

Primary analysis methods used to control thermal treatments of wood and its effect on decay resistance

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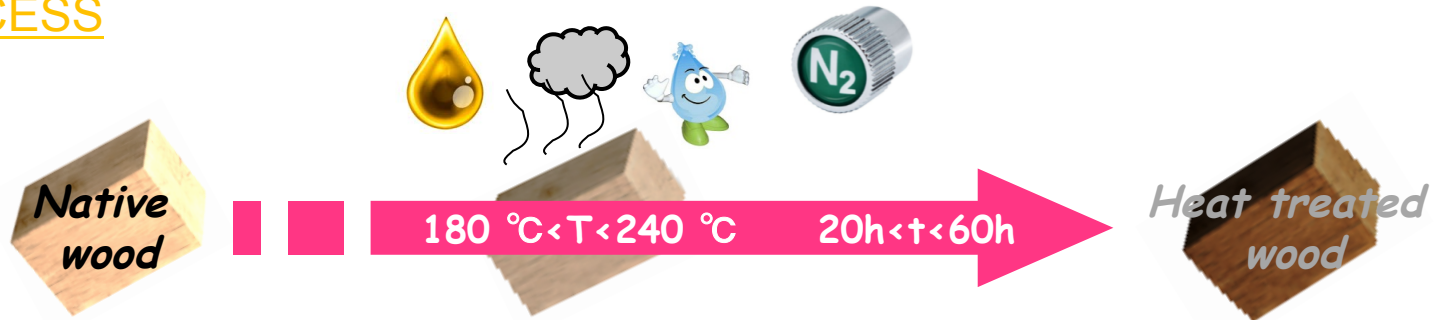
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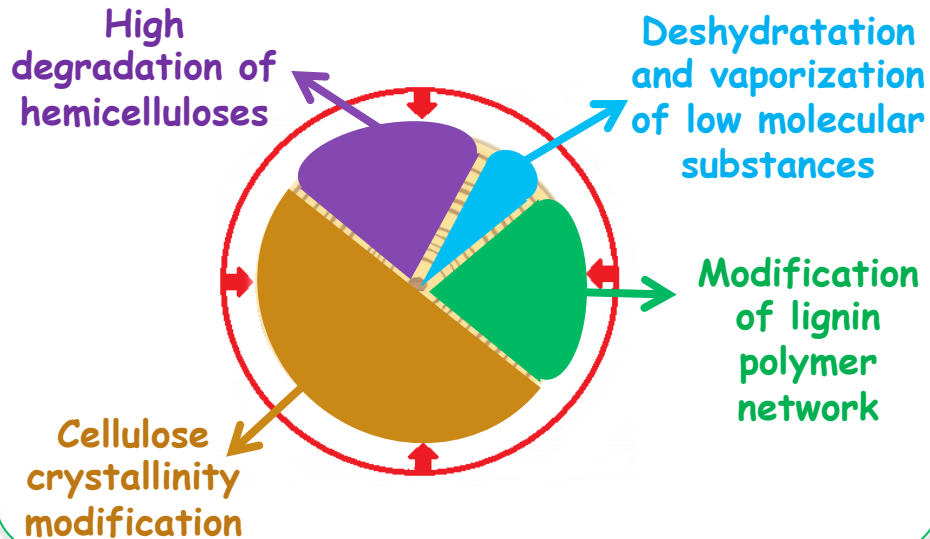
Context

Heat treatment of wood [Generalities]

PROCESS



CHEMICAL MODIFICATIONS



NEW MATERIAL PROPERTIES

Benefits

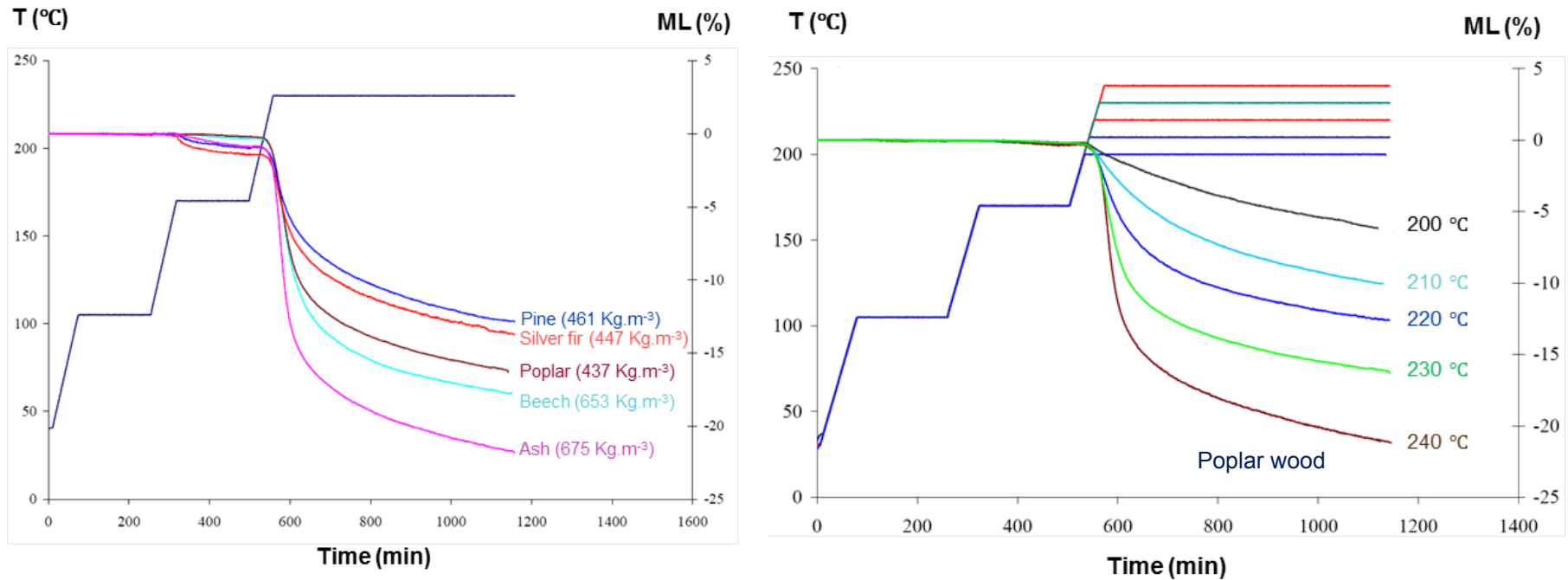
- Durability
- Dimensional stability
- Hydrophobicity
- Color

Disadvantages

- Mechanical Properties

Context

Heat treatment of wood [Wood thermal degradation kinetics]

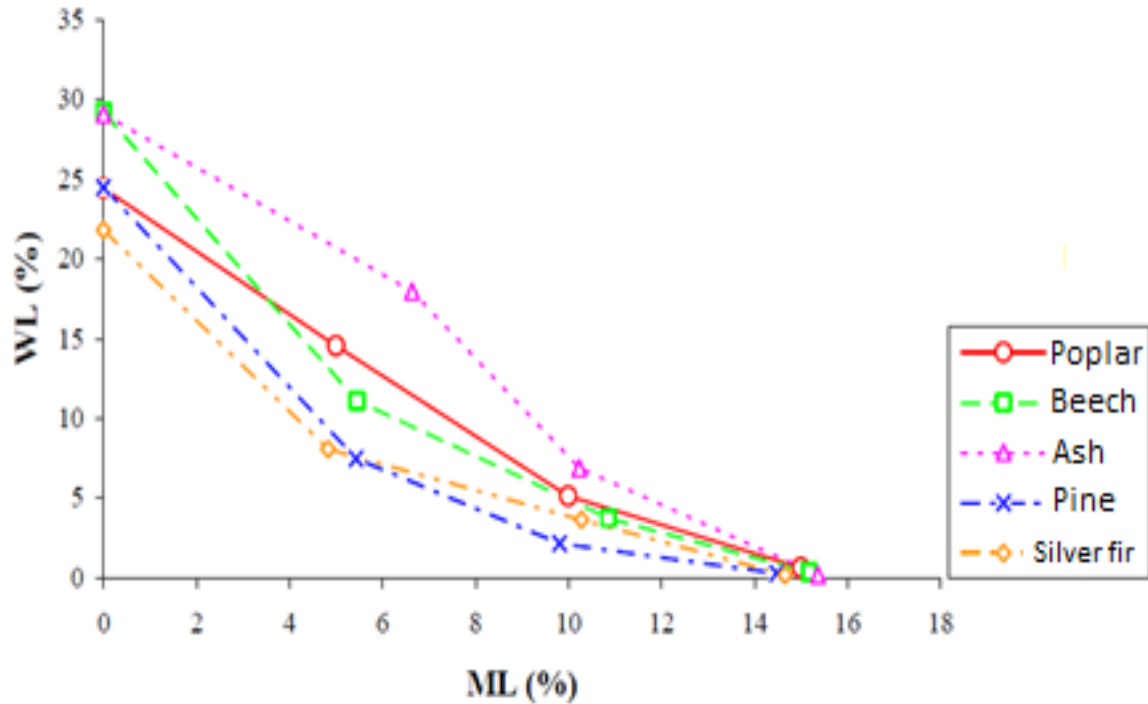


ML(%) evolution for different wood species heat treated at 230 °C according to treatment duration and temperature [Chaouch *et al.* 2010]

- Thermal degradation kinetic is influenced by the nature of wood species [Chaouch *et al.* 2010]
- ML (%) depends on Treatment intensity : temperature – duration [Candelier *et al.* 2011]
- ML (%) defines conditions of 1 treatment for 1 wood species

Context

Heat treatment of wood [Decay resistance prediction]



Correlation between weight loss by *Coniophora puteana* (WL %) and decrease in mass (ML %) for heat treatments on different European wood species at 230°C [Chaouch et al. 2010]

- Treatment intensity characterized by ML (%) could be an indicator to predict heat treated wood durability. [Kortileinen et al. 2000] [Chaouch et al. 2011]

Context

Industrial objectives (**Decay resistance = Modified wood quality**)

Wood thermal degradation is not controlled, due to heterogeneous heat transfer in treatment oven.

Obtain a good heat treated wood quality

Industrial target

Homogeneity and repeatability



Introduction of a fast and accurate system for measuring this mass loss on an industrial scale is very difficult.

- Find indicator control heat treatment process and to predict heat treated wood decay resistance
- Improve the process pilotage by developing of optimal treatment conditions, depending on the desired material properties.

Context

Decay resistance tests

(Issues)



- Standards usually used to test decay resistance of chemically treated wood, needed some amendments to evaluate the resistance of modified wood towards Basidiomycetes, such as a prolonged test exposure.

[Junga and Militz 2005]

- Biological resistance of the heat treated material to its low equilibrium moisture content during a decay test (i.e. the moisture content of the heat treated material is not enough for fungal growth). [Kamdem et al. 2009]

- Thus, durability tests on modified wood should be carried out over a exposure time (i.e. longer than 16 weeks of exposure) so as to overcome a mis-estimation of the durability of the new material (choice of fungi ?).

- **These laboratory wood decay resistance tests are destructive, time and money consuming and derivative methods to predict the durability of the heat-treated materials are crucially needed.**

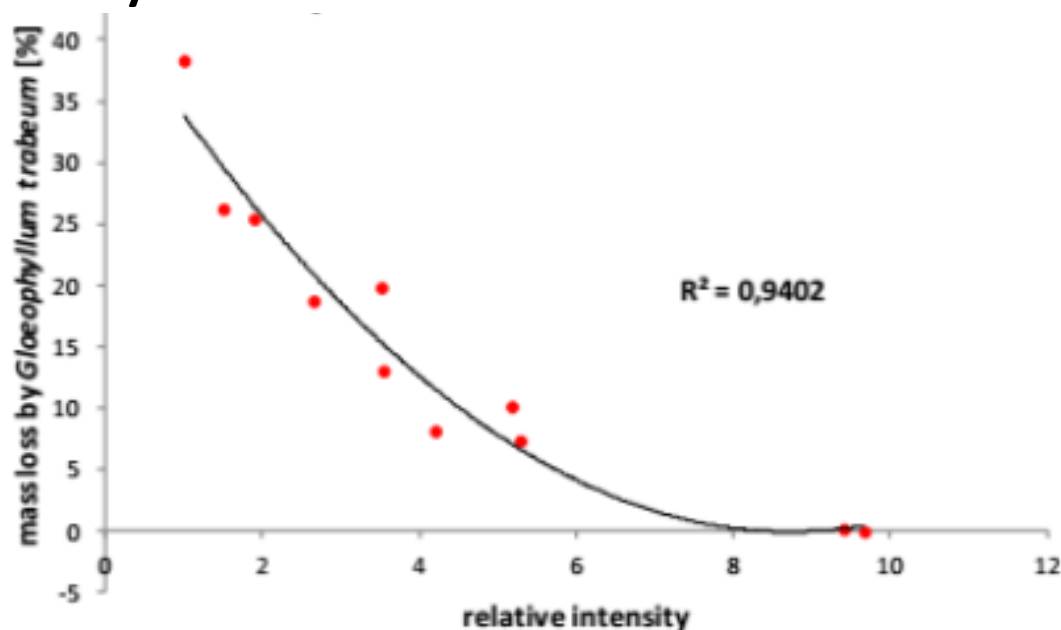
Decay resistance prediction

Spectrometric analysis

[Electron Spin Resonance - ESR]

- Measurement of microwave resonance absorbance in the presence of an applied magnetic field.
- For thermally modified wood, an increase of the ESR signal intensity can be measured due to the formation of free radicals during the process.

Estimation of decay resistance:



Correlation between weight loss by *Gloeophyllum trabeum* (WL %) and ESR- relative intensity
[Altgen et al. 2012]

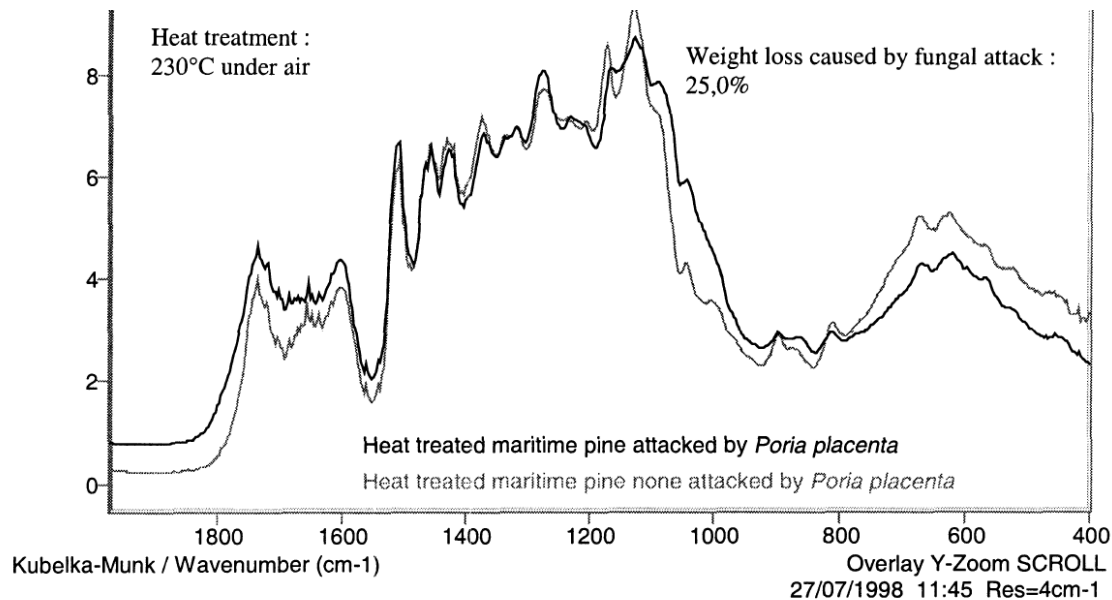
Decay resistance prediction

Spectrometric analysis

(DRIFT- Spectroscopy)

- Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS) is an infrared spectroscopy technique used on powder samples with no preparation.
- Possibility to create referential spectrum related to targeted heat treated wood quality by comparison on wood chemical composition modification.

Estimation of decay resistance:



Infrared spectra of thermal treated maritime pine degraded by *Poria placenta* [Weiland et al. 2003]

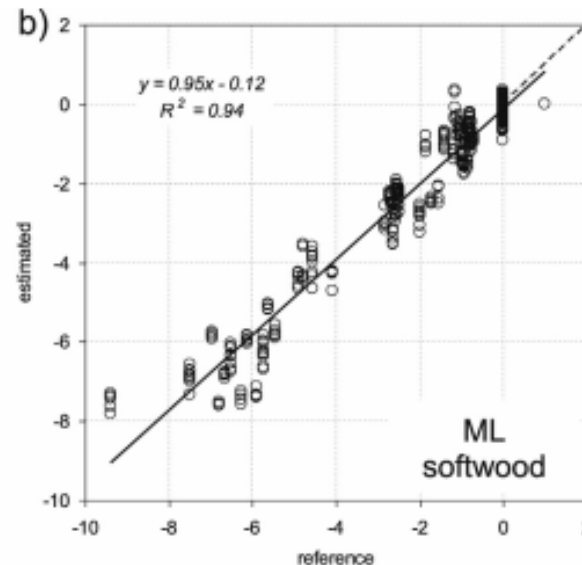
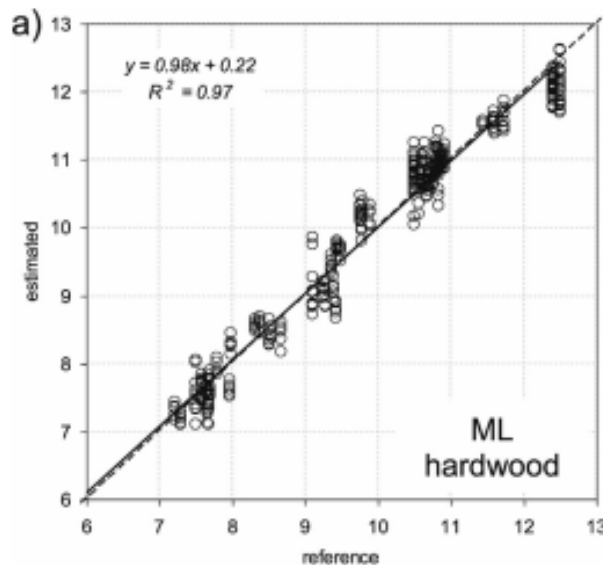
Decay resistance prediction

Spectrometric analysis

(NIR- Spectroscopy)

- Good potential to be used for heat treated wood quality checking. [Bächle et al. 2012]
- Both important parameters characterizing quality of TMW (EMC and ML) were predicted with high accuracy. [Sandak et al. 2015]
- No study has yet been conducted to predict directly heat treated wood durability.

Estimation of ML (%)



PLS predicted versus measured values of Mass Losses of thermally modified softwoods and hardwoods
[Sandak et al. 2005]

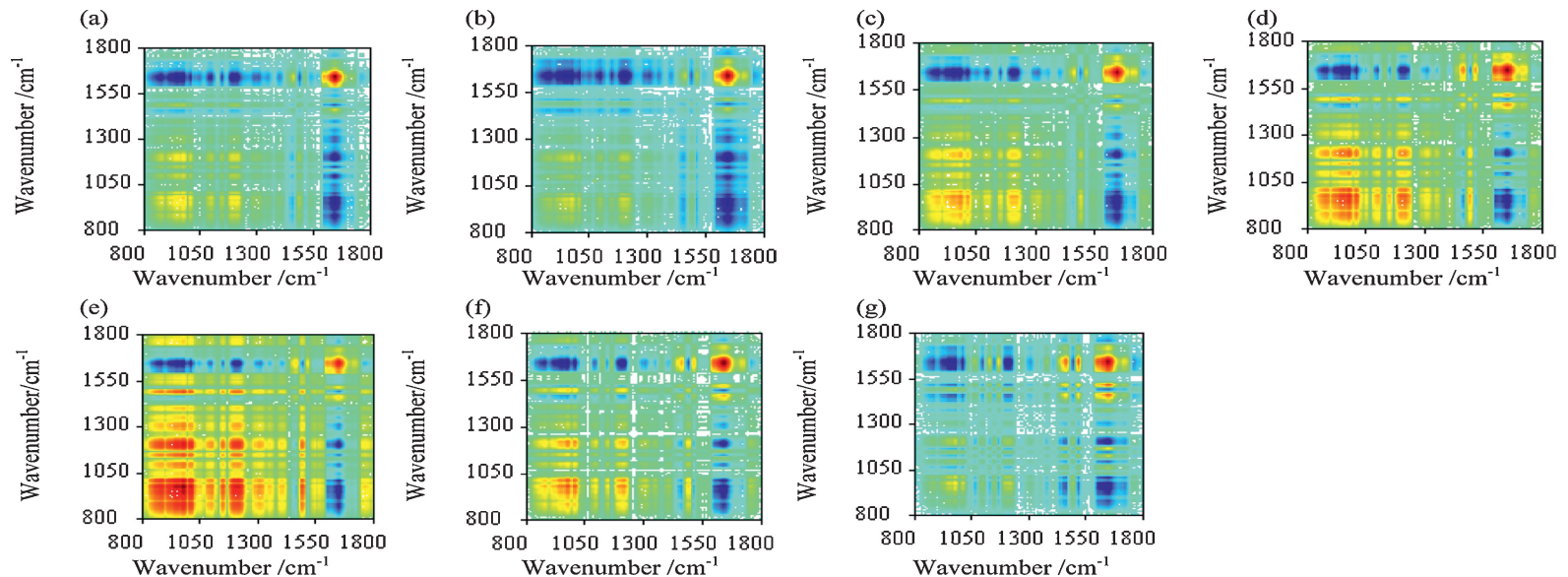
Decay resistance prediction

Spectrometric analysis

(FTIR- Spectroscopy)

- Advantages: accuracy, simplicity, and ability of performing very high number of tests without needs of any destruction to the material.
- FT-IR analysis method shows also good potential utilization to estimate heat treated wood durability, relating to heat treated wood chemical changes.

Estimation of decay resistance:



Synchronous 2D-IR spectra and auto-peaks of wood samples in the 1800–800 cm⁻¹ region of: (a) reference, steam-heat treated wood under (b) 120 °C, (c) 140 °C, (d) 160 °C, (e) 180 °C, (f) 200 °C, (g) 220 °C [Li et al. 2015].

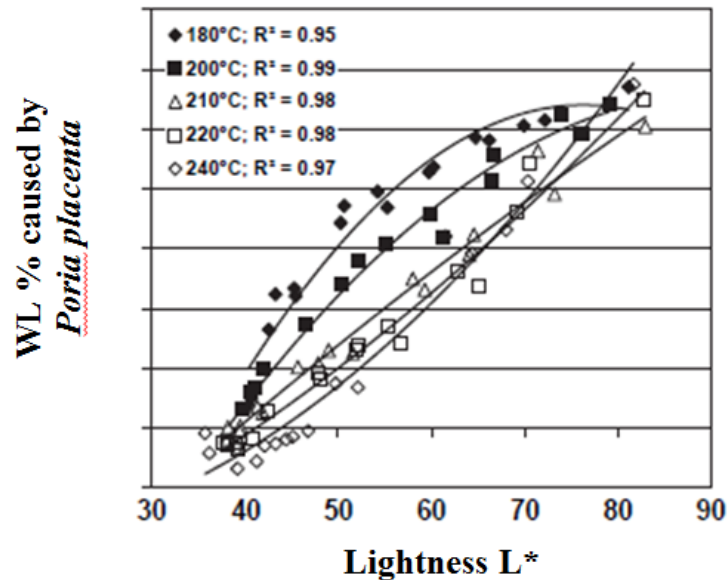
Decay resistance prediction

Colorimetric analyses

(CIELAB system)

- CIELAB system is the most commonly used method to estimate material color.
- Not suitable as predictor of heat treated wood quality because color distribution through the thermally treated boards is not homogeneous (earlywood / latewood, resin...)

Estimation of decay resistance:



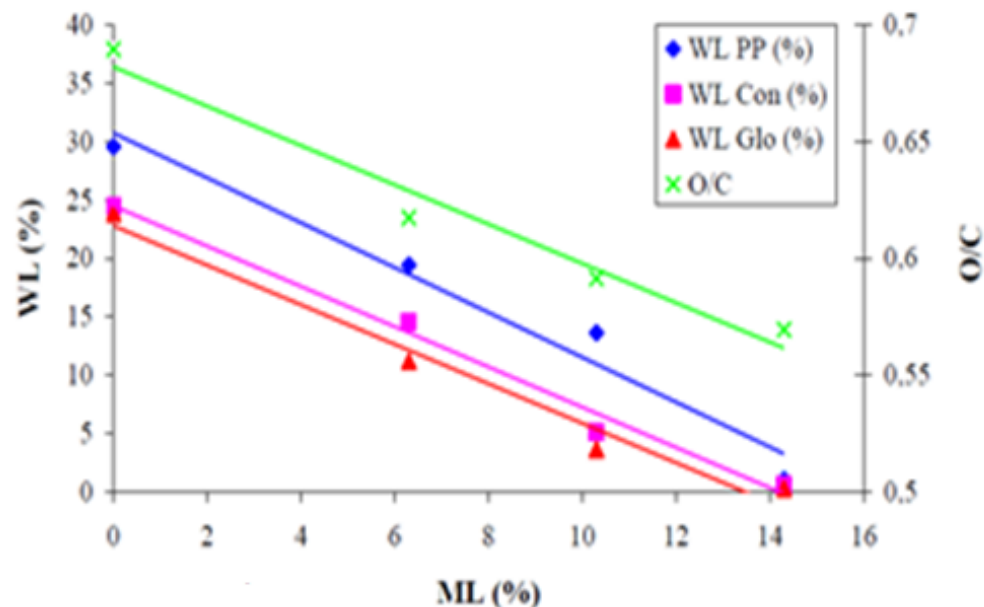
Correlation between weight loss by *Poria placenta* (WL %) and Lightness L^* , for different heat treatments on Norway spruce at different temperatures [Welzbacher et al. 2008]

Decay resistance prediction

Elemental Composition [O/C ratio]

- Carbon content increase according to the treatment intensity level.
- Such a measurement needs specific apparatus and time.

Estimation of decay resistance:



PP: *Poria placenta*
Con: *Coniophora puteana*
Glo: *Gloeophyllum trabeum*

Correlation between weight loss by different rots (WL), Mass loss due to heat treatment (ML) and O/C ratio, for heat treatments performed under nitrogen on poplar at 230°C [Chaouch 2011]

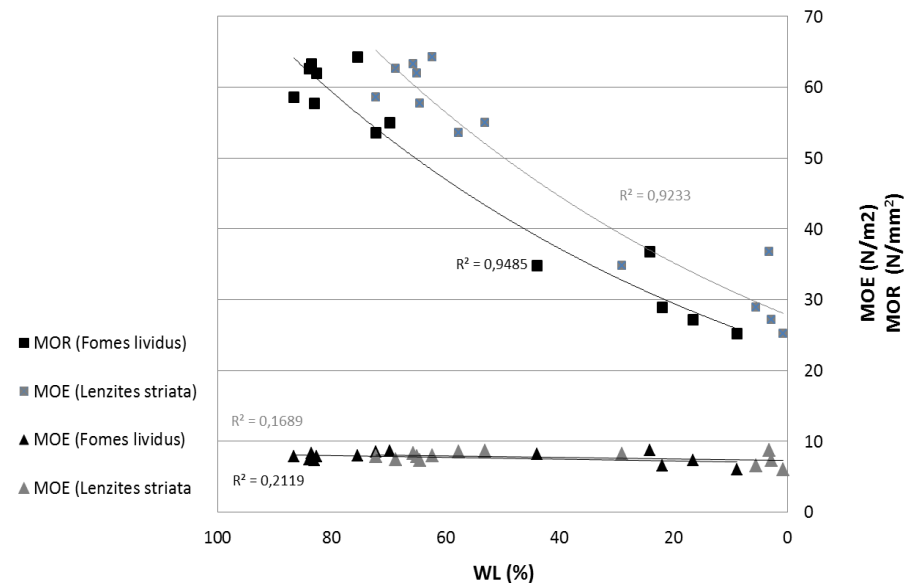
Decay resistance prediction

Industrial Data

(Mechanical properties)

- To perform non-destructive mechanical test to estimate heat treated wood decay resistance.
- Acoustic analysis could be a potential method to do it in an industrial scale.

Estimation of decay resistance:



Correlation between weight loss by two different rots (WL), MOE and MOR in bending destructive test, for heat treatments performed on Malapapaya wood (180-230°C, 30-120 min) [Jimenez et al. 2009]

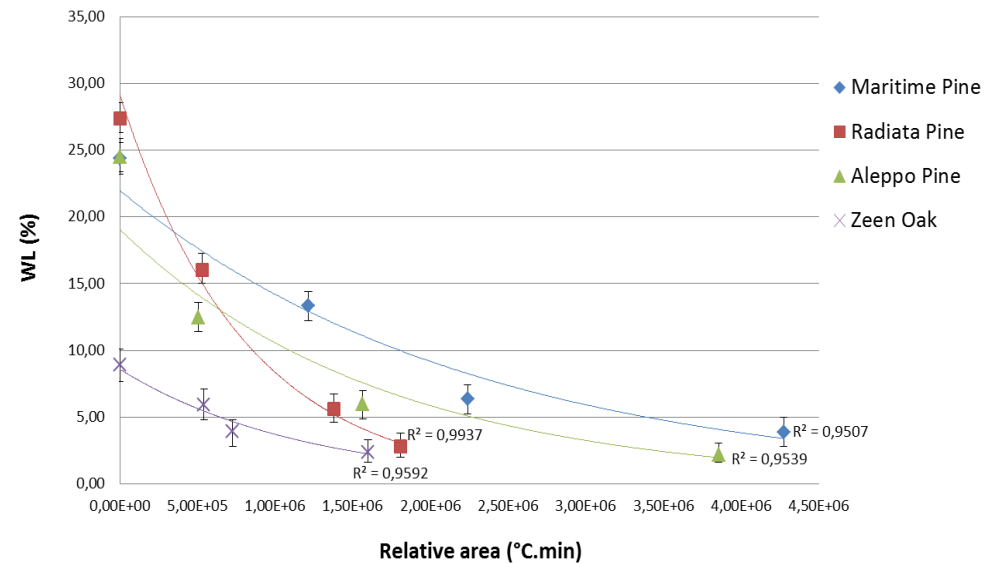
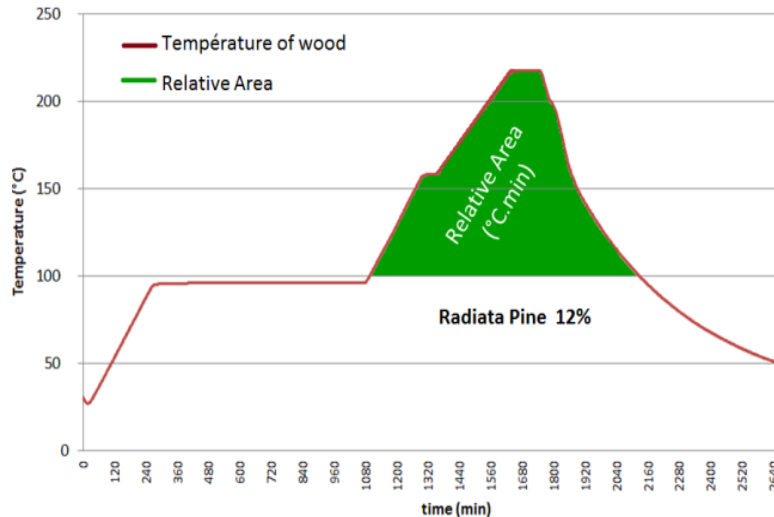
Decay resistance prediction

Industrial Data

(Temperature kinetics)

- Wood thermal modification depends mainly on treatment intensity related to temperature and duration.
- Temperature kinetics [$T^{\circ}\text{C} = f(\text{time})$] should be a good parameter to predict wood Mass Loss and also decay resistance.

Estimation of decay resistance:



Relative area determination and (b) Prediction of Weight Losses due to *Poria placenta* exposure by determination of relative Area, for different wood species [Candelier et al. 2015]

Conclusion

- **ML (%) is an efficient indicator to wood modification level and also, so far the most accurate indicator to heat treated wood decay resistance.**
- **ML (%) is difficult to be measured at industrial scale.**
- **Some analyses could predict this Mass Loss to estimate the wood decay resistance or could predict it directly.**
- **Color methods is too approximate**
- **Elemental or spectroscopic analyses need equipments and analysis time.**
- **Non destructive mechanical tests could be a great industrial solution, but mechanical – physical (MC%) relations remain to be check for heat treated wood.**
- **Temperature kinetics seem to be an interesting solution, but this method must be verified at industrial scale. This solution gives only a global estimation (not for each heat treated wood board).**

THANK FOR YOUR KIND ATTENTION

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