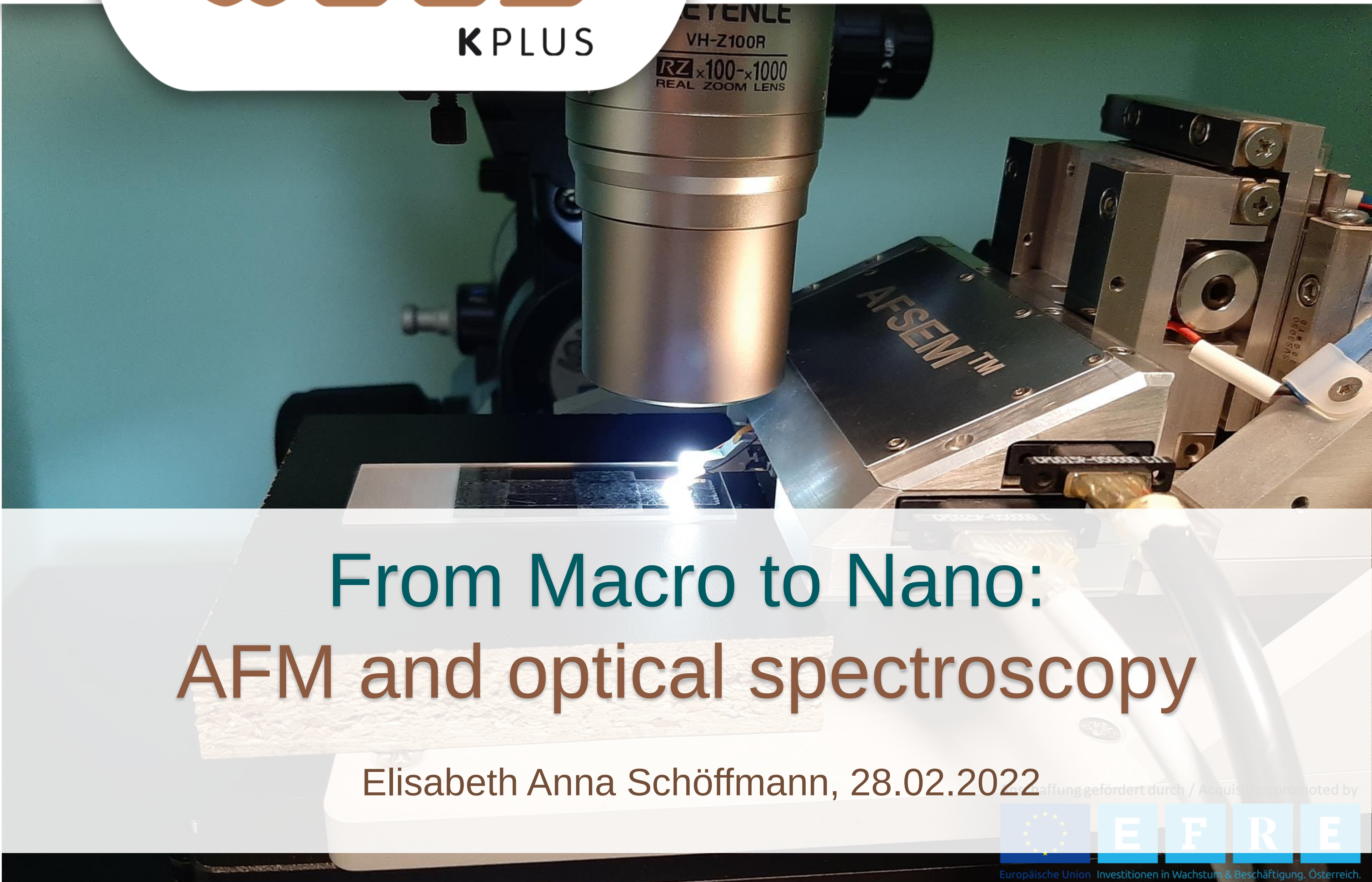
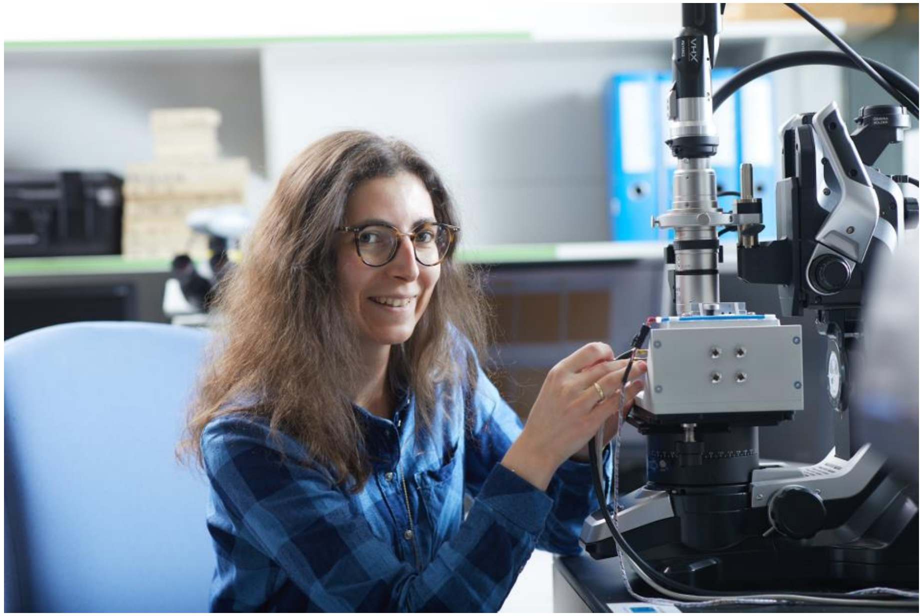


From Macro to Nano:
AFM and optical spectroscopy

Elisabeth Anna Schöffmann, 28.02.2022

Anschaffung gefördert durch / Acquisition promoted by
Europäische Union Investitionen in Wachstum & Beschäftigung. Österreich.

Introduction



Elisabeth Anna Schöffmann

- BSc and MSc at TU Graz
- Since 07/2017 project manager at Wood K plus in St. Veit an der Glan
- Start dissertation project 10/2019 with invest of a new device (AFM)

Wood Kplus Area 3 – St.Veit/Glan

- Headquarter at Linz
- Industry-related research
- Surface characterization
- Invention of new surface characterization methods
- Expertise in IR-spectroscopy and multivariate data analysis

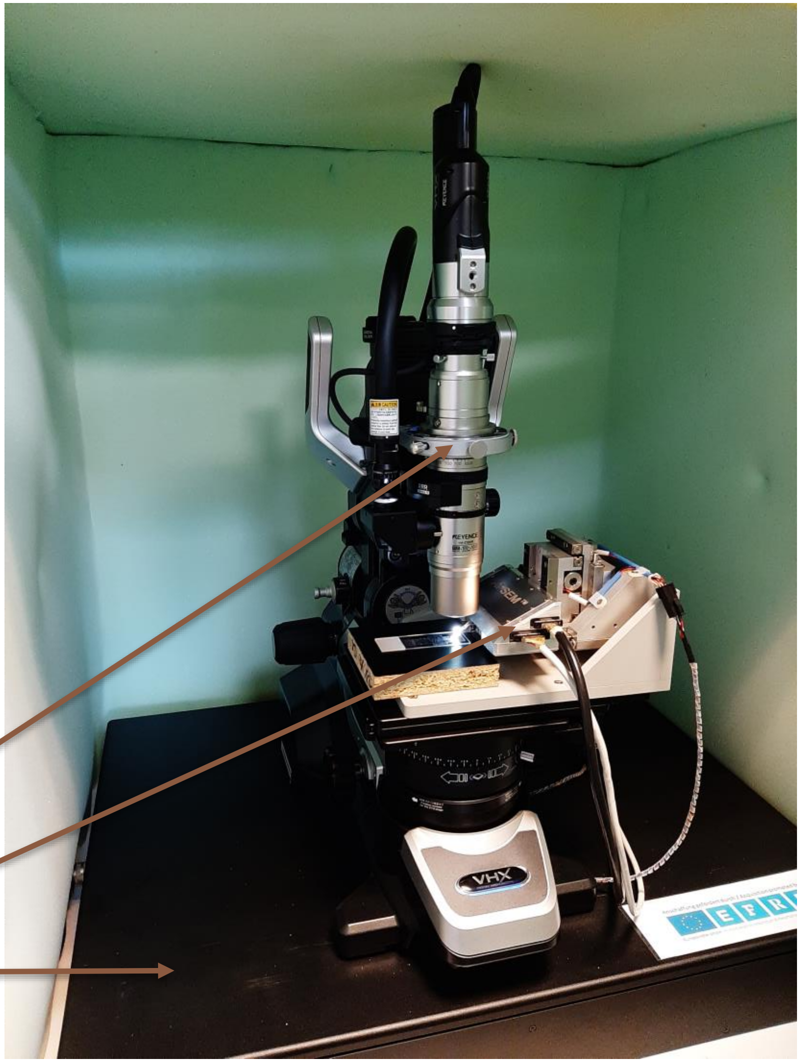
Agenda

- Overview and Theory
  - AFM
  - IR-Microscope
  - MVA/PCA
- Research Question/ Problem
- Work packages/ Key problems
- Outlook

Atomic Force Microscope

Measuring principle:

- Mechanical imaging instrument
- Cantilever (needle on oscillating leaf spring)
- Scans over the sample surface
- Laser alignment
- Topography data



Keyence Digitalmikroskop VHX 950F

AFSEM from Getec (now QD-Microscopy)

Vibration damping plate

The AFM from QD-microscopy (former GETec)

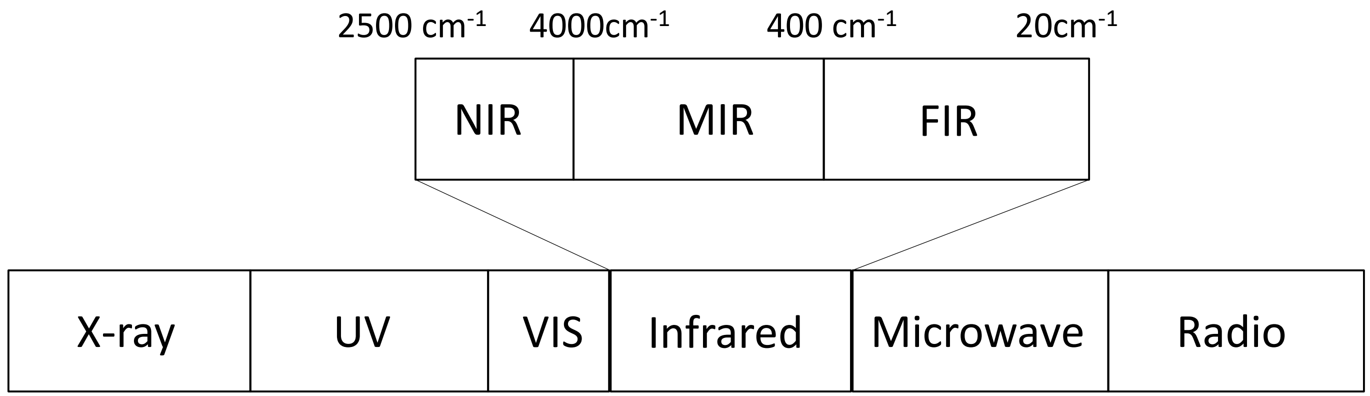
- For the integration into a SEM developed
- No vacuum with our setup
- Combination of AFM and microscopy
- No laser adjustment needed due to self-sensing cantilever technology
- Easy handling
- Max. measurement range

  30 x 30 µm

AFM installed on the digital microscope

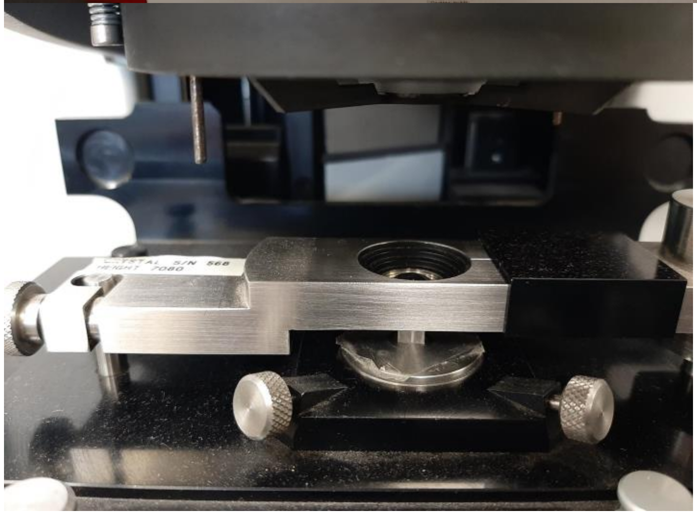
IR-spectroscopy - Basics

- Optical spectroscopy – wide range NIR, MIR, UV-VIS
- Focus on MIR: 4000-600 cm^{-1}
- Exitation of molecular vibrations
- Vibrational transitions – energies are characteristic for functional groups
- Good chemical classification and differentiation



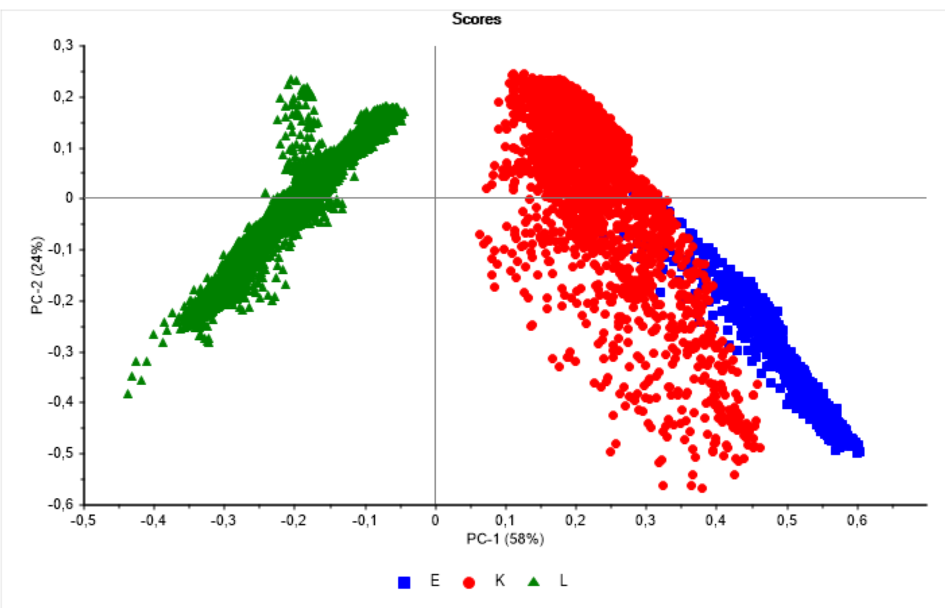
IR-microscope PerkinElmer Spotlight 400

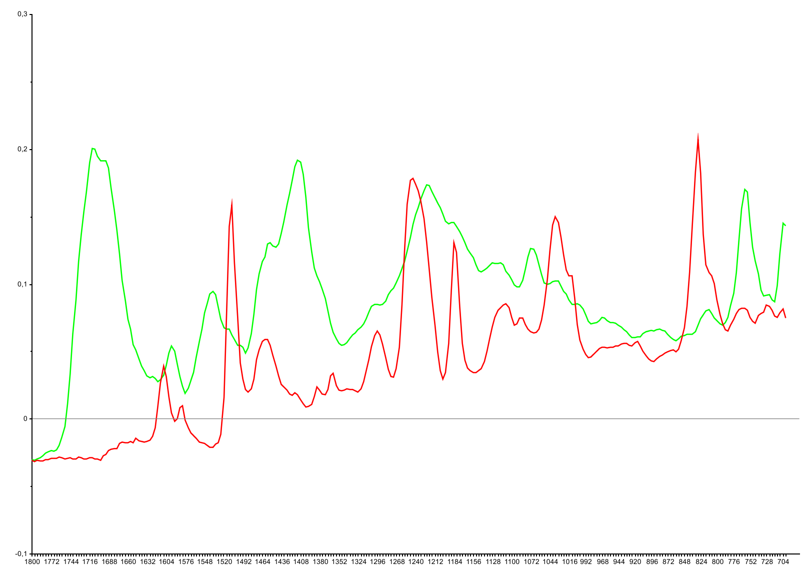
- Transmission and reflection possible
- Spectral range:
  4000-720 cm^{-1}
- Spatial resolution: 3,1 µm
- Spectral resolution: 4 cm^{-1}
- Measurement area in ATR imaging max. 400 x 400 µm
- Integrated PCA analysis
- Chemical characterization of solid samples and small defect areas



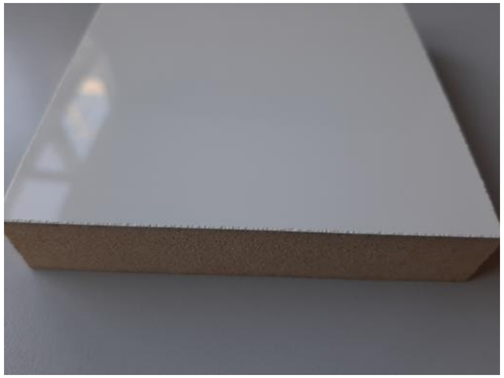
Multivariate Analysis (MVA) - Basics

- More than 4000 spectra from IR-microscopy
- Data reduction – principle component analysis
- Classification/clustering of data
- Translation one spectrum into one data point

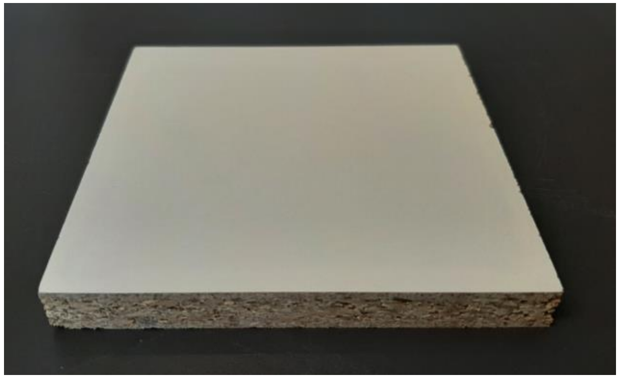




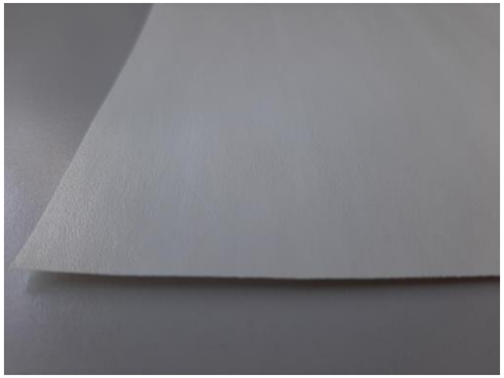
Samples- Macroscopic



Lacquer-coated wood-based panel



Melamine-coated wood-based panel

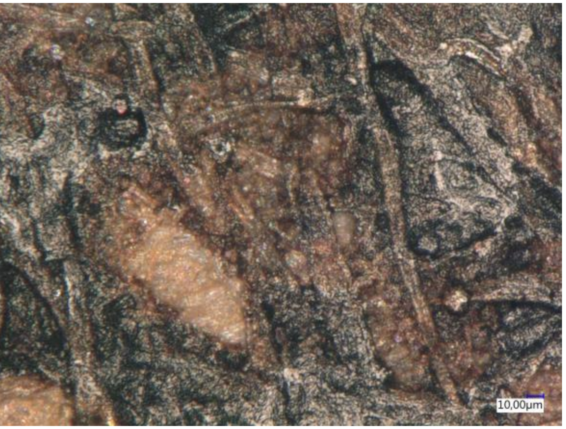


Impregnate

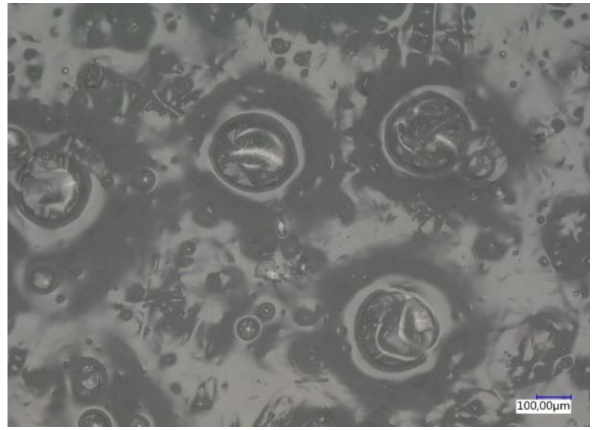
Printed raw paper

Coated paper

Samples- Microscopic



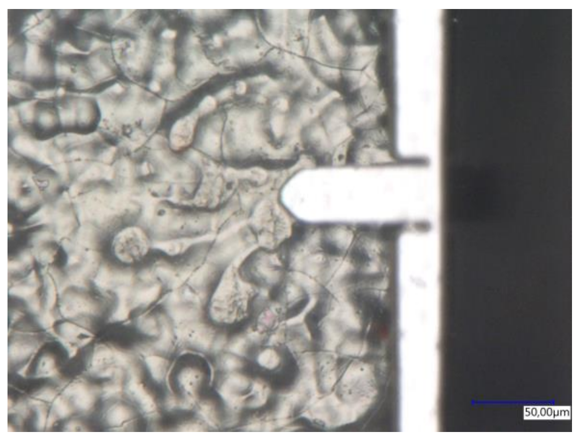
Printed raw paper



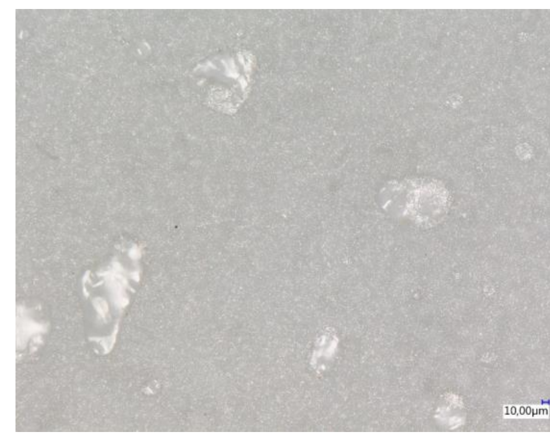
Impregnate

Melamine-coated surface

Lacquer-coated surface



Melamine-coated surface



Melamine-coated surface

Research Questions

Overall goal: correlation of macroscopic, microscopic and nanoscopic properties

1. Sample preparation: How are valid AFM- & IR-microscopy measurements gained?
2. What information can be obtained with AFM & IR-microscopy?
3. Are differences between systematically produced samples visible? How can they be correlated?

1) Sample Preparation of wood-based materials and papers

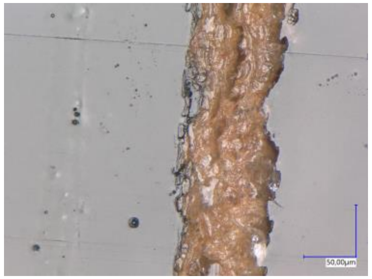
(Publication in progress)

Sample Preparation of wood-based materials and papers

- Traditional sample preparation
  - Cutting for surface analysis
  - Grinding and polishing
- Challenges for micro to nanoanalysis
  - Get small, but representative samples
  - Embedding → porous structures
  - Getting a flat surface → ultramicrotomy
  - Wet cutting → swelling

After cutting (size 10 x 10 cm)

After grinding and polishing (without and with embedding)

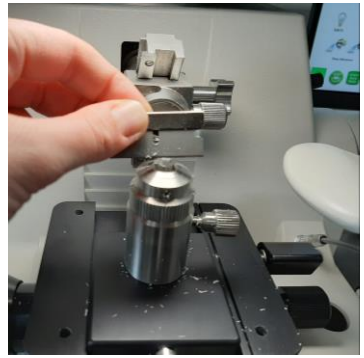


Swelling of raw paper after wet cutting

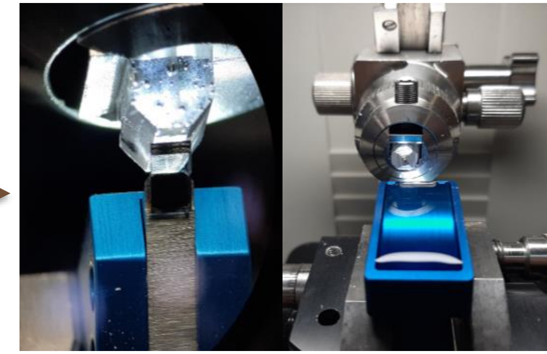
Sample preparation and analysis strategy

Silicon embedding molde, Epofix

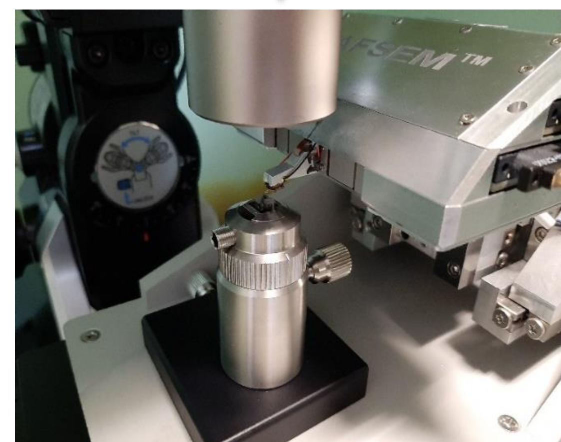
Samples embedded, in sample holder



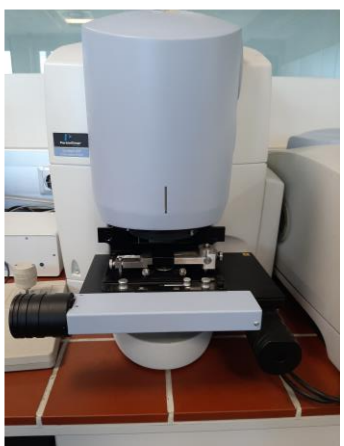
Trimming with razor blade



Ultramicrotome with trim knife and histo knife

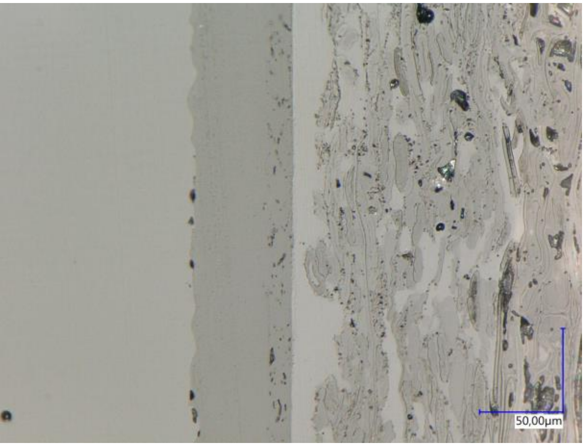


AFM on digital microscope

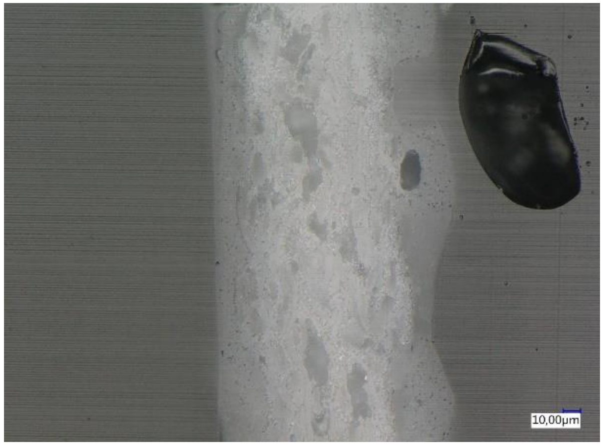


IR-microscope

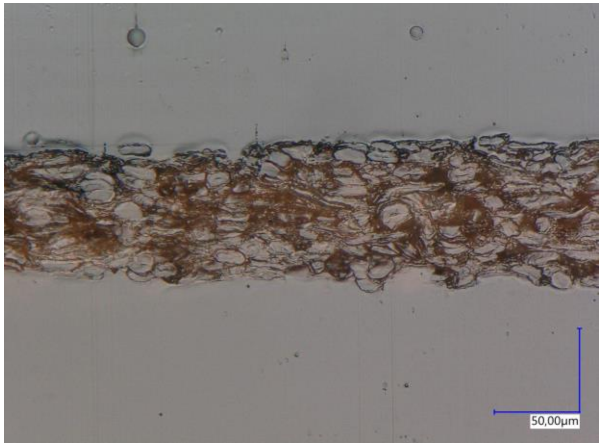
Sample cross-sections - Microscopic



Lacquer-coated wood-based material



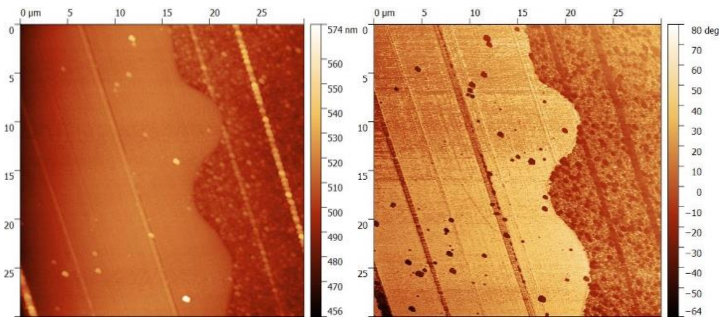
Pigmented impregnate



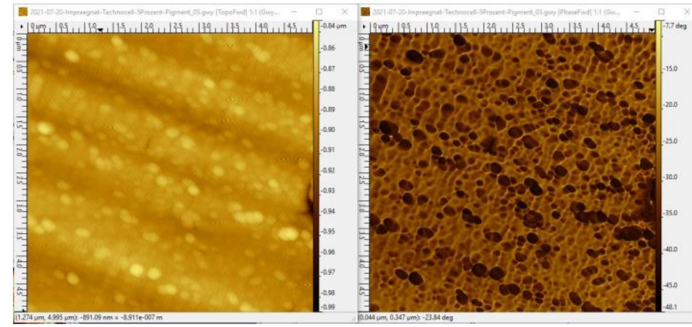
Printed raw paper

2) What characteristic chemical and physical information of wood-based materials, papers and composites can be obtained with AFM and IR-microscopy?

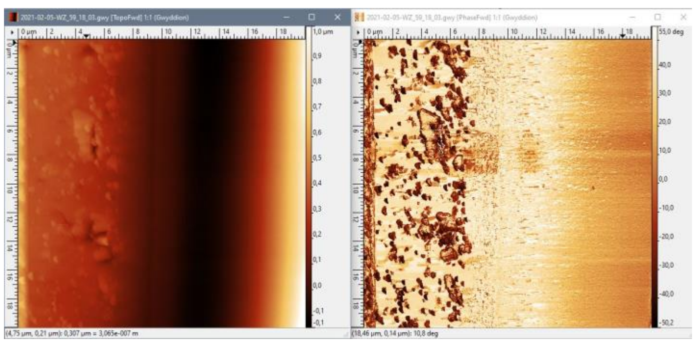
Sample cross-sections - AFM measurements



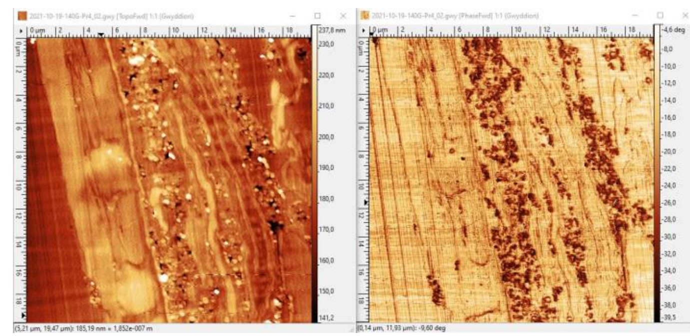
Lacquer-coated wood-based material



Pigmented impregnate

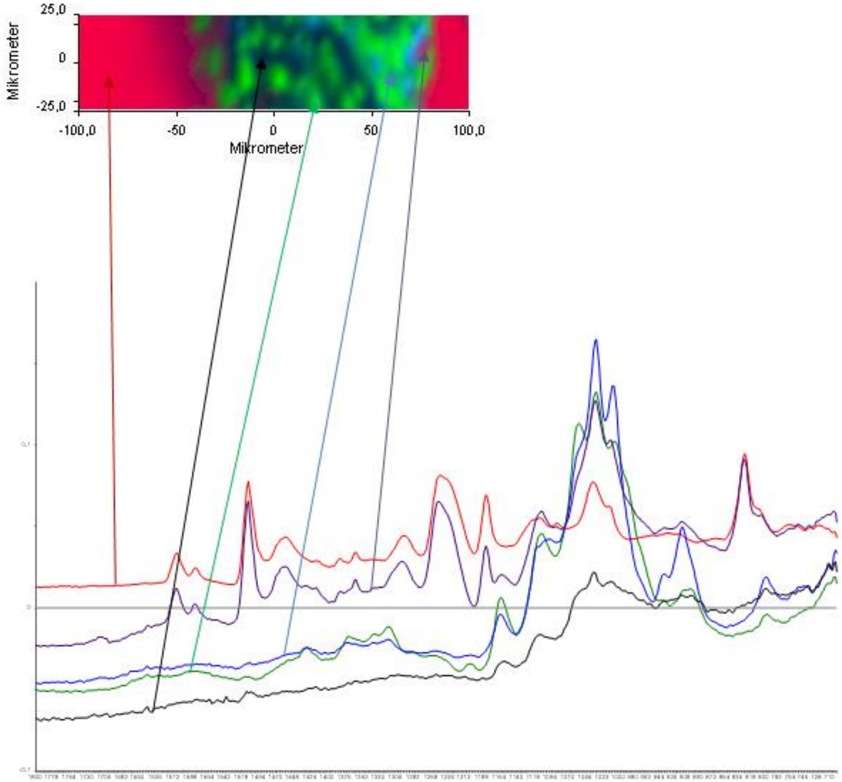


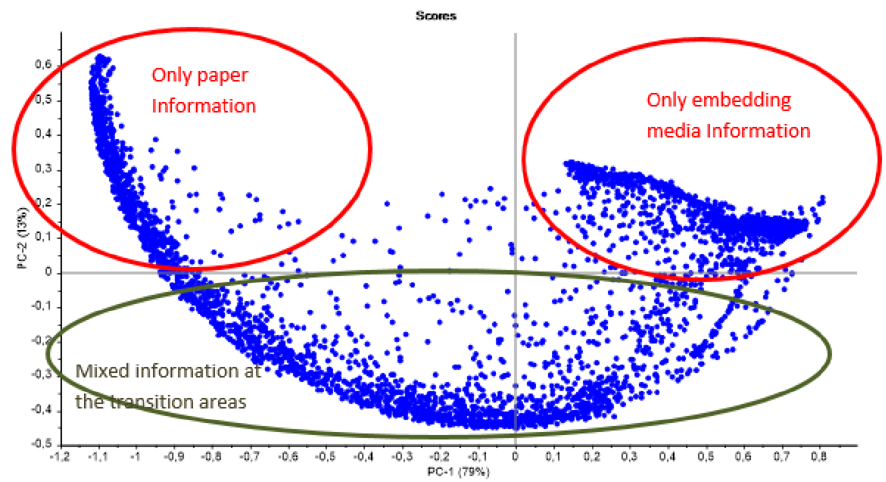
Lacquer-coated wood-based material

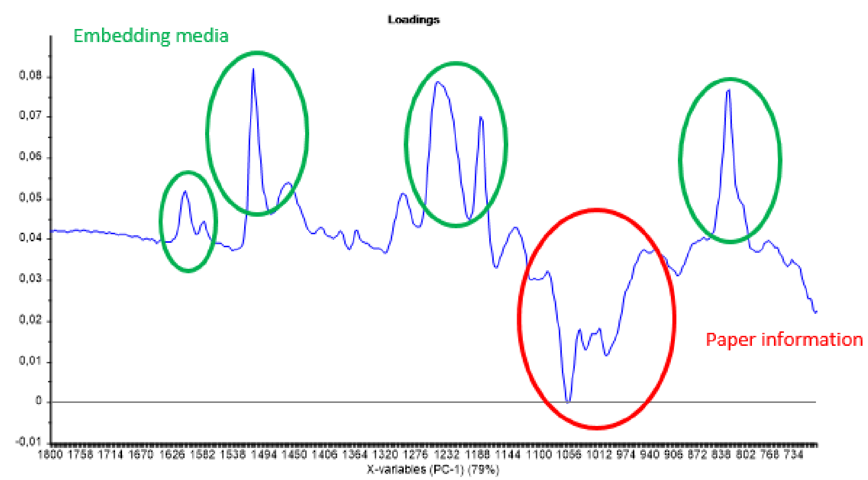


Melamine-coated wood-based material

Sample cross-sections – IR microscopy

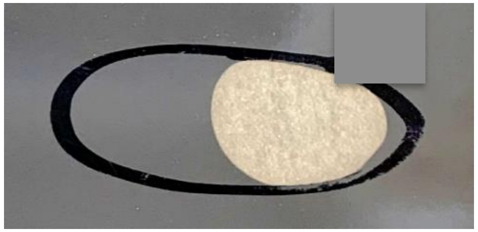






3) Are differences between systematically produced samples visible? How can they be correlated?

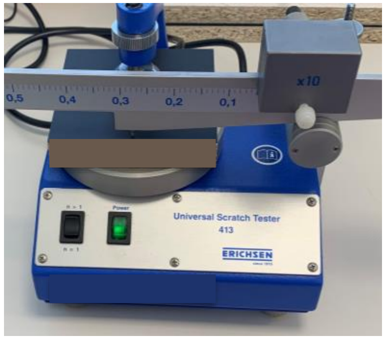
Standard surface testing methods – macroscopic values



Acid test

Porosity test

Water steam resistance



Scratch resistance

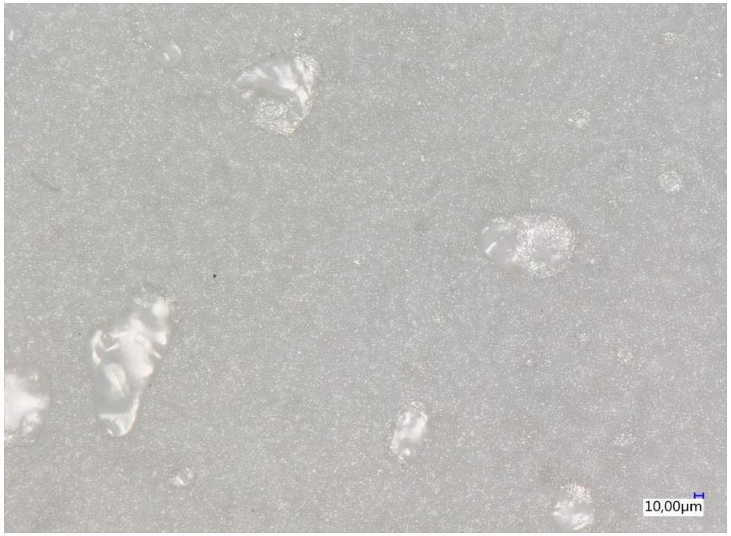
Standard surface testing methods – macroscopic values

3 different pressing temperatures for different macroscopic values

- 130°C, 140°C, 185°C

Test method	Sample 185°C	Sample 140°C	Sample 130°C
Acid test	Good (4,5)	Good (4,5)	Okay (3)
Porosity	Bad to ok (2)	Okay (2,5)	Bad (1,5)
Water steam test (Gloss)	Good (4,5)	Good (4,5)	Bad (2,5)
Scratch resistance [N]	Very good (>5 N)	Okay (3 N)	Bad (2 N)

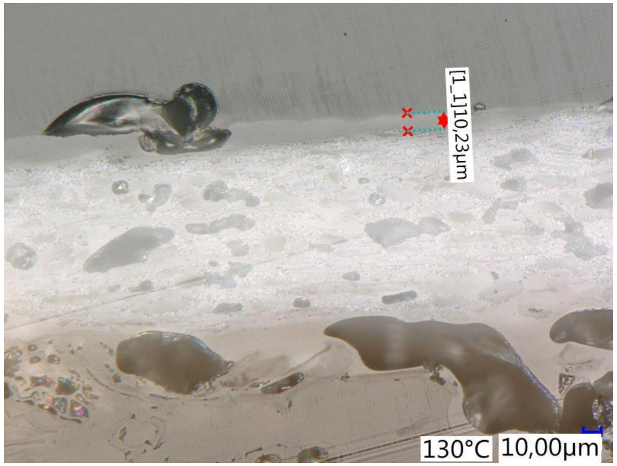
Macro to Nano – Microscopy Evaluation of the Surface and macroscopic tests

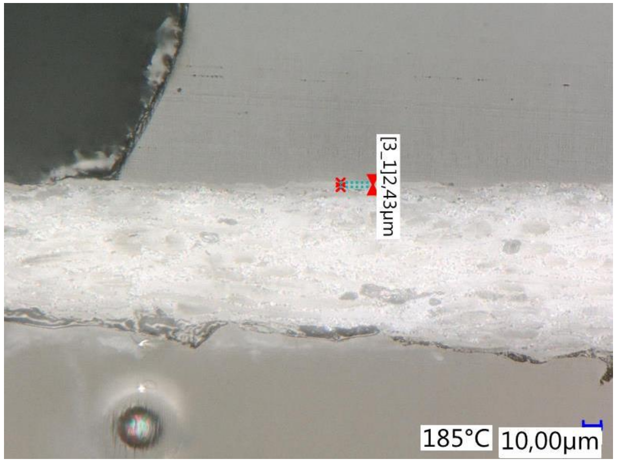


Sample 130°C, 400x

Sample 185°C, 400x

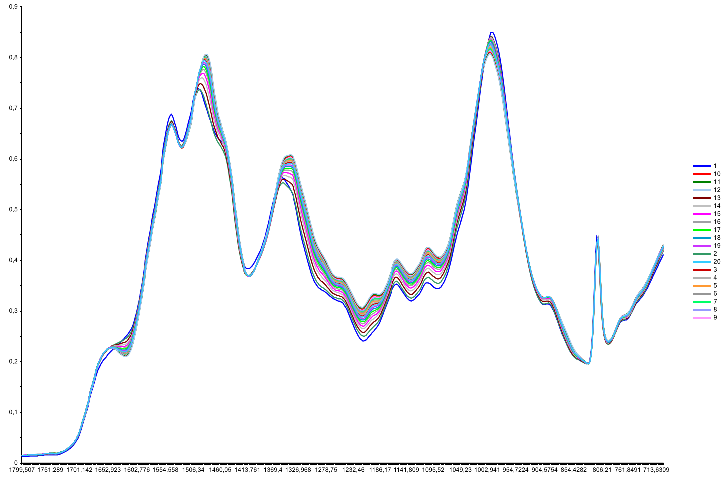
Macro to Nano – Microscopy Cross-sections



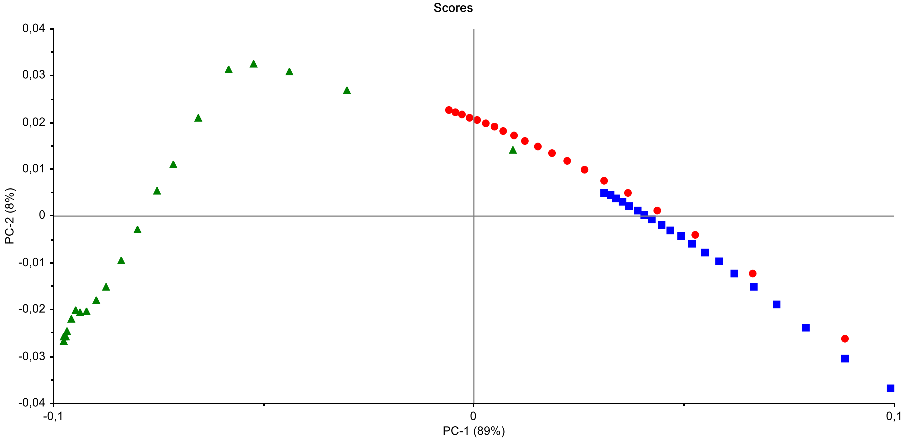


Comparison of the sample cross-sections including the measured resin layer thickness over the paper

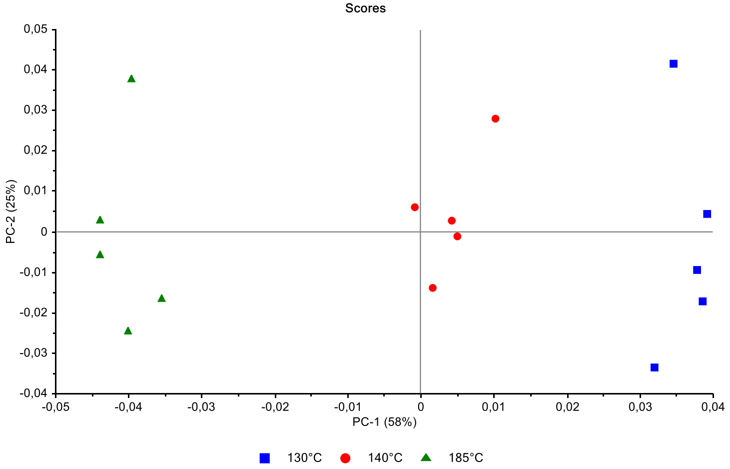
Macro to Nano – impregnates and pressed samples



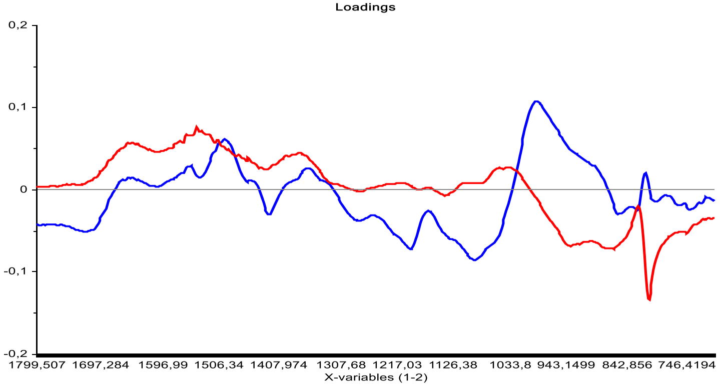
Isotherm spectra, 130°C



PCA Isotherm impregnate (3 temperatures), after UVN



PCA pressed samples (after UVN, groups observable)



Loadings plot pressed samples (blue: PC1, red: PC2)

Outlook

- Force Distance Curve Evaluation
- End-correlation macro to micro to nano
- Publication of obtained results

Thank you for your attention!

Questions?

Kontakt:
Kompetenzzentrum Holz GmbH
Altenberger Straße 69
A-4040 Linz

Tel.: +43 (0)732 2468 6750
Fax: +43 (0)732 2468 6755

E-Mail: zentrale@wood-kplus.at
Homepage: www.wood-kplus.at