Optimization of the bi-oleothermal treatment process for wood preservation and fireproofing

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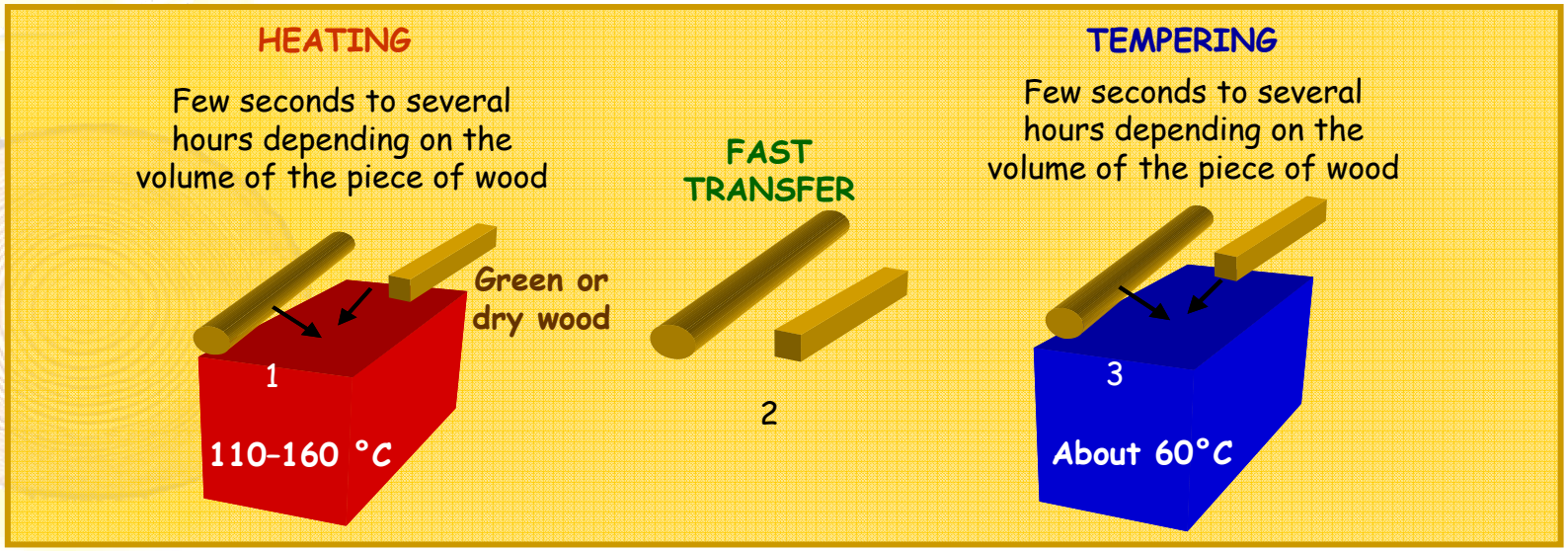
THE BI-OLEOTHERMAL TREATMENT

A 3-step wood treatment process developed by FCBA and CIRAD :

1. Dipping in hot oil (120-160°C), leading to the evaporation of the entrapped water and inducing a positive pressure inside of the wood pores
2. Fast transfer to a second bath
3. Dipping in cold oil (60°C), the thermal shock generating an inversed pressure and inducing the absorption of the fluid (oil + active substances)



PREVIOUS RESEARCH POINTED OUT SOME WEAKNESSES ...

Biological durability

- Oil treatment only slightly improved the resistance against fungal decay when compared to untreated wood (mass loss >3% with EN 113)
- Termites and longhorn beetles were able to damage wood treated with biocide-free oils

Fire behaviour

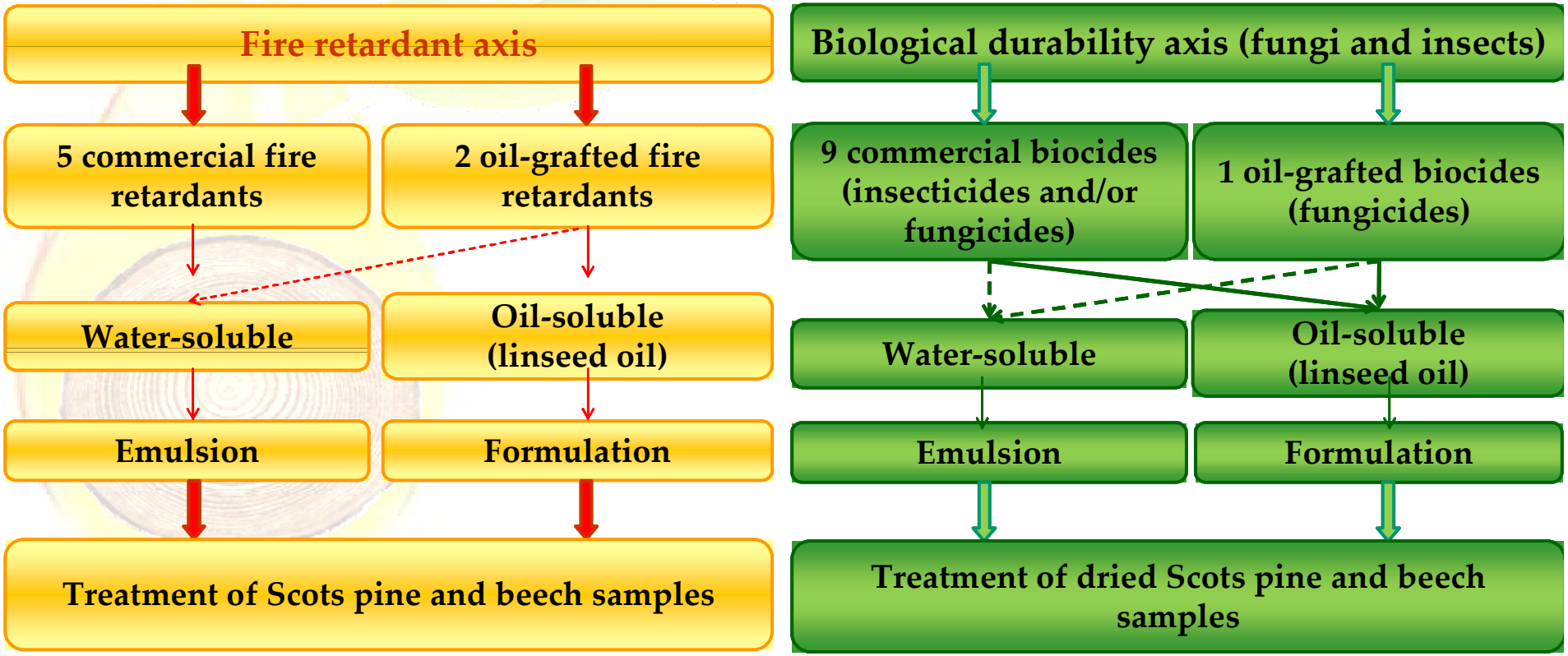
- Oil treatment had a negative impact on the fire behaviour of wood = decreased the fire hazard classification to F (dangerously reactive material), which is the worst possible case
- After traditional fireproofing (phosphate salts impregnation), the fire classification levels was increased to D / E

TO BE IMPROVED ...

By incorporating additives: biocides / fire-retardants

THE CHALLENGE : FOCUS ON THE SECOND BATH TO

- Improve the fire resistance of the wood by incorporating fire retardants
- Improve the fungi and insect resistance by incorporating optimized amounts of biocides = "fill the durability gap"



METHODOLOGY

AXIS	Type of Test	Test Methodology	Sample sizes (L,R,T) (mm^3)	Oil treatment parameters	
				1st bath	2nd bath
FIRE-PROOFING	Single flame	EN ISO 11925-2	125x20x88	20 minutes frying in Thermoleo® oil at +120°C	10 minutes soaking at +60°C in tested linseed oil-based formulations (added or not with biocides and fire-retardants)
	Cone calorimeter	ISO 5660-1	100x10x100	10 minutes frying in Thermoleo® oil at +120°C	
BIOLOGICAL DURABILITY	Decay fungi	Screening test	30x10x5		
	Molds	NF X 41-547 Field test	50x50x10		
	Subterranean termites	EN 117	25x25x6		
	Longhorn beetles	EN 47	50x25x15		

RESULTS OF FIRE RESISTANCE TESTING

. The single flame test (EN ISO 11925-2)

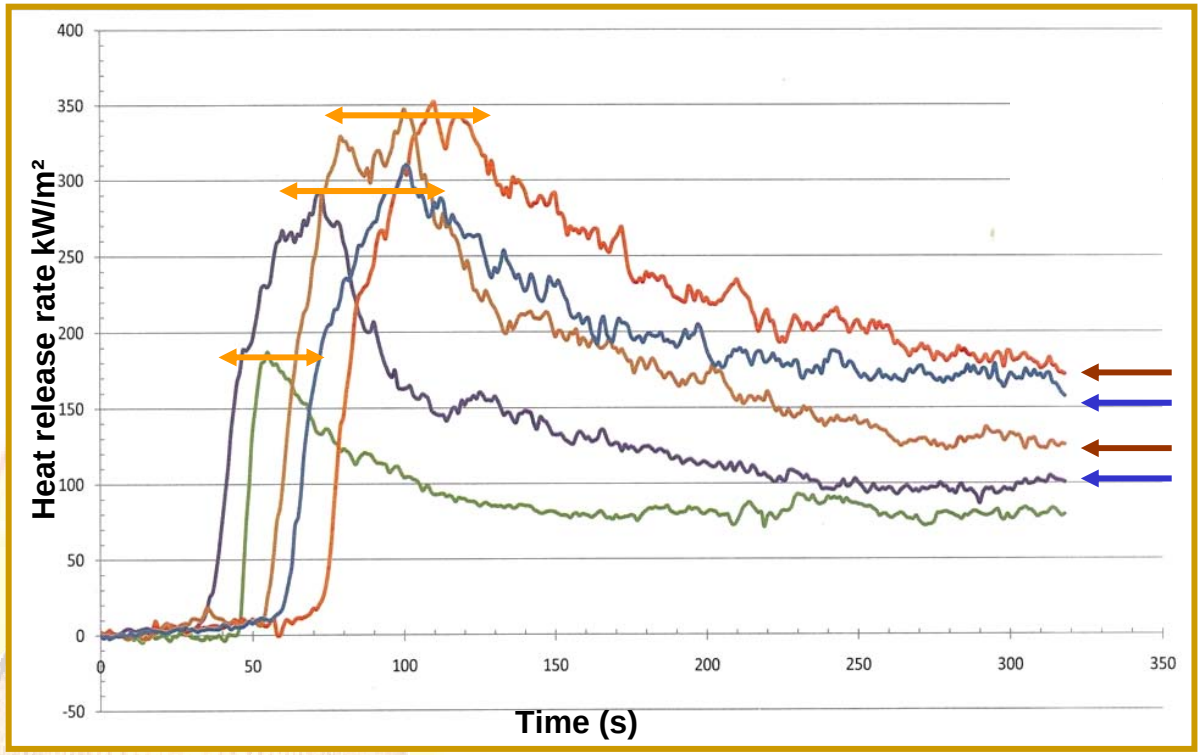
Samples (6 replicates)	Mean flame extinction (s)	Mean max flame height (mm)
Control (untreated Scots pine)	70	97
maleinized linseed oil free from fire-retardants	52	67
maleinized linseed oil grafted with THPC	44	57

The residual flame effect

- ✓ the treated samples displayed better fire behavior than the untreated ones
- ✓ the potential of the grafted maleinized oil with THPC seemed interesting

RESULTS OF FIRE RESISTANCE TESTING

. The cone calorimeter test (ISO 5660-1)



Untreated Scots pine

maleinized oil (0.5 M)

maleinized oil (1 M)

THPC grafted oil (%)

THPC grafted oil (%)

✓ HRR: maleinized oil > grafted oil > untreated wood

⇨ slight improvement with THPC but not enough

✓ HRR 0.5M > 1M maleinized oil and % > % THPC grafted oil

⇨ probably because of higher oil uptake (lower viscosity)

RESULTS OF BIOLOGICAL DURABILITY TESTING

. Resistance against wood decaying fungi (Bravery screening tests – FCBA/CIRAD)

Samples (6 replicates)	Mean mass loss (%)	
	C. puteana	*C. versicolor*
control (untreated Scots pine & beech)	> 40	> 30
biocide-free oils (2 formulations)	>3 (~ 7.5)	< 3
oils with fungicides (7 formulations/ 5 concentrations*)	>3 (~ 6.8)	< 3

* Ce/3 < C < Ce*2

✓ no effect of the added fungicides = inhibition during the process ?
✓ no dose-response (~ same results with all tested products and concentrations)
✓ lower fungal growth than controls

✓ linseed oil efficiently protect wood against CV ?
✓ moisture content too low to allow CV growth ?
✓ no additional effect of the tested fungicides

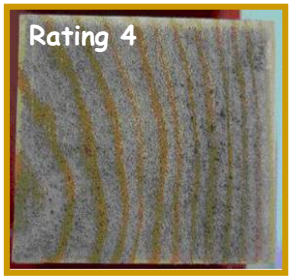
⇨ Biocide-free oils improved wood durability (DC 2 in EN350)

RESULTS OF BIOLOGICAL DURABILITY TESTING

. **Mold tests (NF X 41-547 …)**

o Mold strains: *P. funiculosum*, *A. niger*, and *T. viride*

o Mold development was rated from 0 (no development) to 4 (>50% of the wood surface)



Samples (6 replicates) / Mold rating	Lab test (spore inoculation) without weathering		6 months of natural weathering		2 weeks artificial weathering + spore inoculation	
	impregnation	surface application	impregnation	surface application	impregnation	surface application
control (untreated Scots pine)	6/4*	6/4	6/0	6/0	6/4	6/4
biocide-free oils (2 formulations)	6/0	11/0 – 1/1	6/0	6/0	6/0	6/0
oils with fungicides (2 formulations/ 3 concentrations)	6/0	6/0 3/0 – 3/1	6/0	6/0	6/0	6/0

* 6/4 = 6 samples rated 4

Slight mold growth with one tested biocide

No mold growth but 100% blue stain

No mold growth but blue stain with surface application

Efficient anti-mold protection of the tested oil formulations

RESULTS OF BIOLOGICAL DURABILITY TESTING

. **Resistance against the longhorn beetles *H. bajulus* (EN 47)**

ability of neonate larvae to damage wood samples
number of surviving larvae after 4 weeks exposure

Samples (6 replicates)	% of larvae that bored wood	% of surviving larvae
control (untreated Scots pine)	78	78
biocide-free oils (linseed oil & maleinized oil)	78	78
	50	5
oils with insecticides (4 formulations / 5 concentrations)	0 (2 F)	0
	0 - 63	

The larvae were able to bore into wood samples treated with blown linseed oil

Maleinized oil showed a protective efficiency

✓ Except at low concentrations of 2 formulations, larvae were not able to bore into wood samples treated with oil formulations containing insecticides
✓ In all cases, close to 100% mortality was reached

Efficient protection of biocide-free maleinized oil and insecticide added formulations

RESULTS OF BIOLOGICAL DURABILITY TESTING

. **Resistance against *R. flavipes* termites (EN 117)**

The degree of attack after 4 wks exposure ranged from 0= no attack to 4= strong attack

Samples (6 replicates)	Degree of attack	Survival %	
		CIRAD	FCBA
control (untreated Scots pine)	all rated 4	80	65
biocide-free linseed oils (2 formulations)	ratings from 1 to 3	0 - 21	3 - 0
oils with insecticides (4 formulations/ 5 to 6 concentrations)	ratings from 0 to 3	0	0*

*Except 1P/1C = 8%

Biocide-free oils do not efficiently protect wood

Biocide-free oils are toxic as the mortality was 100% at the end of the test for almost all sets

A dose-response effect was reported for all formulations containing insecticides with the 3 highest tested concentrations for CIRAD test and only the highest for FCBA test

Biocide-free oils did not avoid termite attacks but are toxic

Tested insecticides showed unexpected bad efficacy, suggesting that their uptake or efficacy have been lowered during the process (inhibition reactions with oils ?)

FOLLOW-UP OF THE BIOCIDES IN TREATED WOOD

3 fungicides and 3 insecticides were tracked in the treated wood samples to confirm the uptake of the targeted doses by comparing measured and theoretical values (M and T)

Retention of the active ingredients (kg/m^3)	Fungicides				Insecticides			
	Beech		Scots pine		Beech		Scots pine	
	T	M	T	M	T	M	T	M
R/5	1.94	0.30	2.53	0.40				
R/7	12.00	2.58	5.54	0.95				
R/8	12.50	1.84	4.65	0.53	12.50	6.70	4.65	1.65
R/9	25.30	5.35	9.50	1.66	25.30	9.24	9.50	3.28
R/10	26.90	6.30	10.90	2.28	26.90	10.80	10.90	4.30

✓ Huge difference between the targeted values and those measured in the wood samples, for both species and all of the tested fungicides and insecticides:
4-6 X smaller for fungicides / 2-3 X smaller for insecticides

⇨ The active ingredients were either destroyed in the bath or did not penetrate into the wood samples with the oil

⇨ Subsequent tracking in the oil bath showed that active ingredient were present at the expected concentrations but did not enter the wood samples

CONCLUSIONS & FUTURE WORK

FIRE-PROOFING

- maleinized oil grafted with THPC did not protect wood efficiently
- many technical problems occurred when working with grafted oils

→ Future development will focus on the formulation of more stable emulsions

BIOLOGICAL DURABILITY

- biocide-free blown and/or maleinized oil significantly reduced the risk for the wood of being decayed by insects and fungi
- oils added with biocides did not perform as good as expected
- interactions / inhibitions were suspected to occur between the oils and the biocides, lowering their uptake / efficiency

→ An understanding of these phenomena are necessary before selecting the more efficient biocides and scaling-up the process

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THANK YOU !

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