Workshop on application of NIR spectroscopy for wood science and technology research

NIR & WOOD – SOUNDS GOOD! #2

Assessment of poplar veneers for playwood manufacturing after up-grading phase by vacuum thermal treatment

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crea
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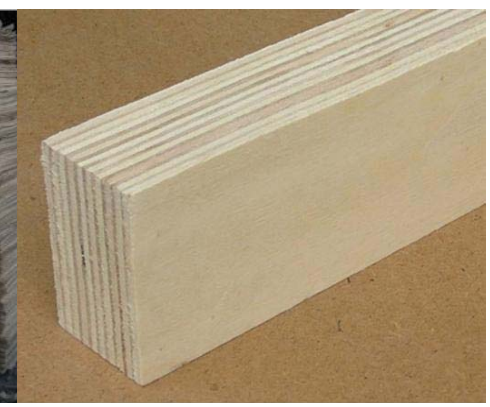
Poplar resources

- fast growing species, widespread in plantations with rotation period of 5 to 20 years
- in Europe poplar plantations cover about 940.000 hectares
- 90% of their production is used for manufacturing of plywood, sawn timber, pulpwood, fuelwood and biomass for energy
- high hygroscopicity of the wood material



Goals

The research presented here is an attempt for detailed **characterization and evaluation** of poplar veneers modified in vacuum conditions.

The goal of this work was to evaluate the **influence of temperatures, time and pressure** on principal indicators of thermal modification process such as **ML** and **EMC** of investigated material.

The other scope was to develop **PLS models** that might be used **for quality control** of treated veneers in industrial scale production

Experimantal samples

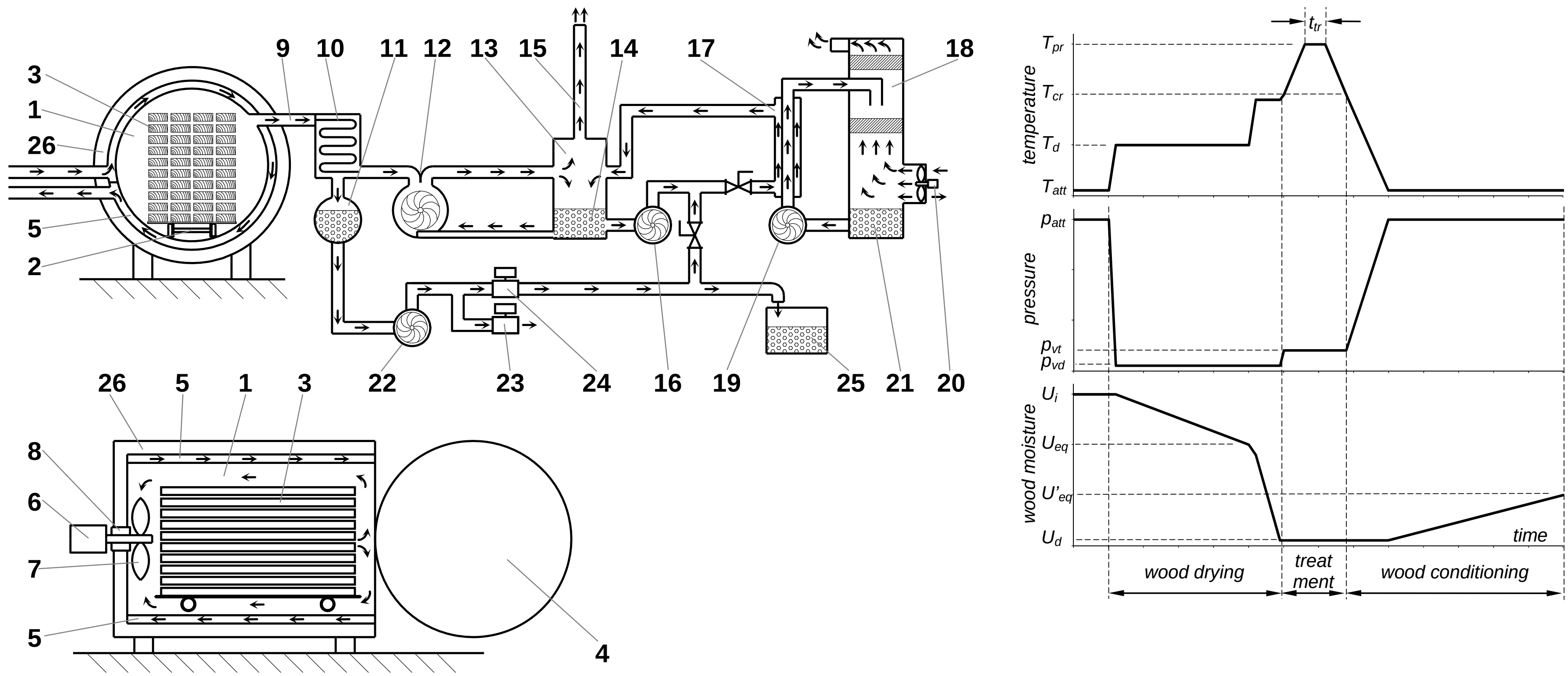
Rotary-cut veneer sheets of Poplar clone ‘I-214’ (*Populus × canadensis* Moench)

One sheet was prepared for each treatment

Veneers were cut to dimensions 360 mm x 150 mm x 2.5 mm

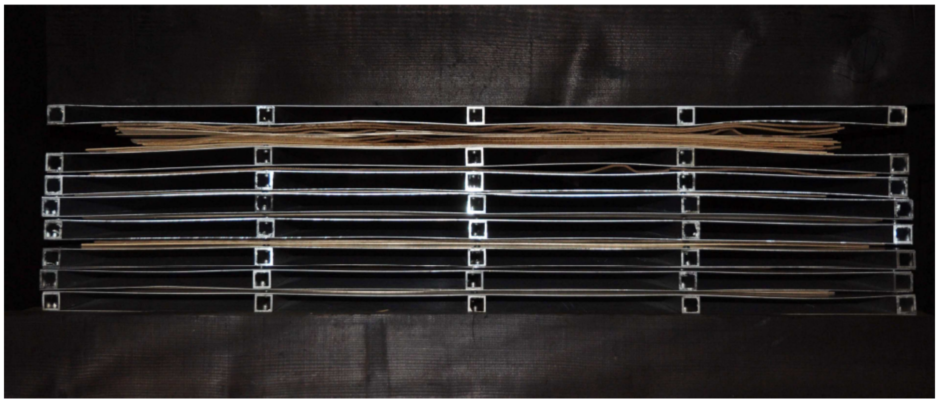
Characterized both before and after vacuum thermal treatment

Thermovacuum treatment



schema of TERMOVUOTO® system and process conditions (Sandak et al. 2015)

Treatment set-up



Aluminium plates heated electrically which produce a heating of the conducting type were used.

Heating and cooling ramp were kept constant at 60°C/h.

The ventilation was disabled so that the test chamber only acts as a sealing system of the vacuum.

38 batch processes with various treatment conditions (temp. from 150°C to 240°C, pres. 100, 250 or 1000 mbar and time from 0.5 to 22.5 h.)

batch number	**#1**	**#2**	**#3**	**#4**	**#5**	**#6**	**#7**	**#8**	**#9**	**#10**	**#11**	**#12**	**#13**
T max [ºC]	240	238	238	238	195	150	174	192	174	213	240	253	239
time [hours]	1.5	1.5	1.5	1.5	6.1	12.4	6.4	22.2	22.5	22.2	22.3	1.1	1
pressure [mbar]	1000	250	100	1000	1000	250	1000	250	250	250	250	250	1000
batch number	**#14**	**#15**	**#16**	**#17**	**#18**	**#19**	**#20**	**#21**	**#22**	**#23**	**#24**	**#25**	**#26**
T max [ºC]	239	239	238	241	239	239	212	203	194	182	174	165	155
time [hours]	0.5	2.12	0.24	2.22	1.07	0.5	1.08	1.1	1.07	1.07	1.07	1.07	1.07
pressure [mbar]	1000	1000	1000	250	250	250	250	250	250	250	250	250	250
batch number	**#27**	**#28**	**#29**	**#30**	**#31**	**#32**	**#33**	**#34**	**#35**	**#36**	**#37**	**#38**	
T max [ºC]	149	223	213	213	211	213	194	173	214	175	240	217	
time [hours]	1.07	1.07	2.24	4.3	0	6.42	6.1	6.4	4.7	12.9	6.6	4.8	
pressure [mbar]	250	250	250	250	250	250	250	250	100	250	250	1000	

Samples characterization

Physical properties

Mass loss (ML) was determined by weighting each sample before the treatment and immediately after it, assuring the wood was absolutely dry (0% moisture content).

The Equilibrium Moisture Content (EMC) was calculated according to the ISO 3130 standard for treated and untreated samples

Color measurement

MicroFlash 200D spectrophotometer (DataColor Int), suitable for direct determination of the CIE L*a*b* colour coordinates was used for the measurement over an 18 mm diameter spot with a standard light source D65 and an observation angle of 10°. Each sample has been measured in 10 zones.

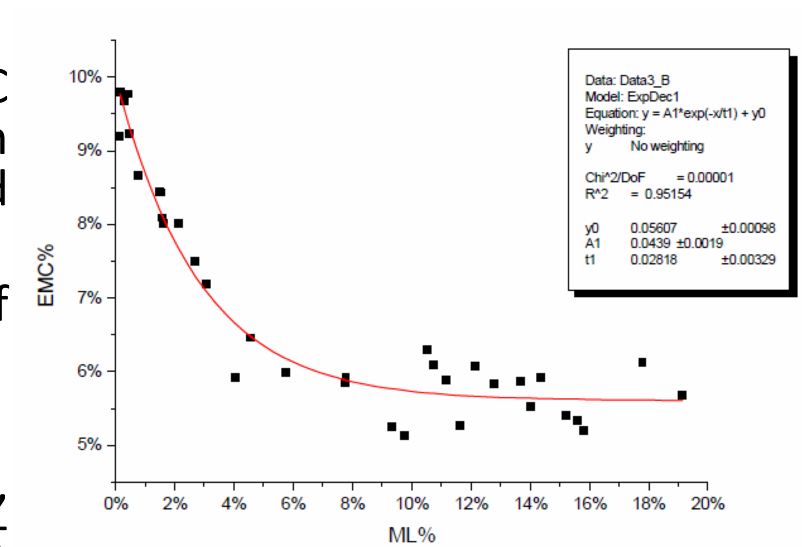
Chemical composition

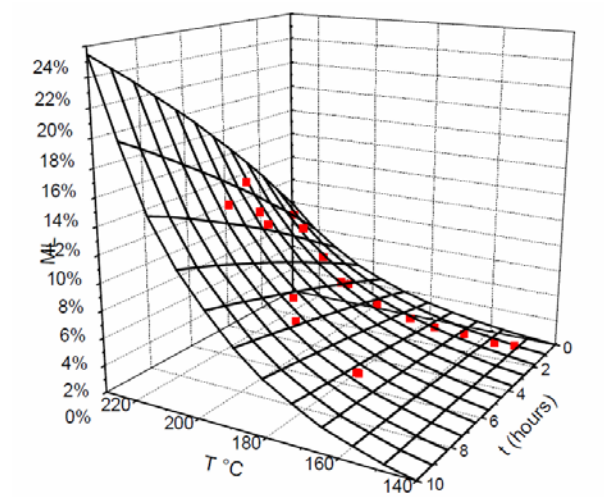
VECTOR 22-N (Bruker Optics GmbH, Ettlingen, Germany) equipped with the fibre-optic probe was used for spectra collection. The spectral range measured was between $4000cm^{-1}$ and $12000cm^{-1}$ and the spectral resolution of $8cm^{-1}$. Each sample has been measured in 5 zones.

Physical properties

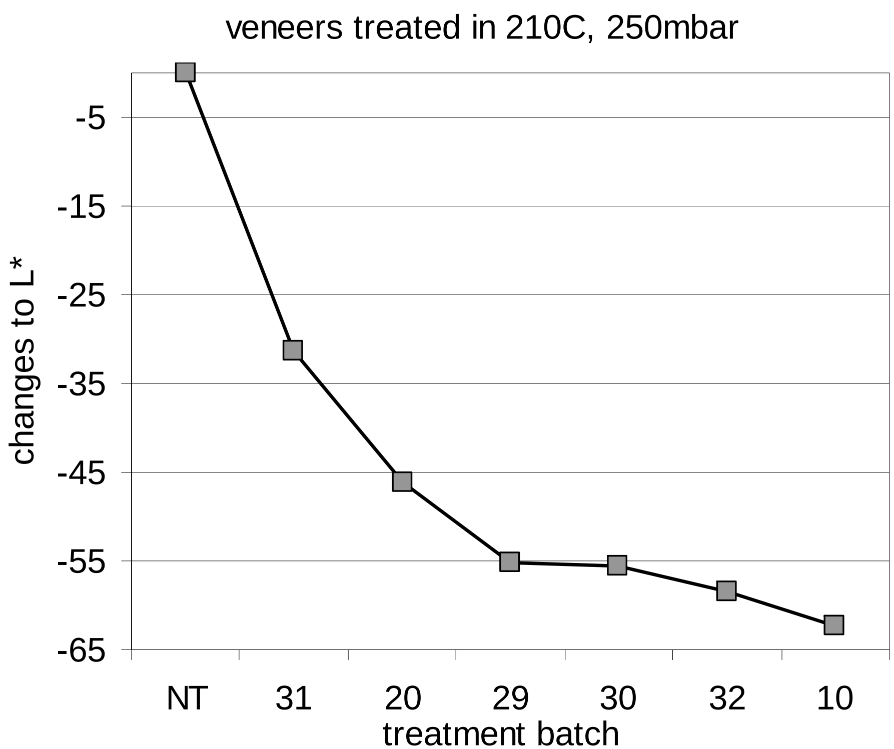
- Treated wood has significantly lower EMC compare to untreated wood and depends on the treatment temperature time and pressure.
- ML is closely correlated with decrease of EMC and leads to reduction of wood density.

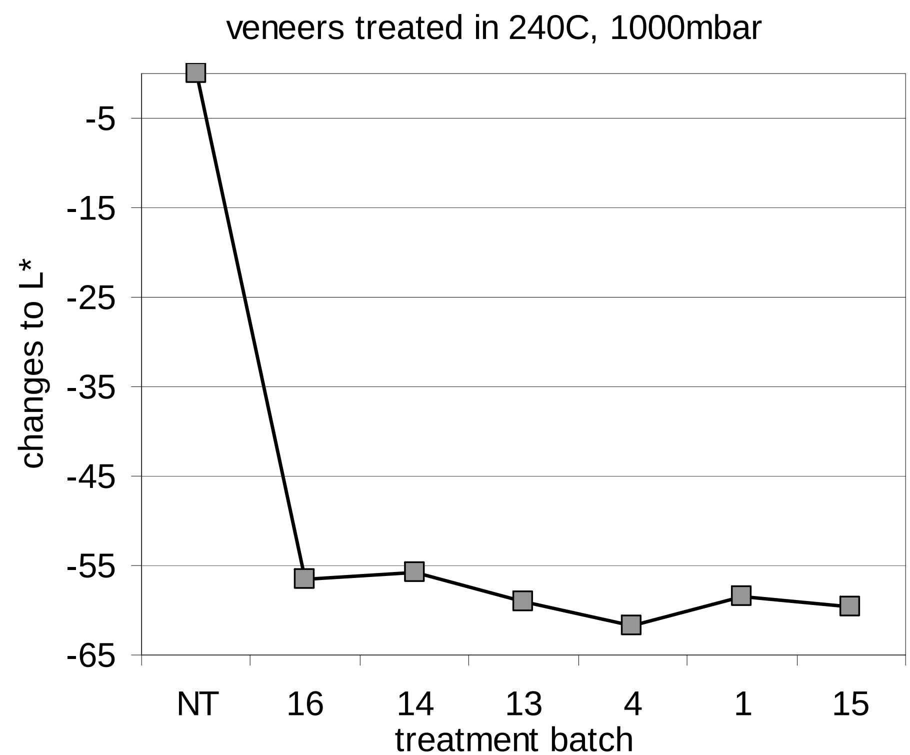
- The mass losses vary from 0% (150°C, 1 h, 250 mbar) to 48% in case of long treatment in high temperature (22h, 240°C, 250 mbar).
- Same treatment parameters (240°C, 250 mbar) but shorter time (6,6h) resulted 24% of mass loss.
- Similar effect (22% of ML) was obtained in slightly lower temperature (213°C), however with longer treatment (22h).





Colour

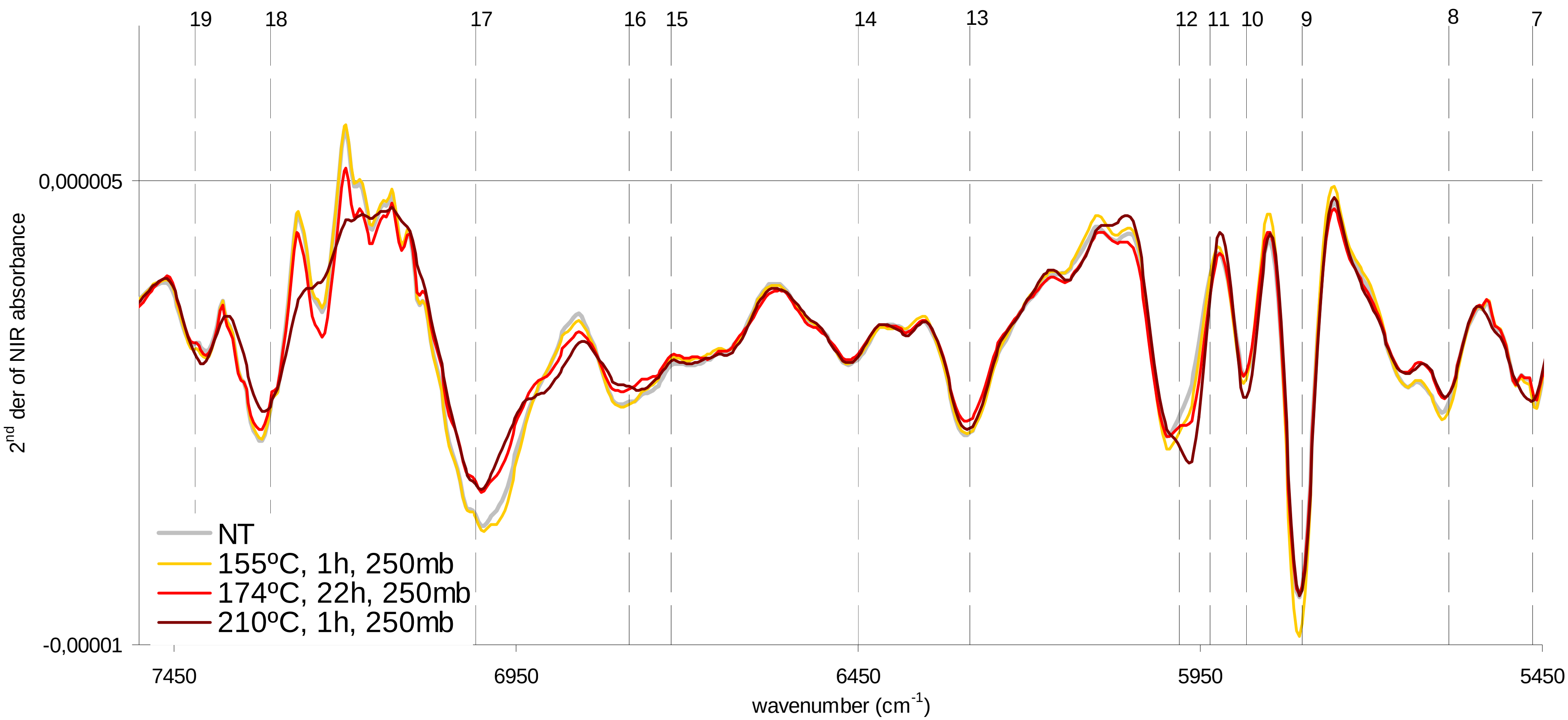




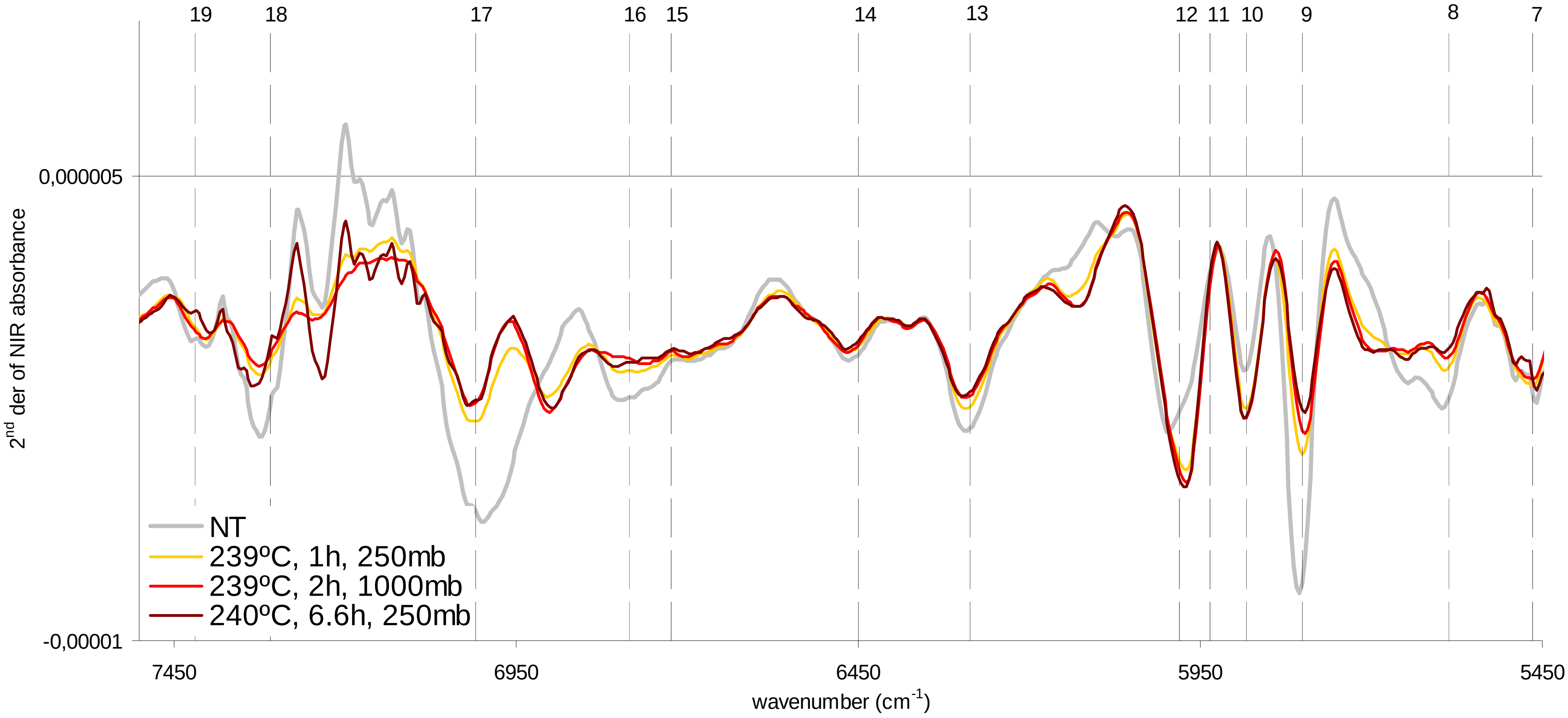
Time 0h 0h 1h 2.2h 4.3h 6.4h 22h

0h 0.2h 0.5h 1h 1.5h 1.5h 2.1h

Veneers in lower temperature



Veneers in higher temperature



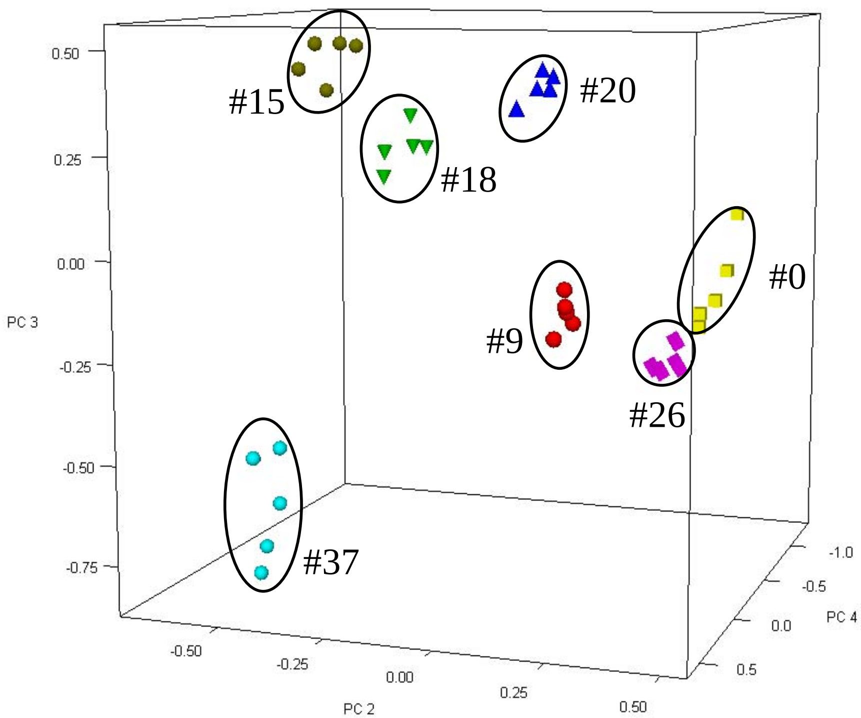
Spectra interpretation

band no.	low temperatute	high temperature	wavenumber (cm-1)	wood comonent	functional group
3	✓	✓	4204	holocellulose	OH
6	✓	✓	5219	water	OH
9	✓	✓	5800	hemicellulose (furanose/pyranose)	CH
12	✓	✓	5980	lignin	CH
13		✓	6286	cellulose crystalline	OH
16		✓	6787	cellulose semicrystalline	OH
17	✓	✓	7003	amorphus cellulose	OH
18		✓	7309	hemicellulose/cellulose	CH

Changes in EMC, ML and colour are effects of:

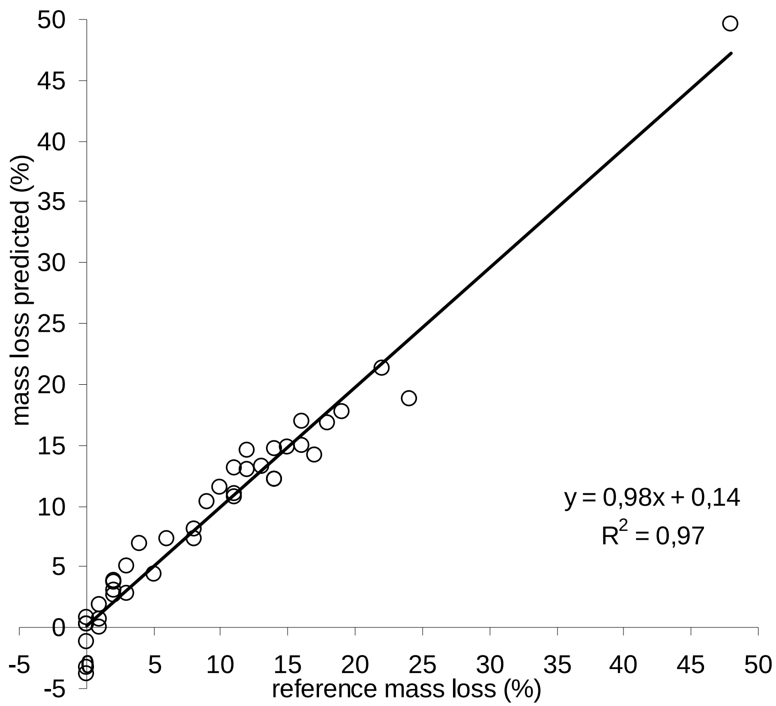
- migration or removal of extractives, low molecular weight sugars and aminoacids
- polymers degradation and its evaporation during the heat treatment process
- reduction of accessibility of hydroxyl groups of wood carbohydrates
- degradation of hemicelluloses and their conversion to less hygroscopic furan-based polymers
- polycondensation and crosslinking of lignin

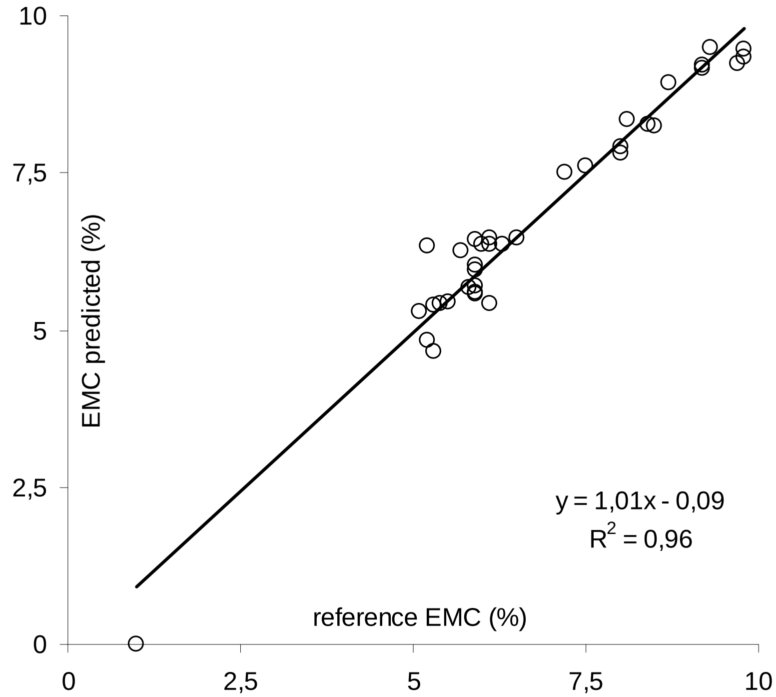
Principal Component Analysis



#0	NT
#26	155ºC, 1h, 250mb
#9	174ºC, 22h, 250mb
#20	210ºC, 1h, 250mb
#18	239ºC, 1h, 250mb
#15	239ºC, 2h, 1000mb
#37	240ºC, 6.6h, 250mb

Partial Least Squares





PLS predicted versus measured values of ML and EMC of VTM veneers

Conclusions

- MVA and chemometric modeling allowed understanding of the process mechanism and its kinetics and might be used for **selection of optimal process parameters**.
- **NIR was effectively used to predict wood physical properties**, considered as reliable indicators of the wood modification advancement.
- **Prediction errors** of validation models based on NIR spectra **were** relatively **small** (1.75% and 0.36% in case of ML and EMC respectively).
- The corresponding coefficients of determination were $R^2 > 0.97$ (ML) and $R^2 > 0.96$ (EMC).
- The **RPD** values 5.3 and 4.3 (for ML and EMC respectively) confirm **superior performance of the PLS**.

Acknowledgment

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