Deliverable n°T2.D2.5.3

Prototype of a highly durable POP panel
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KAÏROS

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Partners

PP Leader : Kaïros

Partners involved : Portsmouth, UBS, Ecotechnilin

Content

1 Context of activity 2.5 – WPT2

In this activity, Kaïros has developed new composite materials, with a monolithic and sandwich structure, using the non-woven preform of slightly oriented flax fibres manufactured by Écotechnilin. These materials are intended for use in point-of-sale advertising. As a result, their surface finishes must be smooth and free of visible defects to meet the aesthetic challenges of this field of application. The environmental footprint of these new materials is reduced thanks to their high recyclability and compostability potential and the use of biosourced raw materials. Kaïros has to ensure that the materials meet the specifications imposed by the POP sector (machinability, aesthetic appearance, light weight, good mechanical strength) while at the same time checking that they can be recycled. These materials are produced using the thermocompression process, which ensures short manufacturing cycle times and low processing costs. Numerous tests, such as mechanical strength tests in different environments, UV ageing tests and scratch resistance tests, are carried out to characterise the new material. The results obtained enable a detailed technical data sheet to be drawn up for the material, enabling it to be compared with conventional petro-sourced materials. The manufacture of composite sheets is also intended to produce a prototype of a typical POP product. The production of a piece of POS furniture will demonstrate the robustness of the material in this field of application.

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2 Prototype of a highly durable POP panel (Livrable T2.D2.5.3)

Several prototype composite sheets with a format of 2,500 mm x 1,300 mm, suitable for POS applications, were produced.

2.1 Materials used

A biosourced and/or biodegradable composite sheet is any composite material with a monolithic or sandwich structure composed of:

- a biosourced and/or biodegradable reinforcement;
- a biosourced and/or biodegradable thermoplastic or thermosetting matrix;
- a biosourced and/or biodegradable core.

Biosourced composite sheets include the following components.

2.1.1 Non-woven preform

The preform, developed and supplied by Ecotechnilin (WPT1), consists of flax fibres co-mingled/mixed with thermoplastic poly lactic acid (PLA) fibres using a carding and needling process so that the fibres are slightly oriented in the direction of the reel unwind. The PLA fibres incorporated into this new reinforcement were loaded with 10% calcium carbonate ($CaCO_3$).

The proportion by weight of flax in the preform is 40%, while the loaded PLA fibres make up 60%. The weight of the comélé is 150 g/m2. The material shown in Figure 1 is supplied by EcoTechnilin, which selected the flax fibres and designed this new non-woven architecture.

Figure 1 : Roll of non-woven flax fibres/CaCO3-filled PLA fibres

For the panels developed for fire resistance, a conventional 350 g/m² non-woven preform was used.

2.1.2 Cork core

One of the sheets manufactured has a sandwich structure with agglomerated cork as the core. The cork used is 8 mm thick and has a density of 250 kg/m3.

2.1.3 PLA film

PLA films are added to the surface of the composite sheet to remedy the problems of porosity on the surface of the composite sheet and obtain a more aesthetic surface finish.

Two films are used:

- A transparent PLA film with a thickness of 350 µm and a weight of 435 g/m².
- A white PLA film filled with $CaCO_3$ and talc (filler content: 30%), 500 µm thick and weighing 740 g/m²

To improve the fire resistance of the sheets, two different PLA films were produced from compounds produced in the laboratory using the following formulations:

- PLA + 30 w% aluminium Trihydroxyde (ATH)
- PLA + 15 w% chestnut tannins + 15 w% lignin

The granules obtained after extrusion are placed in an aluminium frame, then melted and cooled to form a film using the thermocompression process.

In addition, white PLA film with $CaCO_3$ and talc fillers (filler content: 30 w%), thickness 500 µm and weight 740 g/m² is used as a reference.

2.2 Stacking sequence

2.2.1 Fire-resistant panels

Three different compositions were tested:

- A reference composition (1 small 20cm*20cm panel) with the following sequence:
  - 1 filled white PLA film
  - 3 conventional 350 g/m² non-woven preforms
  - 1 filled white PLA film
- A composition with ATH (2 small panels 20*20 cm) with the following sequence:
  - 1 PLA film + 30 % ATH
  - 3 conventional 350 g/m² non-woven preforms
  - 1 PLA film + 30 % ATH
- A composition with tannins/lignin (2 small 20*20 cm panels) with the following sequence:
  - 1 PLA film + 15 % tannins + 15 % lignin
  - 3 conventional 350 g/m² non-woven preforms
  - 1 PLA film + 15 % tannins + 15 % lignin

2.2.2 Durable panels with a new preform

Four sheets with different stacking sequences were produced, with the aim of obtaining a smooth, shiny surface with no apparent defects. Their different draping patterns are detailed in Figure 2.

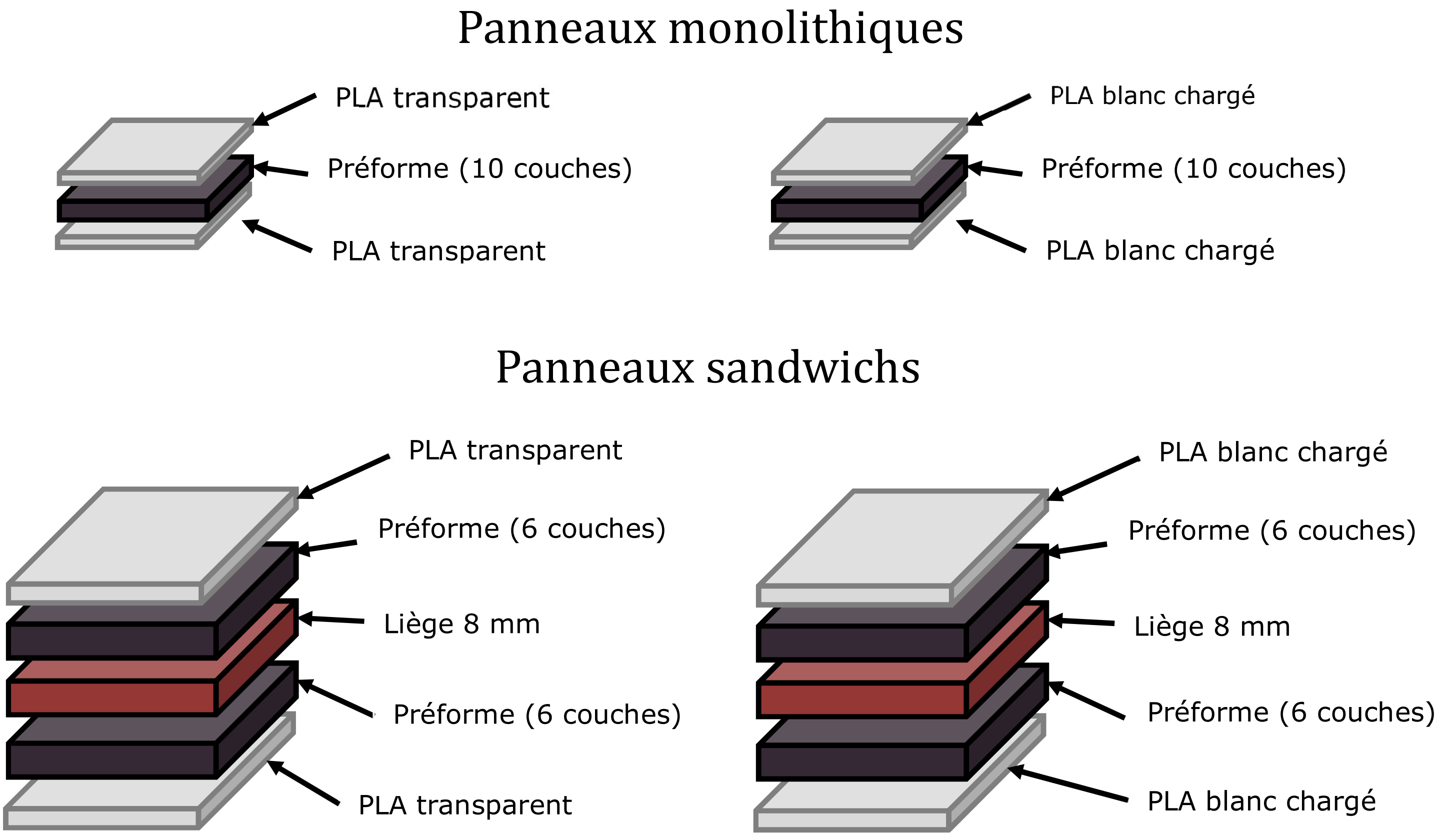


Figure 2: Explanations of the different stacking sequences

Although Figure 2 shows 4 stacks, they were carried out on two panels (2400 mm x 1200 mm after trimming) because the characterisation tests do not require a material surface of an entire panel to be carried out. Figure 3 therefore shows two panels, one monolithic and one sandwich, with the two different films on either side of the panels.

2.3 Fabrication process

The stacked comelts are inserted between two metal plates and then placed in a hot press at 200°C to melt the thermoplastic material. The temperature of the press is set according to the melting temperature of the PLA, and must be below the degradation temperature of the flax fibres. The whole assembly is then transferred to a cold-plate press (10°C) to preserve the amorphous state of the polymer. Optimisation of the process through the acquisition of a chiller has improved the surface finish of the panels through rapid cooling.

The material is applied in the stages described below:

1. Stacking according to the stacking sequences described in 2.2.
2. Insertion of the stack between two mirror-polished stainless steel plates.
3. The whole assembly is heated and pressed in a hot press until the thermoplastic melts. The stacking of the material between two mirror-polished stainless steel plates is used to give the biocomposite material a shiny surface appearance and to transfer it from the hot press to the cold press.
4. The stainless steel plates containing the skin/core stack are pressed into a cold mould to consolidate the sandwich stack.
5. Opening of the press/tooling and demoulding of the stainless steel plates comprising the final biocomposite plate.

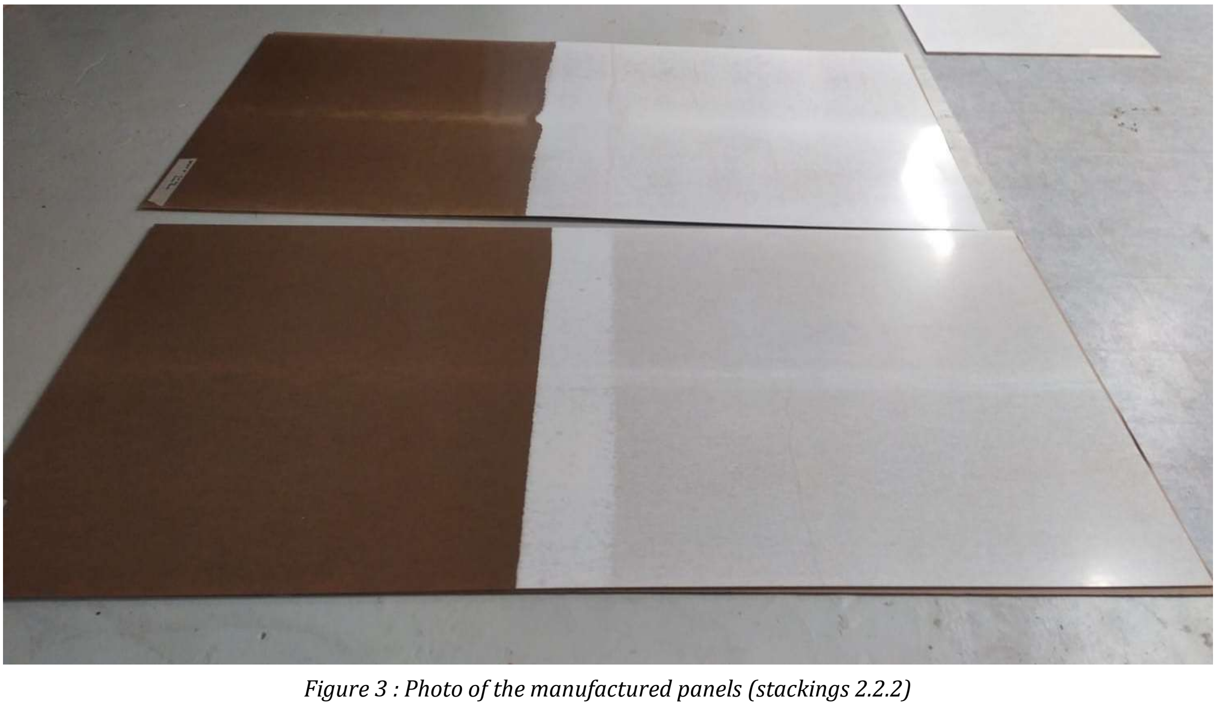


Figure 3 : Photo of the manufactured panels (stackings 2.2.2)

2.4 UV and humidity ageing tests

Accelerated UV and moisture ageing tests were carried out to verify the durability of the solution. After being exposed in the laboratory to various UV and humidity conditions, each sample was tested mechanically for hardness and roughness.

The names of the samples are as follows:

- CNW: The panel composed of the standard preform and transparent PLA on the surface, no filler present
- NNW : The panel consisting of the preform developed here and transparent PLA on the surface
- NNW/$CaCO_3$: The panel made up of the preform developed here and white PLA with a surface charge

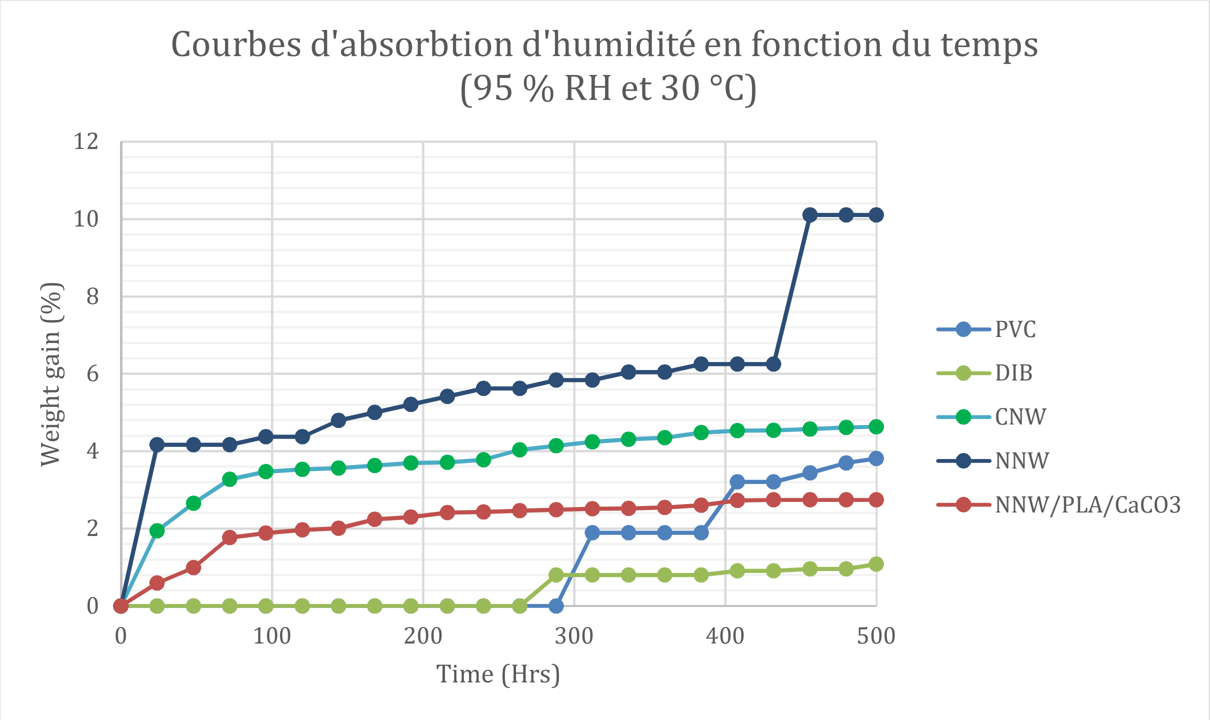


Figure 4 : Moisture absorption results by sample

Figure 4 shows that water absorption is greatly reduced by the addition of fillers on the surface and in the preform. NNW absorbs more water than CNW because the flax fibres in NNW are finer and purer than in CNW, which makes them more sensitive to moisture absorption, due to the exposure of the cellulose layers of the fibres, which are hydrophilic and rich in hydroxyl groups (OH).
PVC absorbs more water after 500 hours than NNW/CaCO3, which is stable after 300 hours. Dibond (DIB) is made of aluminium and polyethylene, two hydrophobic materials that absorb very little water.

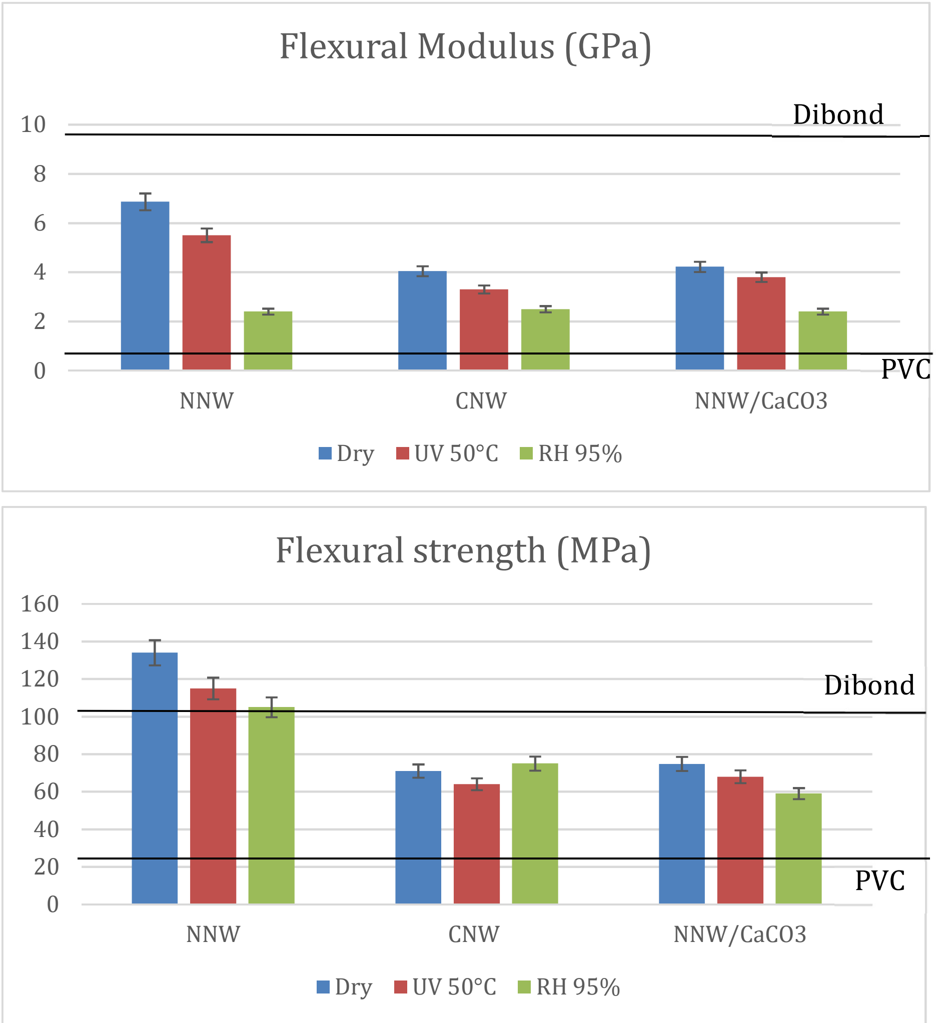
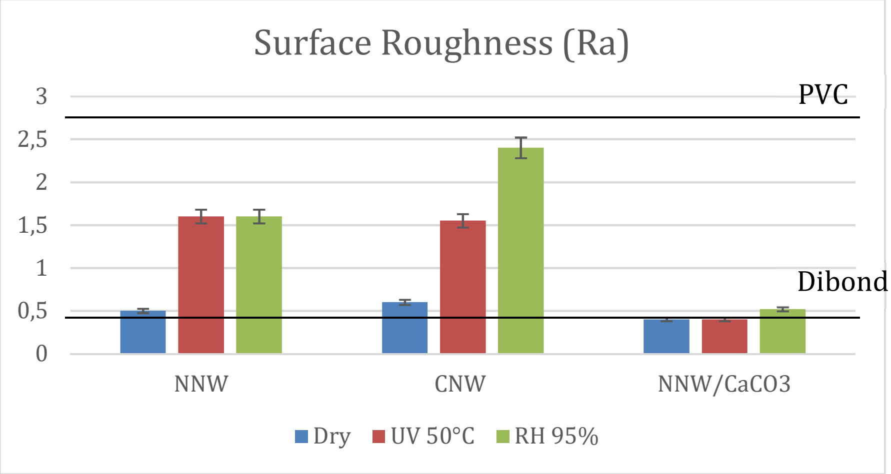


Figure 5 : Résultats des tests mécaniques en flexion des échantillons vieillis et non vieillis

Figure 5 shows the mechanical properties of the samples before and after ageing (UV or humidity). Ageing affects the mechanical strength of the panels, but the mechanical properties remain far superior to those of PVC, proving that ageing of the panels is not prohibitive for the applications concerned.

With regard to roughness, Figure 6 shows that the addition of surface fillers (NNW/CaCO3) maintains a low surface roughness after ageing. The fillers limit surface cracks caused by chain scission and polymer recrystallisation. NNW has a lower roughness than CNW after ageing in moisture. The presence of fillers in the preforms (no fillers for CNW) seems to attenuate this increase in roughness after ageing.
In terms of hardness, UV ageing has little impact on this property, whereas exposure to moisture makes the material less hard, but still superior to the hardness of PVC.



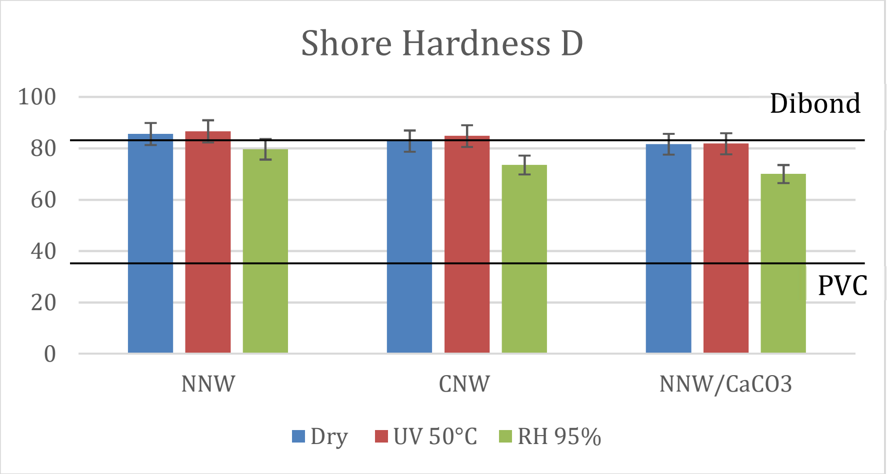


Figure 6 : Résultats des tests de dureté et de rugosité des échantillons vieillis et non vieillis

2.5 Fire resistance tests

The fire resistance tests are radiation tests and comply with standard NF P 92-501. They were used to characterise the sheets by calculating a "Q" quotient, which enables them to be classified according to the M classification of standard NF P 92-507.

The results are shown in the table below:

COMPOSITION	CLASSEMENT M (QUOTIENT Q)
RÉFÉRENCE	- M4 (58.04)
LIGNINE + TANINS	- M4 (56.73) - M4 (54.64)
ATH	- M3 (32.81) - M3 (36.03)

The composition lignin + tannins did not provide the desired flame retardant effect. The aromatic compounds that make up lignin and tannins could be used to create a layer called a 'char' during thermal decomposition, which would create a barrier layer to protect the material and slow down its degradation. Here, it could be that the concentration of lignin and tannins is not high enough, or that their purity needs to be improved to achieve M3 classification.

ATH reacts differently to thermal degradation. The decomposition of aluminium hydroxide is endothermic and ATH decomposes into aluminium oxide (Al_2O_3), releasing water molecules which help to retard flames. Its effect is visible in tests, as the material achieves M3 classification.

3 Conclusion

The addition of $CaCO_3$ fillers to the PLA fibres in the preforms means that they are more resistant over time to external stresses (UV, humidity). The panel that appears to be the most durable is the one where the fillers are both on the surface (white PLA film with fillers) and in the core (PLA fibres with fillers). The effect of the fillers can be seen in the change in roughness, where after 500 h of ageing it remains equivalent to its initial level.

As far as fire resistance is concerned, ATH is currently the only method tested that has a flame-retardant effect, enabling the slabs to be classified in the M3 category of standard NF P 92-507.

In this extension, the production of the new flax/PLA preforms was slowed down by the delay in the supply of PLA fibres, developed specifically for this extension. We have therefore focused our tests on ageing in order to gain a better understanding of ageing mechanisms and their impact on mechanical properties and roughness. The tests we have carried out on this point go beyond what was originally envisaged. However, the biodegradation tests, initially planned, could not be carried out due to lack of time. They will be carried out after the project, using our own funds, and will supplement those already carried out during the first phase of the project.