IECF 2024 Conference

The 4th International Conference on Forests

23–25 September 2024 | Online

MDPI

BEST CONDITIONS FOR ACETYLATION OF URUGUAY-GROWN *PINUS TAEDA*

María Eugenia Cardozo [1,*] , Pablo Raimonda [2] , Claudia Marcela Ibáñez[1]
[1] Laboratorio Forestal, Grupo Deterioro y Preservación, Centro Universitario Regional Noreste. UdelaR. Tacuarembó, Uruguay
[2] Departamento de Ensayo de Materiales, Facultad de Ingeniería, UdelaR. Montevideo, Uruguay,
meugeca@gmail.com

INTRODUCTION

Several preservation methods have been developed, tested and used to protect the wood and increase its resistance to deterioration. Today, there is a growing interest in non-biocidal wood protection methods.

Chemical modification of wood improves its performance, dimensional stability and esistance to deterioration, and once the wood is out of service, it can be disposed safely.

Chemical modification is the reaction between a chemical reagent and the hydroxyl groups of wood polymers, resulting in a single covalent bond.

In this study, the wood reacts with acetic anhydride (AA), resulting in the esterification of accessible hydroxyl groups in the cell wall; acetic acid forms as a by-product.

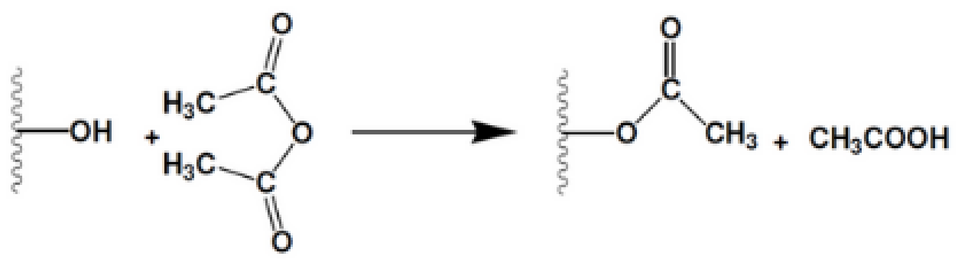

Figure 1: Acetylation reaction

When some hydroxyl groups of cell wall polymers are replaced with acetyl groups, the hygroscopicity of wood is reduced.

This work is the first study on the acetylation process of *Pinus taeda* wood, a species commercially used in Uruguay, with acetic anhydride (AA) in liquid phase.

METHOD

Samples

Pinus taeda was obtained from a contemporaneous and monospecific plantation Cerro Largo, Uruguay (2º33'95.59"S, 54º44'22.01"O).

Sapwood specimens (1,0 ± 0,3) mm x (1,0 ± 0,3) mm x (1,0 ± 0,3) were xtracted with ethanol for 24 hours in Soxhlet.

Acetylation reaction

Extractive-free wood was placed in a 1-liter stainless-steel reactor with temperature (type K thermocouple) and pressure (vacuo-manometer) control.

Acetylation process:

- Initial vacuum (30 min)
- Reaction mixture -K2CO3 (1,1 mmol/g of dry wood), N,N-dimetilformamide (DMF) and AA (97:3 v/v) was added.
- Pressure of 6 kg/m2 (60 min)
- Reaction mixture was removed; the reactor was heated with an electrical resistance for 2 and 6 hours at 60, 80 and 100°C.
  At the end of the process, the excess reagents were removed in Soxhlet equipment using Hexane:Ethanol:Acetone (4:1:1 v/v/v) for 8h.

Figure 3: Reactor

Factorial design

A factorial design 2^2 considering 3 repetitions in the midpoints.

The independent variables were the reaction temperature and the reaction time.

Wood weight gain percentage (WPG) and its chemical changes were used as response variables.

Figure 3: Wood samples

Durability assay:

The specimens modified under the best reaction conditions as determined previously were exposed to two fungi (EN 113 standard):

Trametes versicolor (white rot) and *Gloephyllum separium* (brown rot).

They were cultured for 120 days at 22ºC and 75% HR. Mass loss was evaluated

Figure 4: Culture flasks

RESULTS & DISCUSSION

Run	Temp (°C)	Time (h)	WPG (%)	Std. Dev
1	60	2	2,64	0
2	60	6	4,79	0
3	80	4	5,85	0,52
4	100	2	5,5	0
5	100	6	5,69	0

Table 1: weight percentage gain (WPG)

The highest mass gains were obtained under the highest temperature and longest time (6 hours, 100°C)

Statistical analysis of the design shows a higher incidence of the temperature variable on WPG results, not being as affected by time.

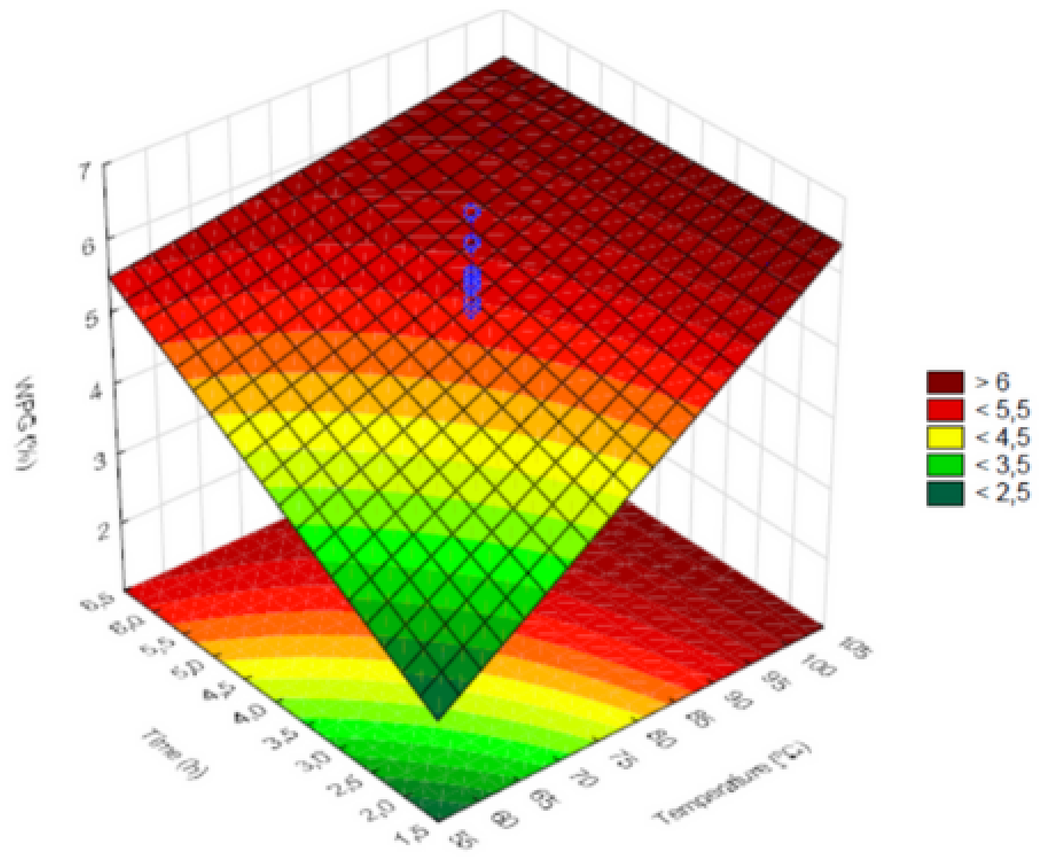

Figure 5: 2^2 design; MS Residual=,4706236 DV: WPG (%)

The highest WPG were obtained when the reaction temperature was over 100°C.

FTIR spectra of the acetylated wood under different conditions compared to not-acetylated wood were consistent with the results of the design analysis, in which the variable that resulted in the greatest differences in the spectra was a temperature of over 100°C.

	Control	Acetilated Samples
T. versicolor	22.08 (0,33)	2.03 (0.27)
G. separium	20.12 (0,23)	2.29 (0.42)

Table 2: Weight losses against wood rot fungi.

Wood was successfully protected against decay fungi, Mass losses of specimens were below 3%

CONCLUSION

The best acetylation conditions were **after impregnation** 6 hours of 100 °C

Temperature was the most important variable in the reaction process.

The treatment protects wood against decay fungi.

FUTURE WORK / REFERENCES

- Does the dimensional stability of the wood improve with modification?
- Can the process be carried out without the use of DMF?

1. Hill C. A. S.: Wood modification: Chemical, thermal and other processes. Chichester: Jhon Wiley and Sons. England, 2002.
2. Ibañez, C. M., "Madera, biodeterioro y preservantes," Montevideo: Facultad de Agronomía-Udelar, 2009.
3. Rowell R. 2014. Acetylation of wood – A review. International Journal of lignocellulosic products 1 (1): 1-27.
4. Jebrane M, Pichavant F, Sèbe G. 2011. A comparative study on the acetylation of wood by reaction with vinyl acetate and acetic anhydride. Carbohydrate Polymers 83: 339–345.