NIR & WOOD – SOUNDS GOOD! #2

Monitoring thermally modified wood performance by NIR. Case of study: surface treatment

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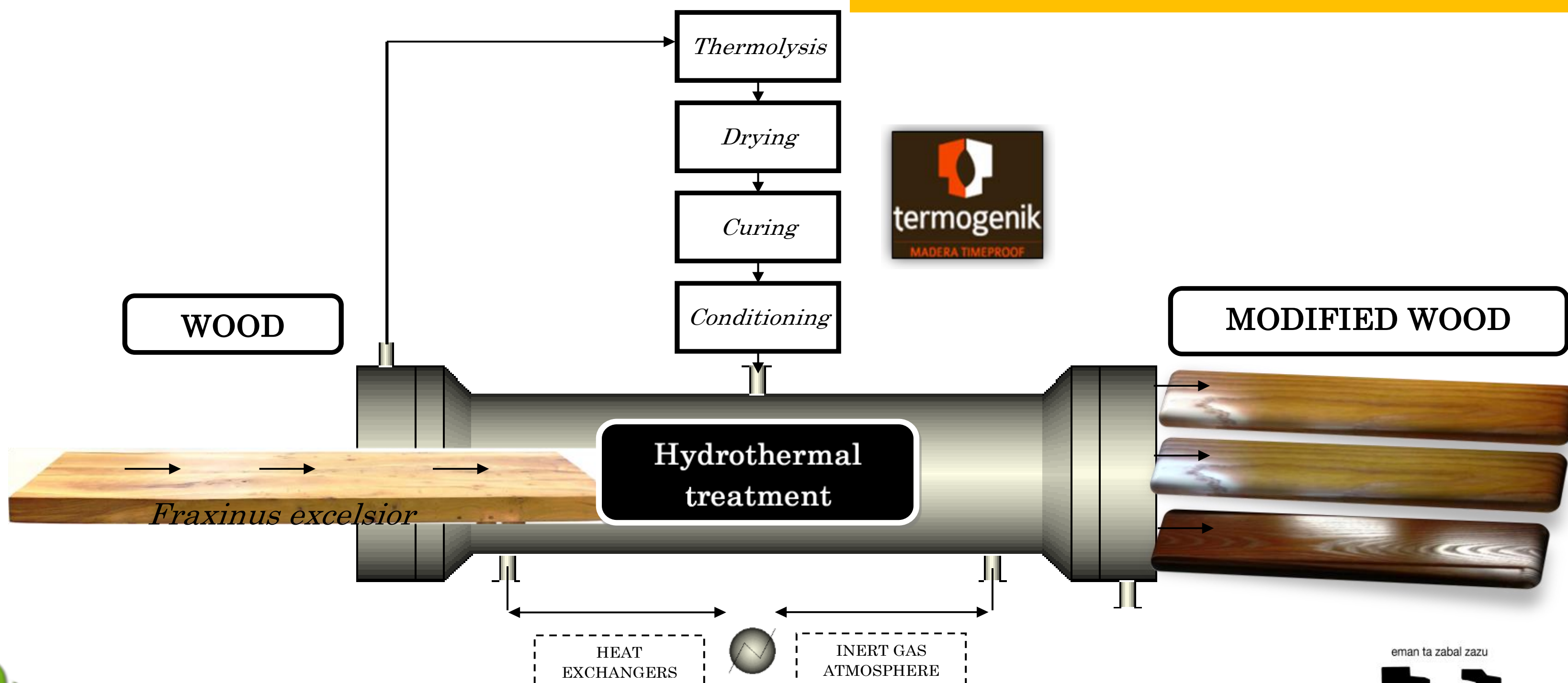
Monitoring thermally modified wood

Industrially modified species:
Fraxinus excelsior
192°C
202°C
212°C



termogenik
MADERA TIMEPROOF

BioRP
Bio Refinery Processes

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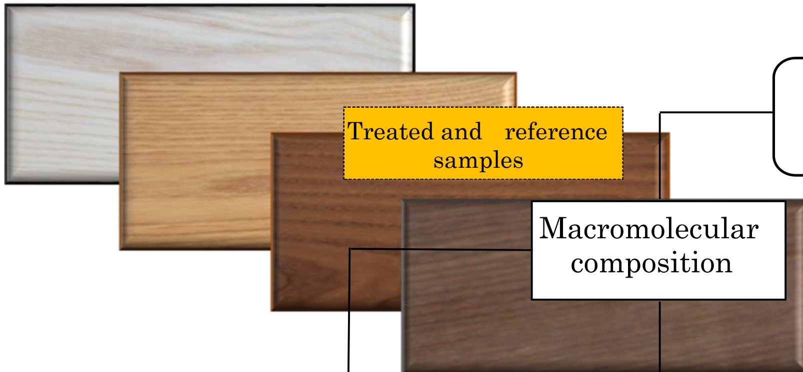
Monitoring thermally modified wood



Treated and reference samples

Thermally modified wood

- Macromolecular composition
  - Thermogravimetric Analysis
  - Wet chemical analysis
    - Ash TAPPI T211 om -93
    - Ethanol -Toluene extraction TAPPI T264 cm
      - Extractive free wood
      - Hollocelulose [a] (Wise method)
        - Hemicellulose (a – b)
        - α-cellulose [b] (Rowell method)
      - Lignin TAPPI T222 om - 98
- Chemical structure
  - FT-IR Spectroscopy (800 to 4000 cm^{-1})
  - NIR Spectroscopy (4000 to 8000 cm^{-1})
- Physicochemical characterization
  - Oven dry basis
    - Moisture content
    - Specific gravity
    - Water uptake
      - pH
      - Acidity
  - Surface evaluation
    - Contact angle (surface free energy)
      - Optical appearance (CIE-Lab)

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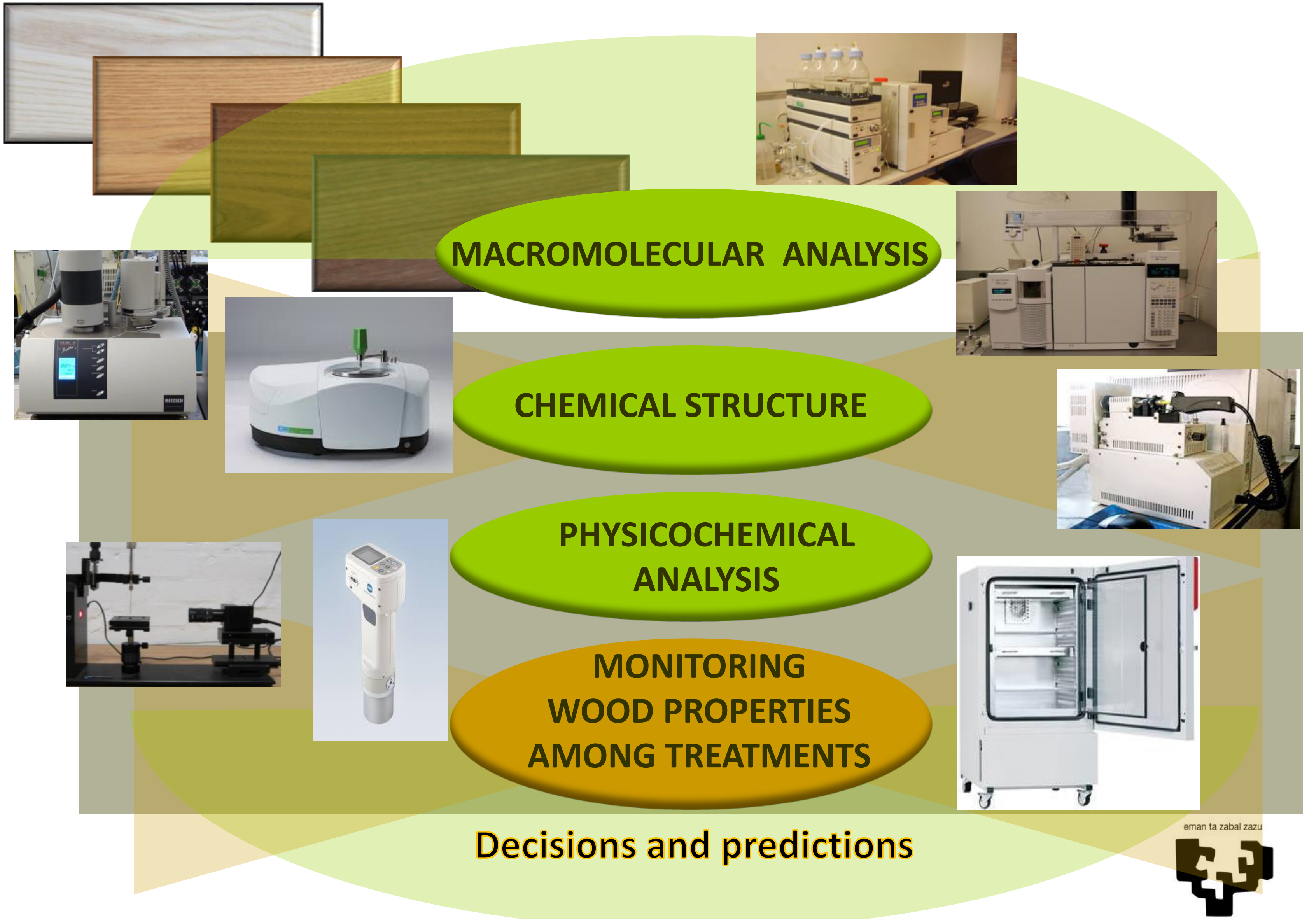

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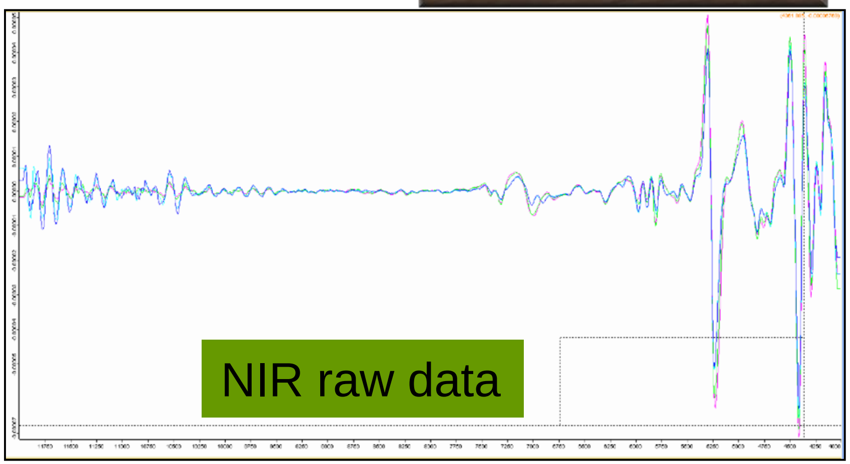
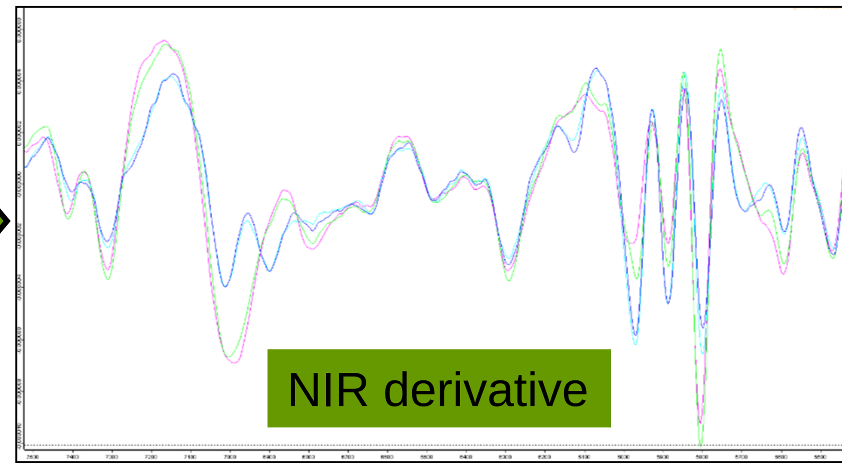
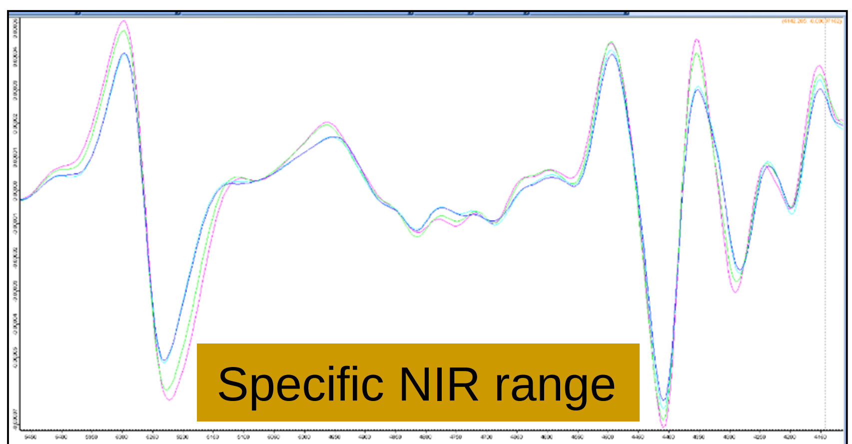
Multivariate analysis of multi-sensors data

Preprocessing data







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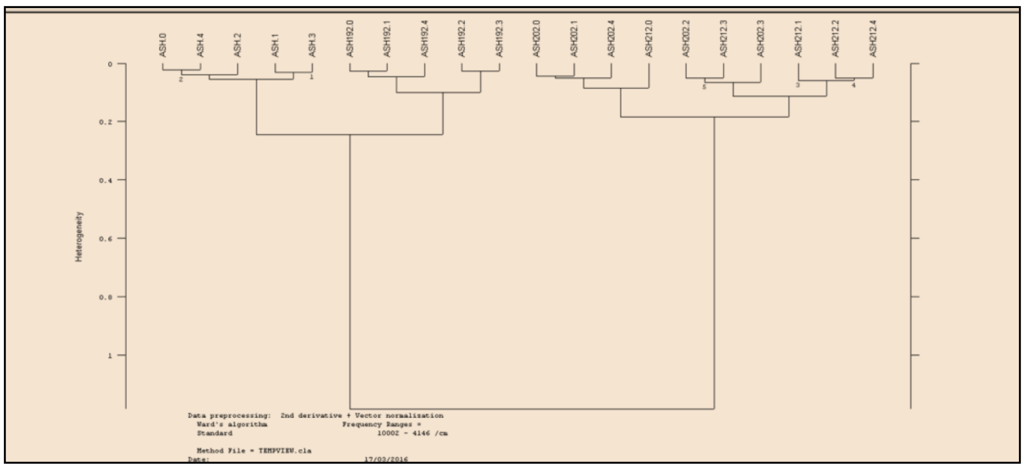
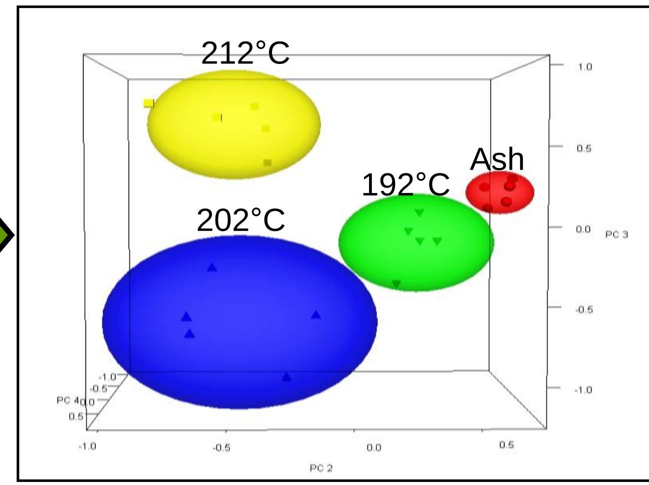
Multivariate analysis of multi-sensors data

Data fusion

Cluster analysis

PCA





Data was divided as a dendogram in four main classes (without supervised statistical algorithms). Heterogeneity should be determined by an expert.

Principal component analysis decomposed highly correlated data in 4 plotted PC

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Multivariate analysis of multi-sensors data

Identity test

Cluster analysis

PCA

	ID	Group1	Group2	IP-Level	S	Threshold1
1	1	ash	ash192	IP1: New	0.951993	0.141386
2		"	ash202	IP1: New	1.906599	"
3		"	ash212	IP1: New	2.327233	"
4	2	ash192	ash	IP1: New	0.952004	0.375173
5		"	ash202	IP1: New	0.977602	"
6		"	ash212	IP1: New	1.648072	"
7	3	ash202	ash192	IP1: New	0.977605	0.586747
8		"	ash212	IP1: New	1.383708	"
9		"	ash	IP1: New	1.906604	"
10	4	ash212	ash202	IP1: New	1.383700	0.390028
11		"	ash192	IP1: New	1.648064	"
12		"	ash	IP1: New	2.327225	"

PCA and CA can be useful for differentiation of samples and classified unmeasured samples into defined groups

Result of IDENT evaluation:

Sample name: wood
Sample: D:\NIR Rene\Ash.0
Date and time (measurement): 2016/03/15 11:00:15 (GMT+1)
Method file: D:\NIR Rene\only ash 5 spectra.faa

Hit no.	Sample name	Hit qual.	Threshold	Group
1	wood	0.07973	0.14139	ash
2	wood	0.42286	0.37517	ash192
3	wood	1.19121	0.39003	ash212
4	wood	1.30935	0.58675	ash202

IDENTIFIED AS ash

✓ OK

It is possible to discover trends when playing with sensor data and validating clusters within threshold (S>1)

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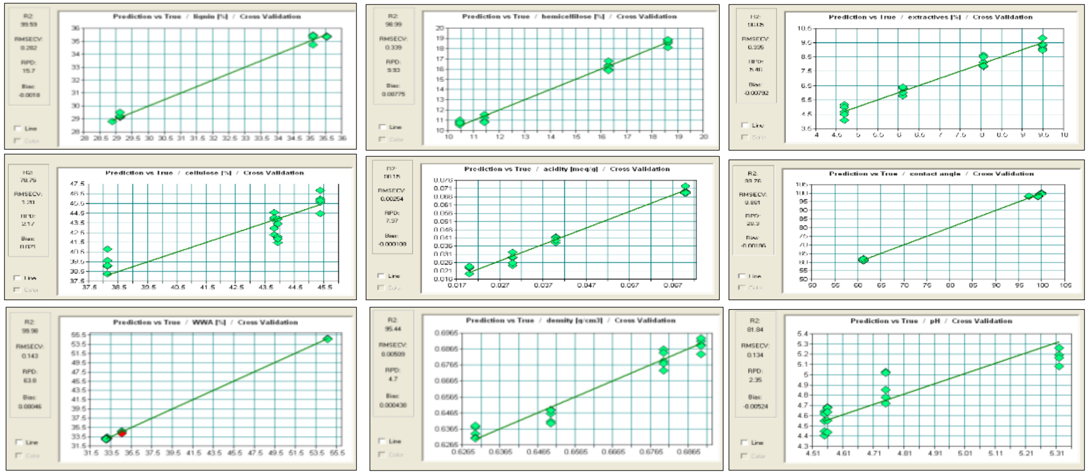
Multivariate analysis of multi-sensors data

Partial Least Squares

Calibration data set

PLS



Analysis [%]	Lignin	α-Cellulose	Hemicellulose	Extracts
Ash	28.88	45.38	28.88	4.69
Ash192	29.13	43.81	29.13	6.11
Ash202	35.12	43.93	35.12	8.06
Ash212	35.54	38.14	35.54	9.49

Analysis	Density	Acidity	pH	WWA	Contact angle
Ash	0.69	$6.74\ 10^{-2}$	4.55	54.65	61.31
Ash192	0.68	$4.35\ 10^{-2}$	4.55	34.59	99.96
Ash202	0.65	$2.79\ 10^{-2}$	4.75	33.13	99.19
Ash212	0.63	$1.59\ 10^{-2}$	5.32	33.03	97.22

Models PLS

The development of Partial Least Squares (PLS) model starts with computation of PCs on the base of calibration dataset.
The PLS model has to be validated after calibration.

Depending on the number of available samples two validation strategies are possible: cross-validation or test-set validation.
The high value of R2 and small value of RMSEP indicates excellent PLS models.

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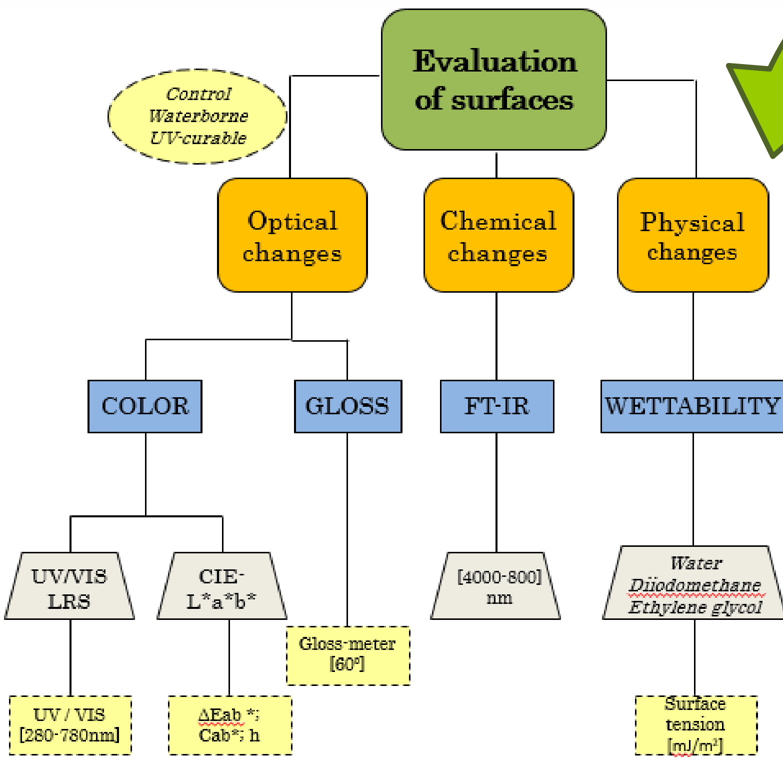
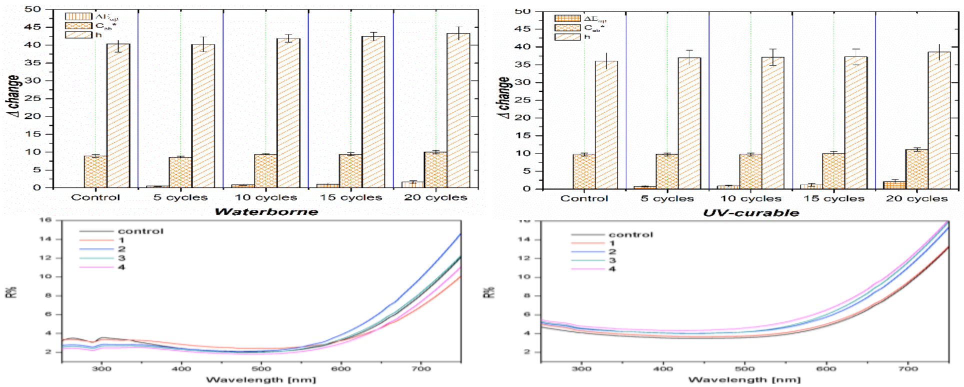
Monitoring surface treatment



Monitoring surface treatment

Preprocessing data



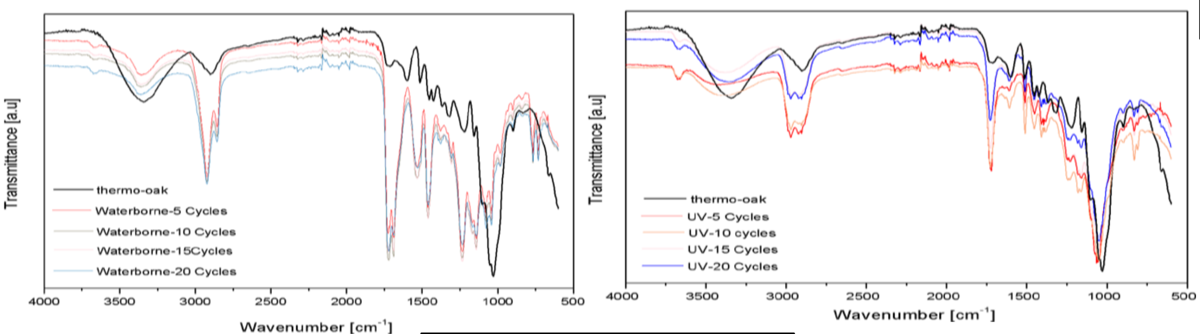
COLOR RESPONSE

Table 1. Values of contact angle and surface tension after weathering test

Sample	Coating	Aging cycles	Contact angle[1] [deg] water	Contact angle[1] [deg] Diiodo-methane	Contact angle[1] [deg] Ethylene glycol	Surface tension [mJ/m²]
Thermo -Oak 170 ºC	Waterborne	C	72.3±2.1	60.8±0.9	71.2±1.1	31.7
		5	69.2±1.2	57.4±0.7	69.9±1.0	33.7
		10	69.0±1.2	60.5±0.6	71.3±0.9	32.9
		15	70.7±1.5	56.7±0.8	70.4±1.4	33.2
		20	69.6±1.1	57.9±0.8	71.0±0.8	33.2
	UV-curable	C	63.1±3.1	40.9±0.7	50.8±0.9	41.4
		5	61.9±2.1	48.9±0.4	48.8±0.7	42.7
		10	60.4±1.4	46.7±0.7	44.6±0.9	44.5
		15	57.7±2.1	49.7±1.3	51.4±1.0	43.4
		20	53.9±1.5	59.1±0.6	53.2±1.6	43.3

[1] Average value of five measurements; C= Control samples

WETTABILLITY



CHEMICAL CHANGES

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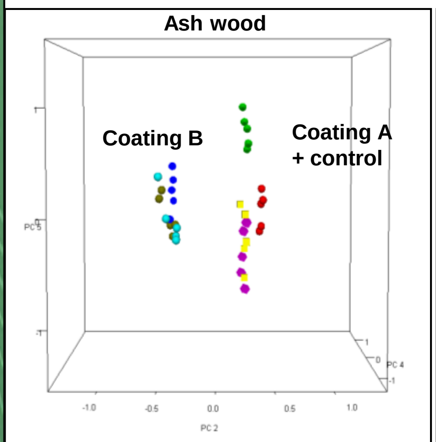
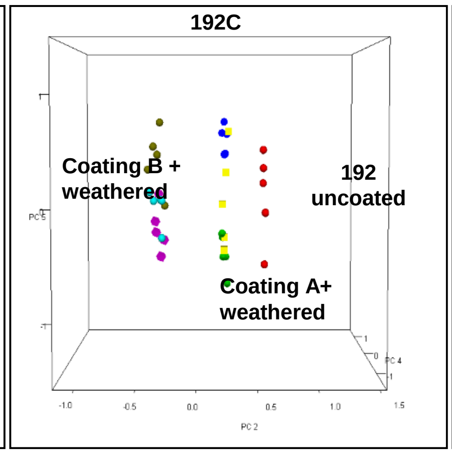
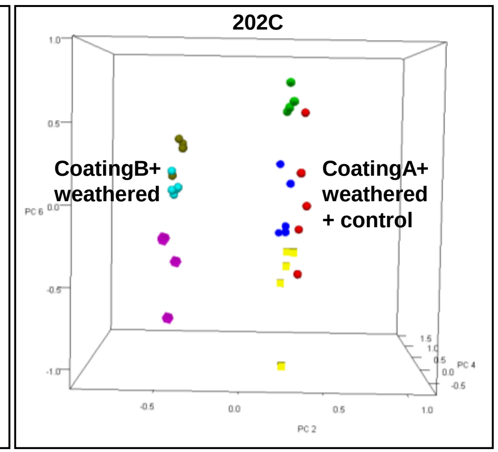
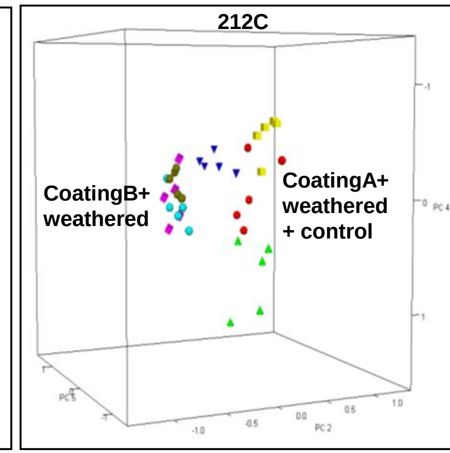
Monitoring surface treatment

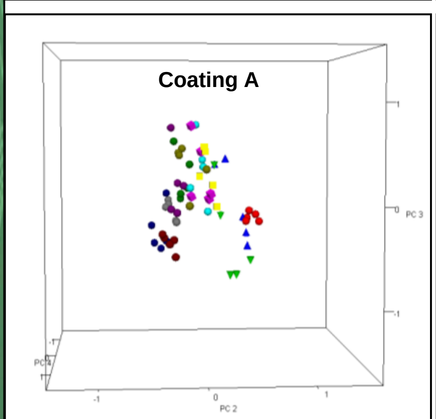
Data fusion

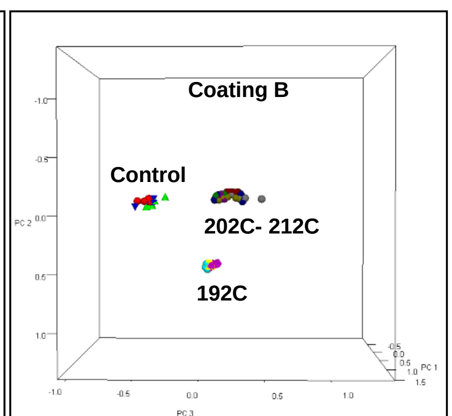












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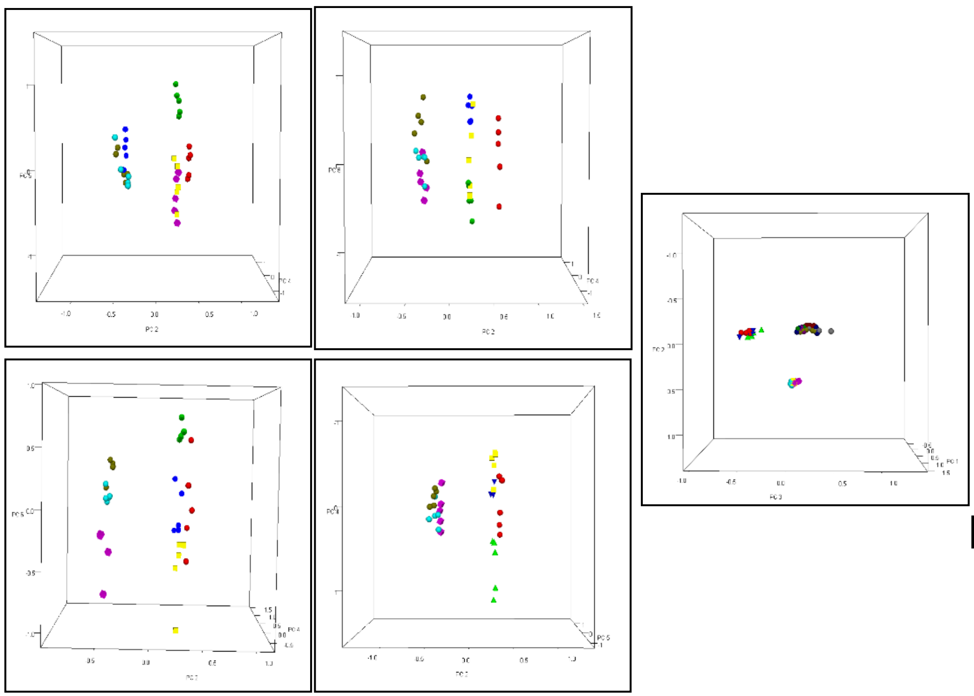
Data fusion



Using all samples or divided by groups?

Using FT-IR, UV-VIS or TGA along with NIR?

How many repetitions per sensor and what amount of data is enough?

TO BE CONTINUED…

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Performance of bio-based building materials

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