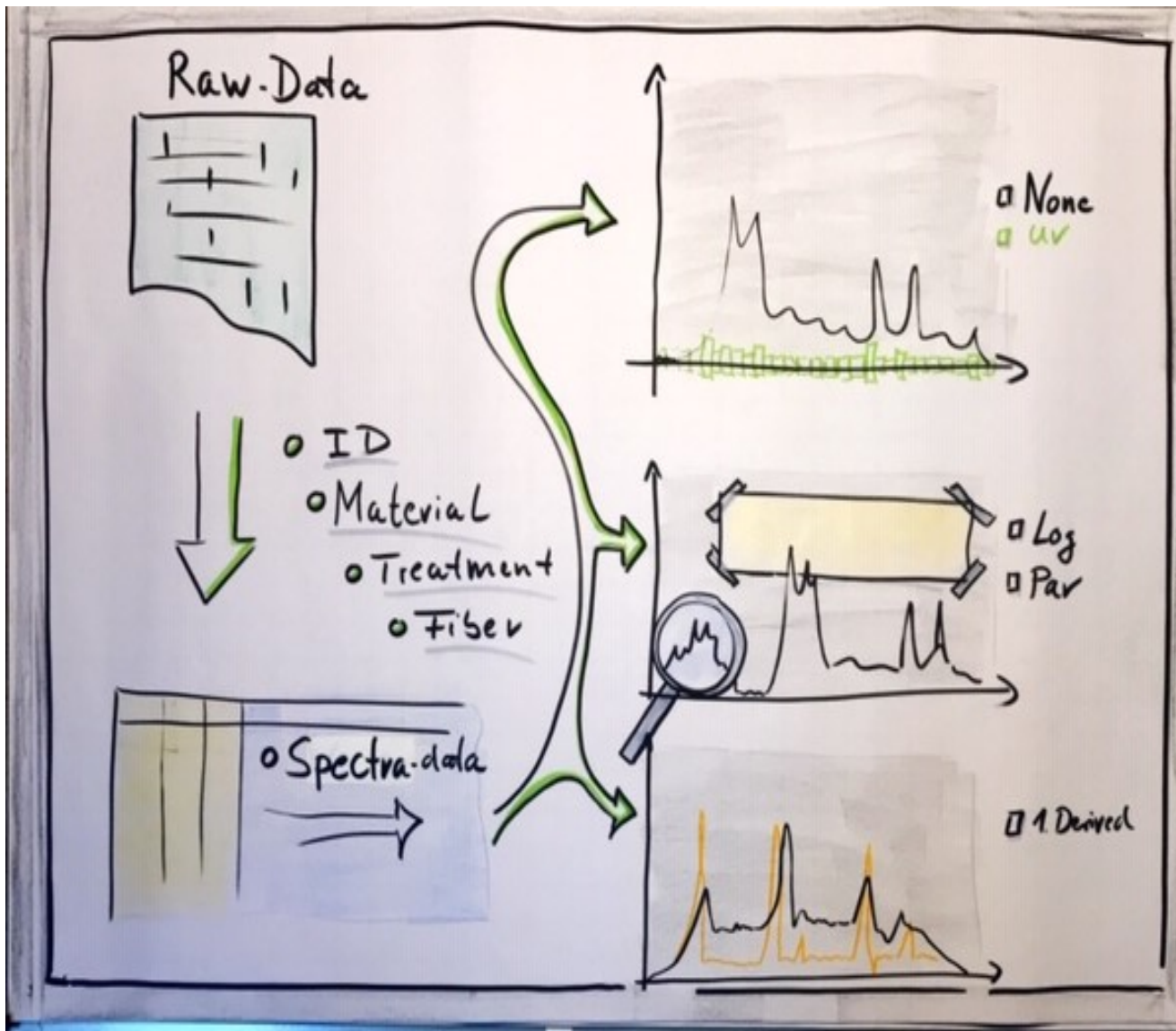
Spectra

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

1	2	3	4	5	6	7
Primary ID	TYPE	FIBRE	DEGRADATION	3996,37	3996,44	3996,52
UVIot_New_1	Regenerated Cellulose	UVIot	New	0,01257	0,01364	0,01471
UVIot_New_2	Regenerated Cellulose	UVIot	New	0	7e-05	0,00214
UVIot_New_3	Regenerated Cellulose	UVIot	New	-0,0031	-0,00099	-0,00087
Galaxy_New_1	Regenerated Cellulose	Galaxy	New	-0,00733	-0,00489	-0,00446
Galaxy_New_2	Regenerated Cellulose	Galaxy	New	-0,00012	-0,0001	-9e-05
Galaxy_New_3	Regenerated Cellulose	Galaxy	New	0,00021	0,00022	0,00022
Danufi_New_1	Regenerated Cellulose	Danufi	New	0,00141	0,00148	0,00154
Danufi_New_2	Regenerated Cellulose	Danufi	New	0	3e-05	7e-05
Danufi_New_3	Regenerated Cellulose	Danufi	New	8e-05	9e-05	0,0001
Viscose_New_1	Regenerated Cellulose	Viscose	New	-0,00253	-0,0024	-0,00220
Viscose_New_2	Regenerated Cellulose	Viscose	New	0	6e-05	0,00013
Viscose_New_3	Regenerated Cellulose	Viscose	New	0,0018	0,0018	0,0018
Model_New_1	Regenerated Cellulose	Model	New			
Model_New_2	Regenerated Cellulose	Model	New			

Worksheet

Select data Overview Variables Observations Transform Lag Expand Scale Spreadsheet

Variables: 1761, selected: 0

Primary ID	Type	Block	Mo...	Avg.	Std. d...
X 3996,37	UV	---	1	0,000...	0,00...
X 3994,44	UV	---	1	0,000...	0,00...
X 3992,52	UV	---	1	0,000...	0,00...
X 3990,59	UV	---	1	0,000...	0,00...
X 3988,66	UV	---	1	0,001...	0,00...
X 3986,73	UV	---	1	0,001...	0,00...

Set scaling

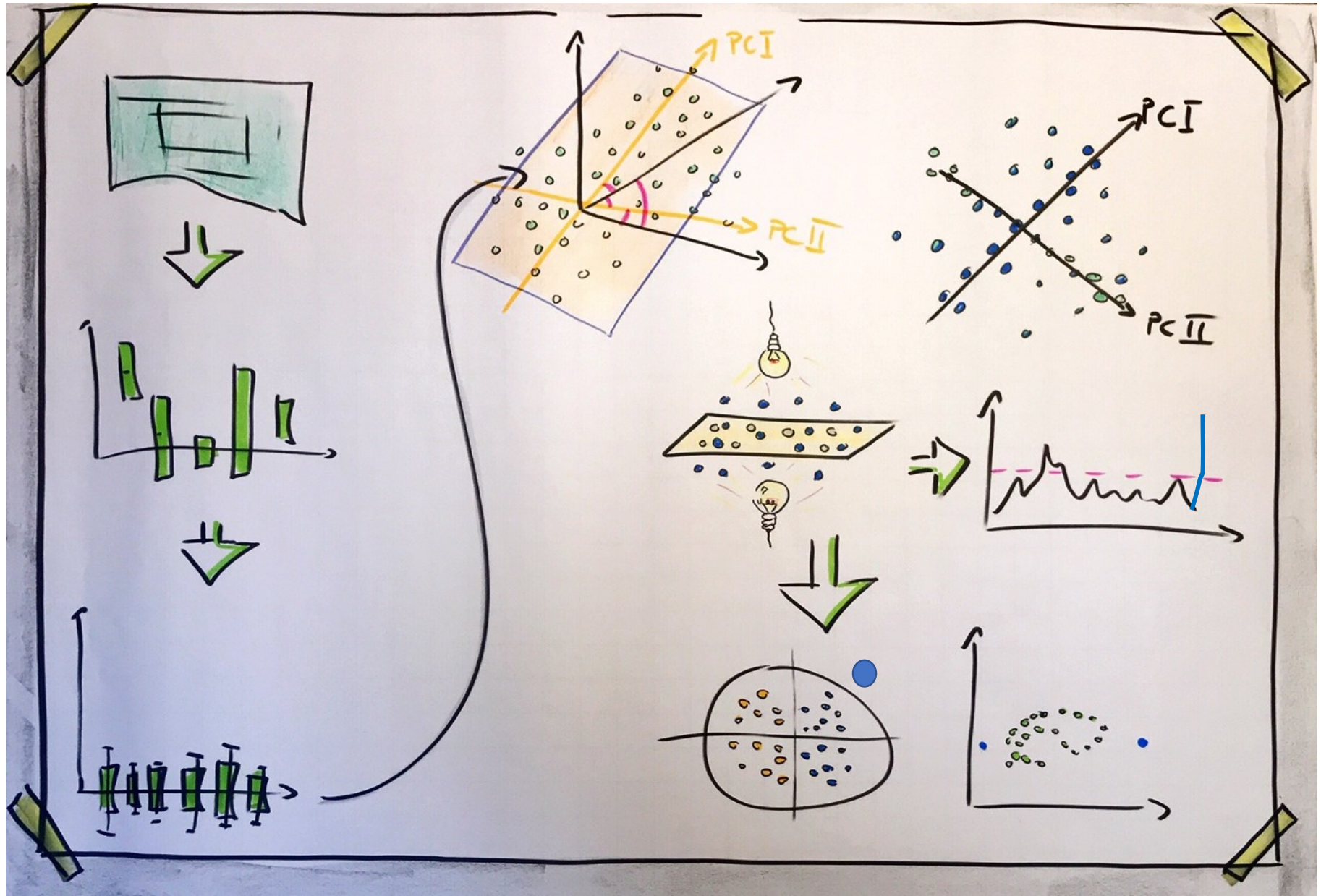
Type: UV Set

Block: 1 Set

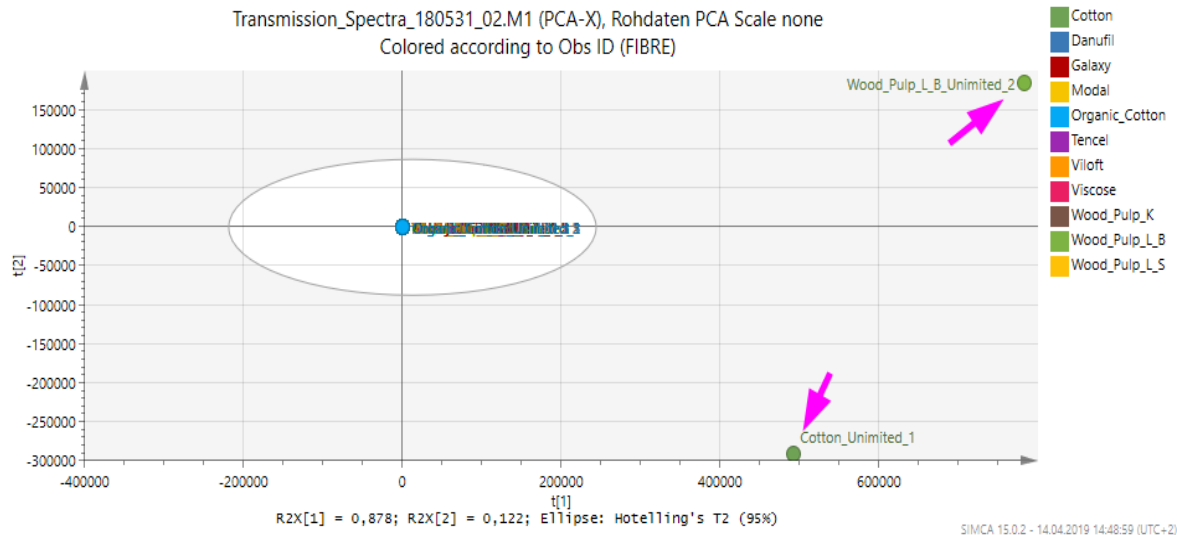
Block weight: 1/sqrt Set

Spectral filters* Time series filters* Dataset summary* Missing value map* Trimming Spectra overview

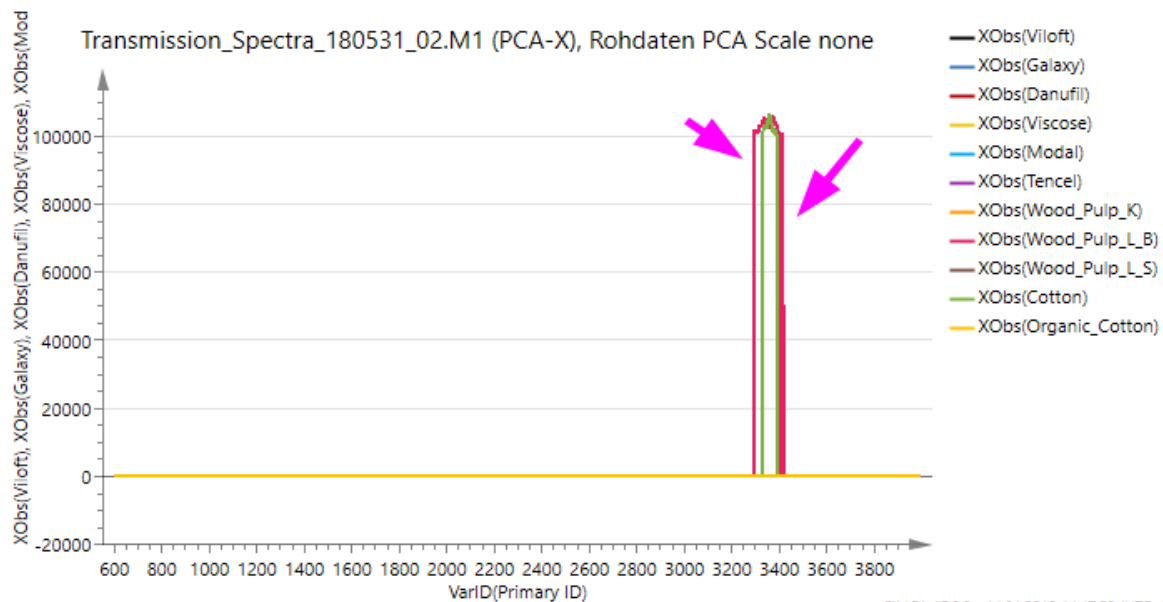
Very brief instruction to the PCA principles



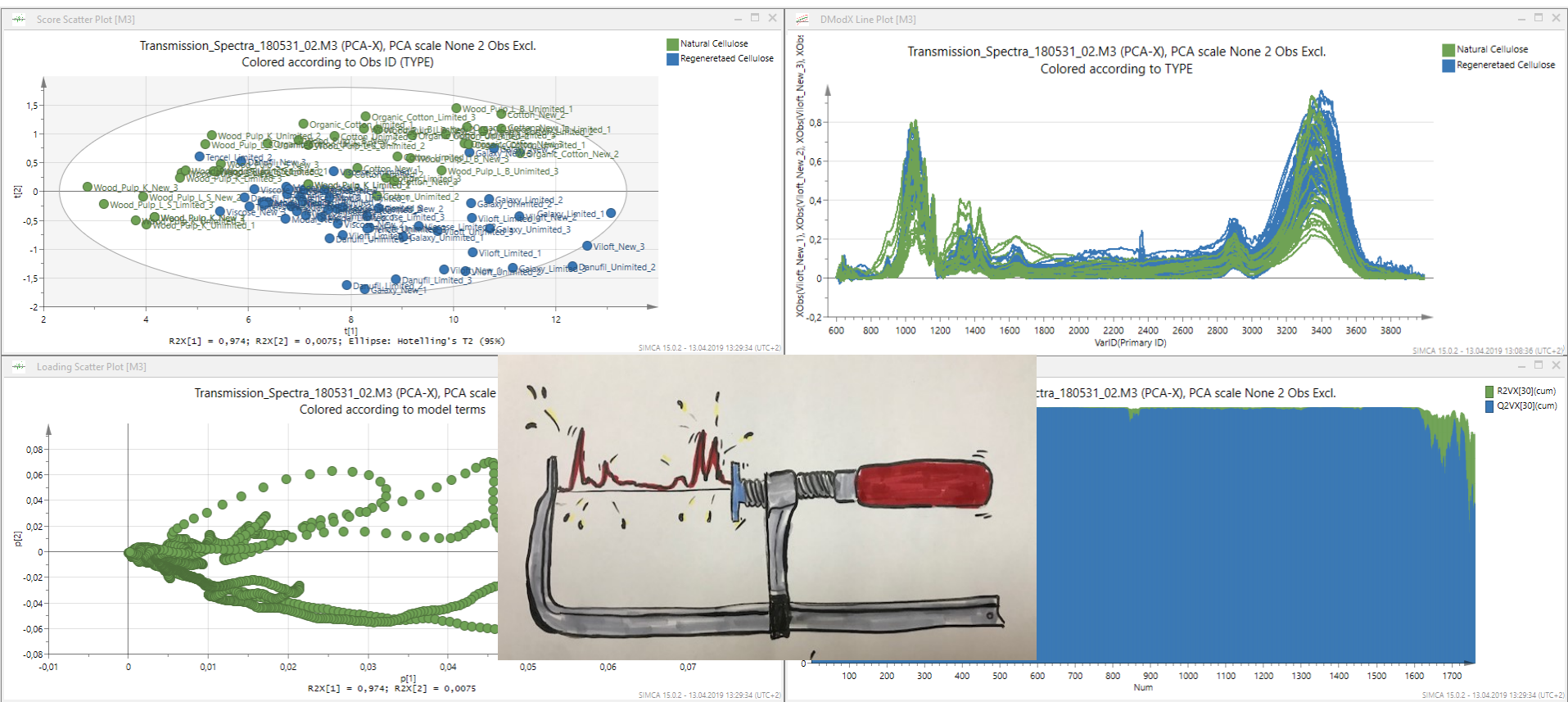
Check the data due to abnormalities with PCA-Model (no Scaling)



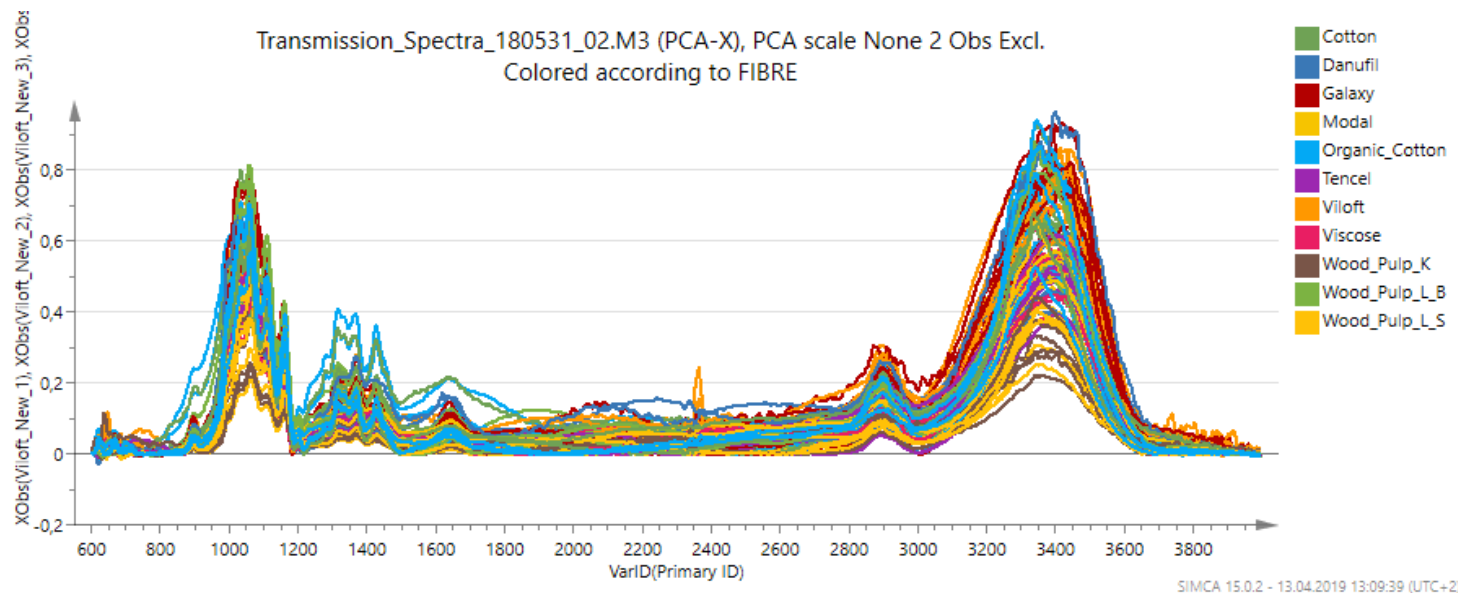
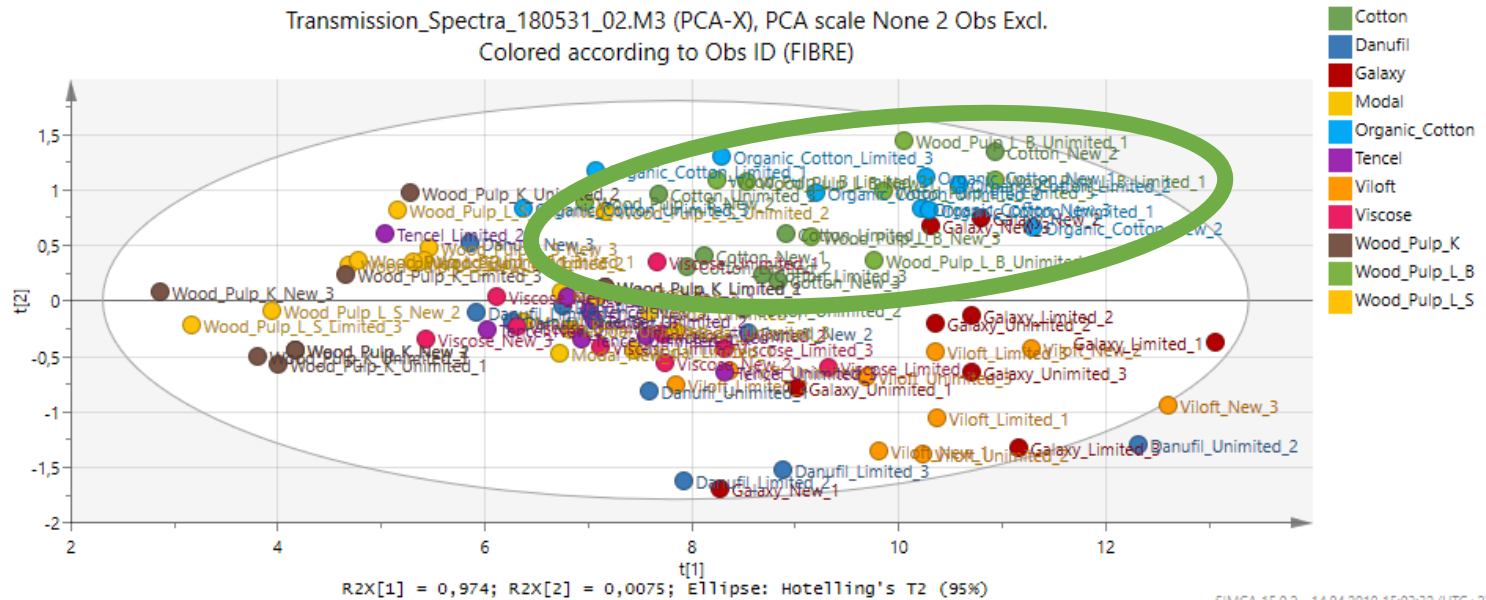
2 Observations
excluded!



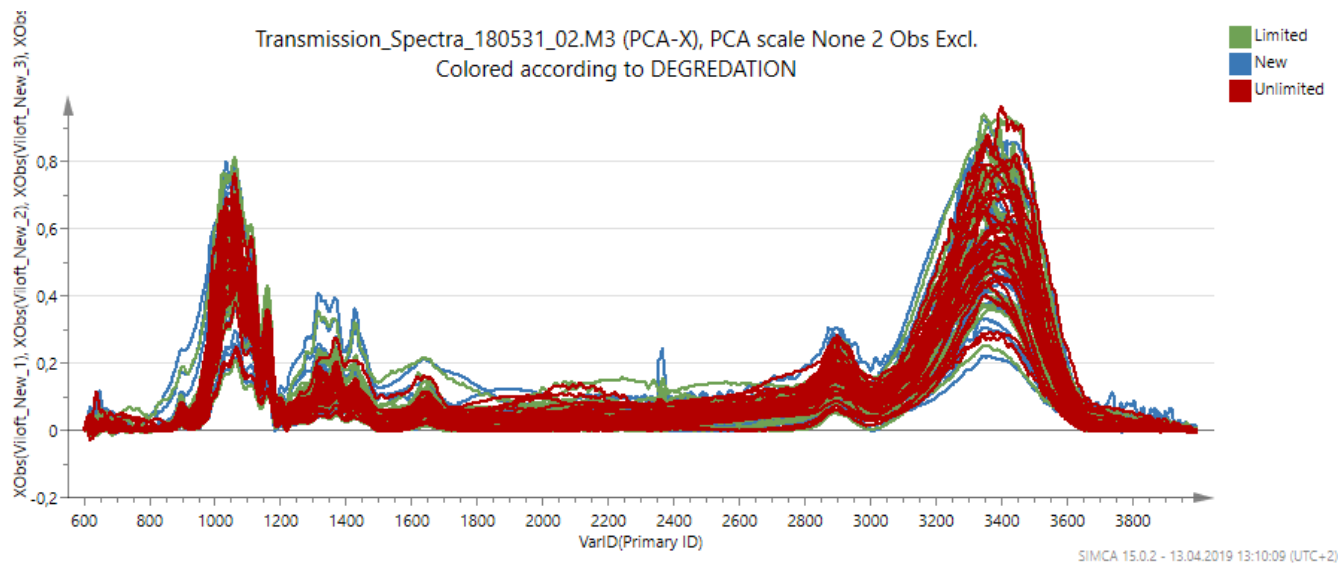
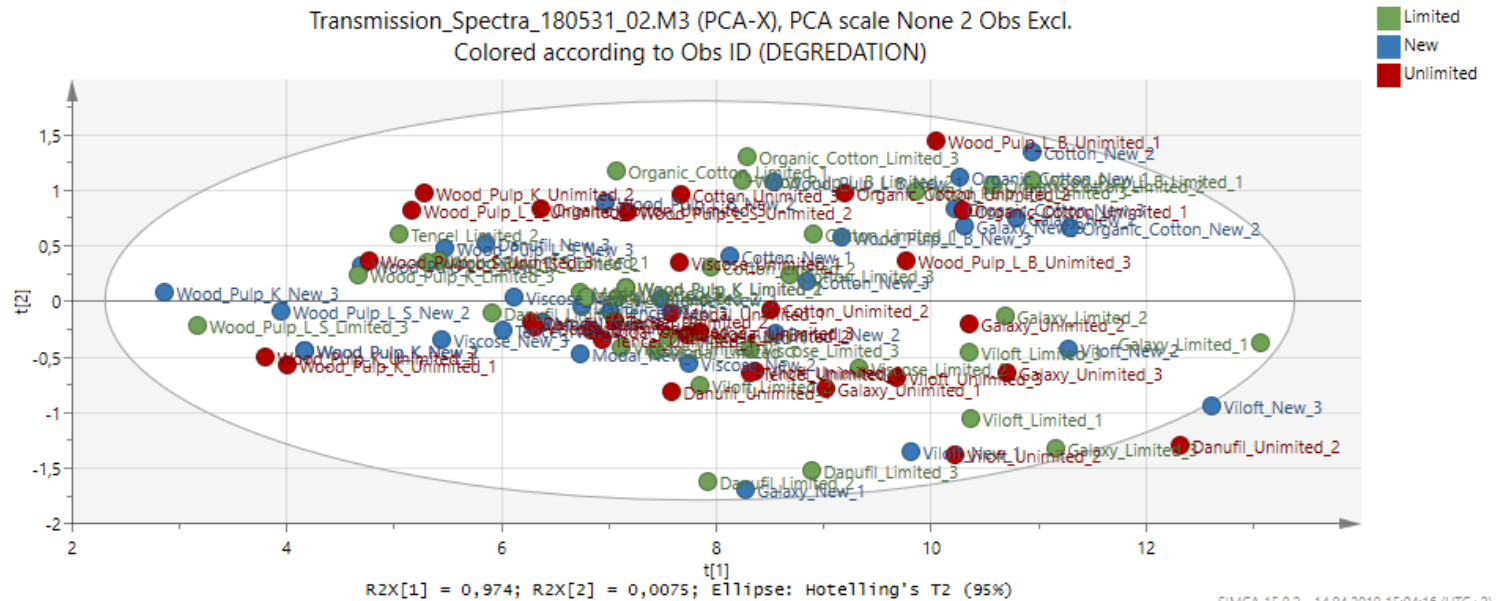
Fast overview for displaying imported data in SIMCA (without outliers)



Corresponding Plots - Example data reduction I → Cellulose Type

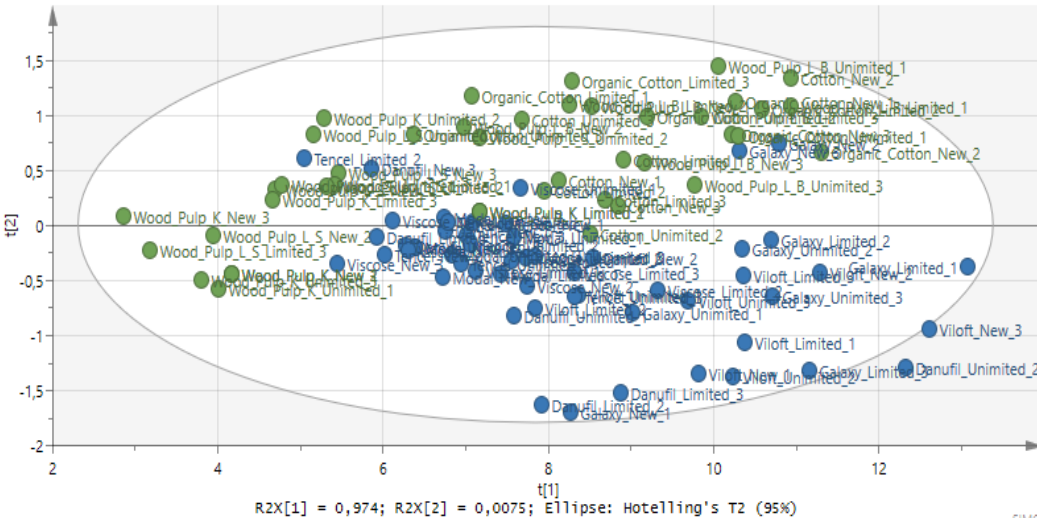


Corresponding Plots - Example data reduction II → Treatment



Basic Analysis

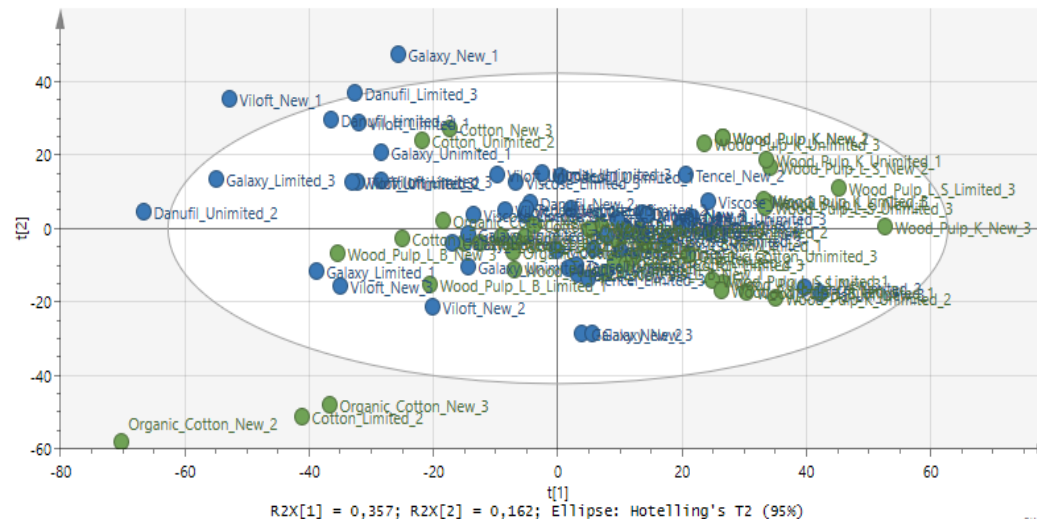
Transmission_Spectra_180531_02.M3 (PCA-X), PCA scale None 2 Obs Excl.
Colored according to Obs ID (TYPE)



- PCA Model
- 2 Obs. excluded
- no scaling

SIMCA 15.0.2 - 13.04.2019 11:56:06 (UTC+2)

Transmission_Spectra_180531_02.M4 (PCA-X), PCA Scale UV 2 Obs Excl
Colored according to Obs ID (TYPE)



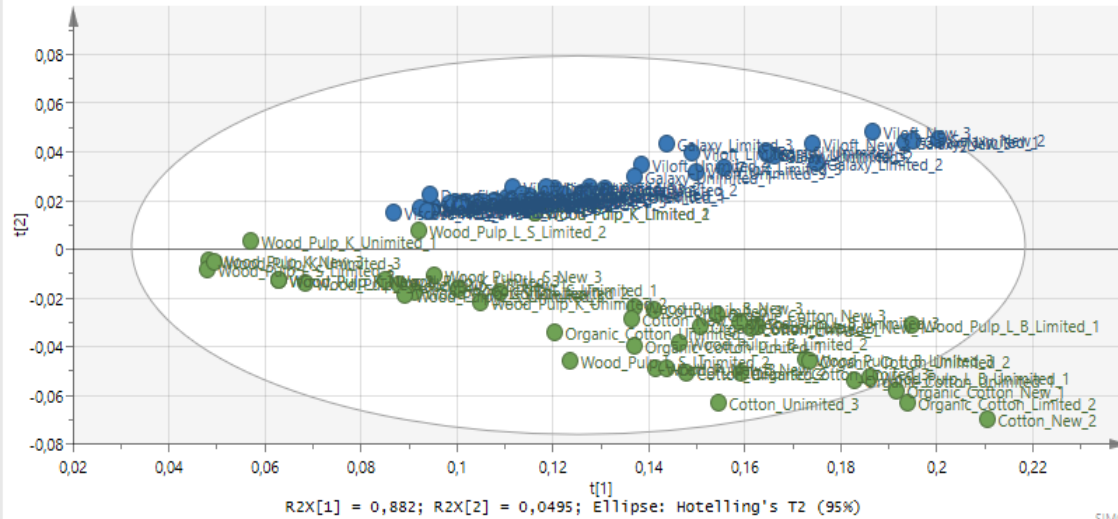
- PCA Model
- 2 Obs. excluded
- UV scaling

SIMCA 15.0.2 - 13.04.2019 11:56:34 (UTC+2)

Basic Analysis – with filter 1st Derv.

Transmission_Spectra_180531_02.M9 (PCA-X), PCA Filter 1 Derv. 2 Obs Excl. Scale None
Colored according to Obs ID (TYPE)

■ Natural Cellulose
■ Regeneretaed Cellulose

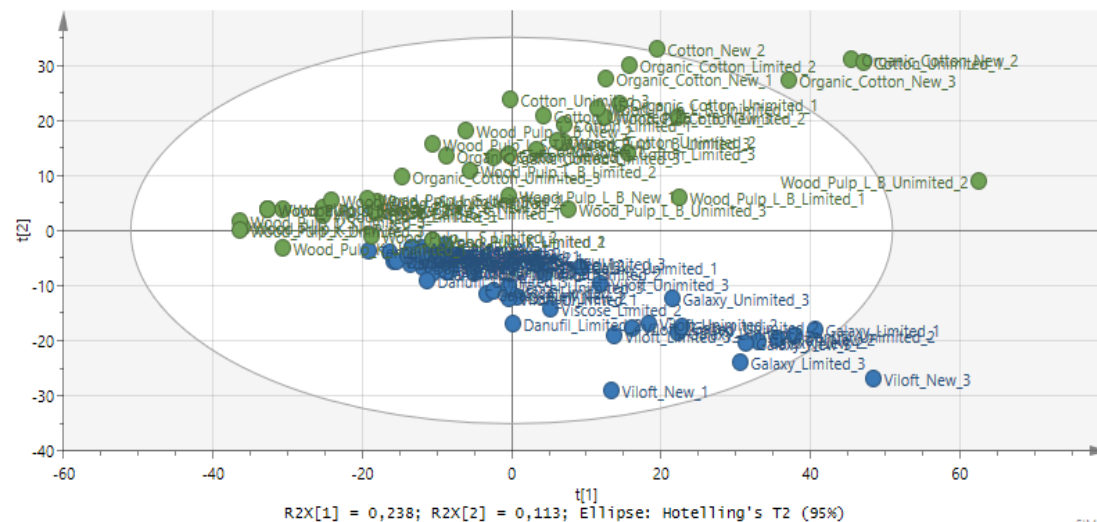


SIMCA 15.0.2 - 13.04.2019 11:54:22 (UTC+2)

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

Transmission_Spectra_180531_02.M7 (PCA-X), PCA X Filter 1 Derv. Scale UV
Colored according to Obs ID (TYPE)

■ Natural Cellulose
■ Regeneretaed Cellulose



SIMCA 15.0.2 - 13.04.2019 11:52:54 (UTC+2)

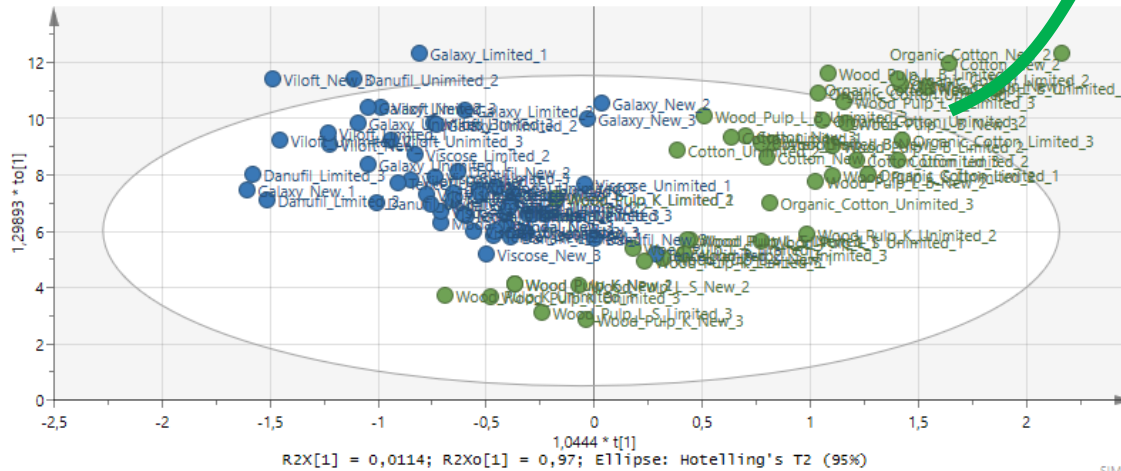
- PCA **Class** - Model
- 2 Obs. Excluded
- Filter 1st Derv.
- **UV** scaling



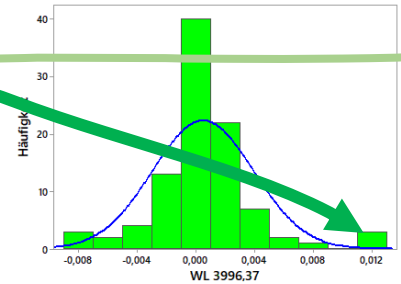
97,94 %

Advanced Analysis with OPLS-DA

Transmission_Spectra_180531_02.M5 (OPLS-DA), OPLS DA Scale None, 2 Obs Excl.
Scaled proportionally to R2X
Colored according to Obs ID (TYPE)

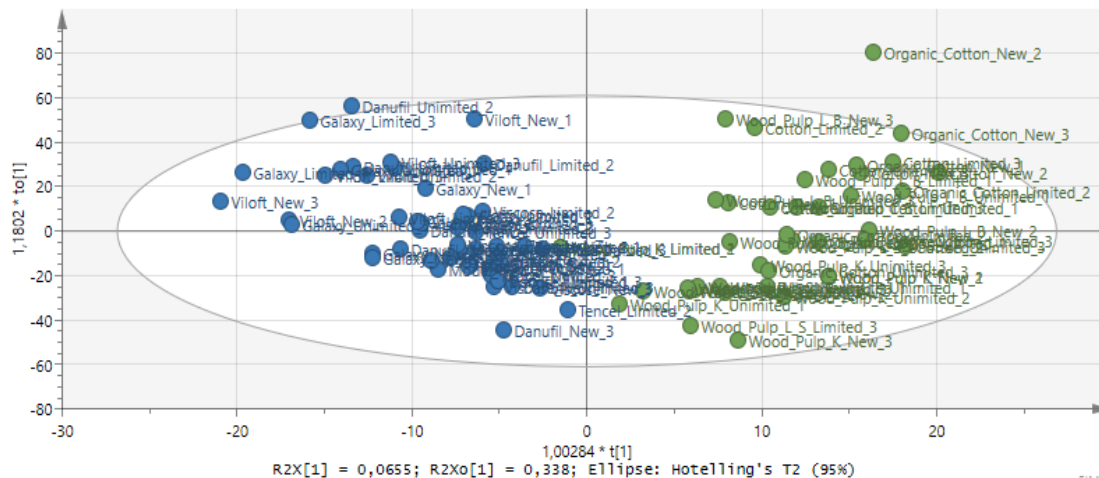


■ Natural Cellulose
■ Regenerated Cellulose



OPLS-DA Model
2 Obs. excluded
no scaling

Transmission_Spectra_180531_02.M6 (OPLS-DA), OPLS DA Scale UV, 2 Obs Excl.
Scaled proportionally to R2X
Colored according to Obs ID (TYPE)



■ Natural Cellulose
■ Regenerated Cellulose

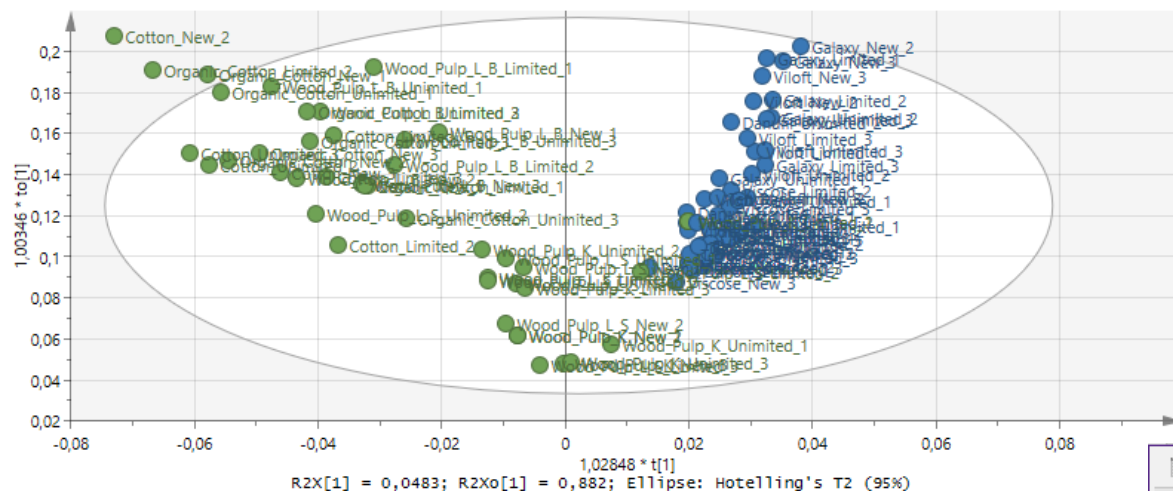
OPLS-DA Model
2 Obs. excluded
UV scaling

SIMCA 15.0.2 - 13.04.2019 11:57:18 (UTC+2)

SIMCA 15.0.2 - 13.04.2019 11:57:48 (UTC+2)

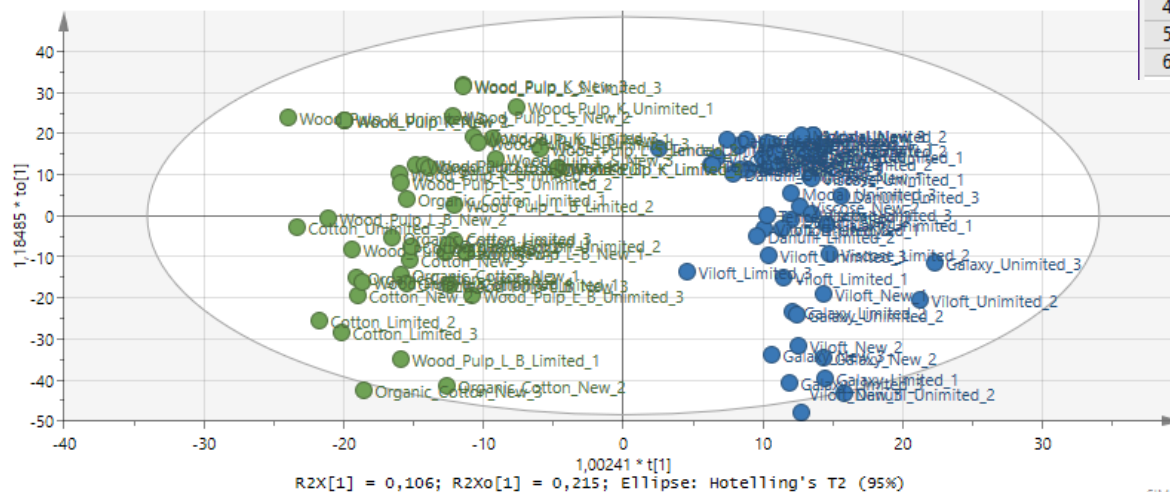
Advanced Analysis

Transmission_Spectra_180531_02.M10 (OPLS-DA), OPLS-DA Filter 1 Deriv. 2Obs Excl. Scale None
Scaled proportionally to R2X
Colored according to Obs ID (TYPE)



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

Transmission_Spectra_180531_02.M11 (OPLS-DA), OPLS-DA Filter 1 Deriv. 2Obs Excl. Scale UV
Scaled proportionally to R2X
Colored according to Obs ID (TYPE)



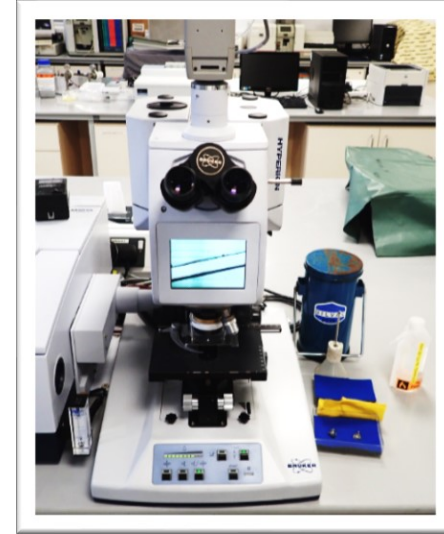
- OPLS-DA Model
- Filter 1st Deriv.
- 2 Obs. Excluded
- **UV** scaling

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				



100 %

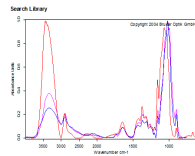




Transmission

ATR

Library Search

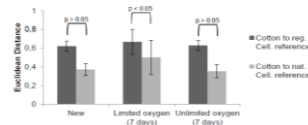


No results



No results

Own database

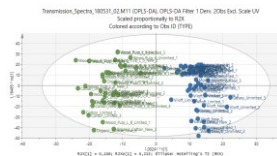


64 % - 91 %



18 % - 28 %

Multivariate data analysis



100 %
(except to outliers)



97 %

Outlook







Frederik Obermeier (M.Sc.)
R&D Plastic Engineering
Research Fellow and Ph.D. Student

Hochschulstraße 1
D-83024 Rosenheim

Email: frederik.obermeier@th-rosenheim.de

Tel: +49 8031 805-2266

Web: www.th-rosenheim.de/forschung-entwicklung

M O S E R S T E F A N
 **PROCESS**
OPTIMIZATION



Dipl.-Ing. (FH) Stefan Moser
Consultancy, Analysis & Training

Weberweg 3
D-83131 Nußdorf am Inn

Email: info@stefan-moser.com

Mobil: +49 (0) 170 811 240 3

Web: www.stefan-moser.com