

INDUSTRIAL SCALE-UP OF BIO-BASED INSULATING PANEL PRODUCTION.

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Abstract

ISOBIO Project aims to produce highly insulating panels from bio-resources. This study focuses on the constraints associated to the production of panel at the industrial scale using thermal treatment. Three types of processes are considered (wet processes for rigid panels, dry processes for rigid or flexible panels and Stramit process®) and the limits of each process are identified according to the formulation characteristics (moisture content, binder type and content), binding mechanisms (melting, thermosetting or drying), process parameters (pressure, time and temperature) and targeted material properties (density and thickness). Cavac Biomatériaux industrial oven is used to study the dry and wet processes for the production of rigid panels. The temperature distribution in the material thickness was recorded. Difficulties to reach sufficient temperature level for high panel thicknesses or for high water content are observed. As a consequence, an excessively long drying time is needed to produce rigid panel using the wet process. Only small panel thicknesses are possible options. The addition of thermosetting binder is an alternative way to reduce water content and processing time. Rigid panel are easily produced using the dry manufacturing process and a hydraulic press with hot plates. Various tests are performed adjusting panel characteristics and formulations. Interesting results are obtained with hemp shiv bonded with a bio-based thermosetting binder. The binder content ranged from 5% to 23% and the targeted density from 155 to 245 kg/m³. Mechanical characterization shows that the binder formulation, proportion, water content and panel density are crucial parameters to be adjusted to optimize panel manufacturing process and properties. The dry process appears more versatile than wet process and leads to lower processing time. It allows the production of hemp shiv insulation panels within a large range of density and having compressive and flexural strength reaching 1 and 1.7 MPa respectively.

Keywords

Industrial production, thermal insulating panel, bio-based material.

1 INTRODUCTION

This study takes part of the ISOBIO project which aims to produce bio-based insulation materials from agricultural wastes with high thermal performances and low embodied energy. In this paper, work focuses on design and production of low density thermal insulation panels. Mineral binders are excluded because of their high densities which penalize the thermal conductivity of the composites. Several studies focused on the development of such low-density material composed of bio based aggregates bound with an organic biobased binder [Mati-Baouche 2015] [Palumbo 2015] [Umurigirwa 2014] [Viel 2017]. They demonstrate that low thermal conductivity, high moisture buffering value and sufficient mechanical properties can be reached. However, few studies present how the material manufactured at the laboratory scale could be produced at the industrial scale. This study deals with this issue.

Based on the existing technologies and processes used for lignocellulosic materials production, identify constraints associated to the industrial scale manufacture of biobased insulation panel is necessary in order to optimize the material formulation according to the targeted properties but also to the manufacturing process.

Three industrial manufacturing processes of lignocellulosic insulation materials are considered: the wet and the dry process inspired from the wood industry and the Stramit process®. These processes requiring a thermal treatment differ in the binding mechanisms of bio-based aggregates involved during processing.

The wet process is an adaptation from the paper manufacturing technology as it involves high amount of water. Indeed, aggregates or fibers are bound with the self-binding properties of lignocellulosic chemical constituents (lignins, hemicelluloses) or with "drying

adhesives” which develop their binding properties after thermal treatments and water evaporation. This process was the most used to produce low density rigid wood fiber insulation panel.

The dry process requires the use of thermoset or thermoplastic binders which respectively polymerize or melt by heat activation. Using this process, the moisture in the binder-lignocellulosic aggregates mix before the thermal treatment can be lower than 20% which allows to remove the evaporating step required in the wet process. Low density flexible or rigid panels can be manufactured using this process. The binder content (weight proportion) is generally lower than 10% of the composite, even lower than 5% for rigid panels using highly reactive PMDI binder. [Thoemen 2010]

The Stramit process® (<http://stramitinternational.com/>) allows the production self-bonded wheat straw panel by making the most of the advantages of the wet and dry wood insulation manufacturing processes. Indeed, this process does not require the addition of a binder (as the wet process) and does not requires the addition of water to the raw aggregates (as the dry process). The panel is manufactured through an extrusion process. The combination of heat, pressure and moisture initially present in the raw aggregates allows to mobilize lignins and hemicelluloses which act as a binder.

The work focus on the production of bio-based aggregates panels. The first part focuses on the manufacture of rigid panels using Cavac industrial oven. Two formulations are tested: a self-bonding wheat straw panel and a hemp shiv composite bound with a synthetic thermosetting resin. The binding conditions, and the release of water during thermal treatment are analyzed. The second part focuses on the manufacture of panels composed of hemp shiv bound with a bio-based thermosetting binder using a hot thermopressing process (traditional way of production for dry process). The effect of the composite and binder formulation on the panel processability and the resulting mechanical properties will be studied.

2 MATERIALS AND METHODS

2.1 Raw materials

Biobased aggregates:

A commercial building grade of hemp shiv (Biofibat®, Cavac Biomatériaux) is used. Wheat straw is crushed and calibrated using Cavac defibering line.

Binder:

Two types of binders are tested: a synthetic acrylic binder (Acrodur 950L®, BASF) and a bio-based thermosetting binder. The bio-based thermosetting binder is formulated using bio-based water soluble macromolecules (matrix) and a crosslinker. Five different binder formulations are tested (Tab. 29).

Tab. 29: Thermosetting bio-based binder formulations

Binder reference	Matrix dry proportion (w/w%)	Crosslinker dry proportion (w/w%)
C5%	95	5
C10%	90	10
C20%	80	20
C30%	70	30
C35%	65	35

The solid contents of the acrylic binder and of the bio-based thermosetting binder are 50w% and 40±2w% respectively (w/w).

2.2 Cavac Industrial oven

This oven is currently used to produce industrially low density (30 – 40 kg/m³) flexible panels from hemp, flax and cotton vegetable fibers such as the product Biofib Trio® (Cavac Biomatériaux, France). This dry manufacturing process can be divided according to the following steps: vegetable fiber mixing with thermoplastic bicomponent fibers (bio-based or synthetic), mat forming on the continuous belt and mat heating in a continuous oven illustrated Fig. 63. The oven is composed of three heating chambers and two cooling chambers. In the heating chambers, the heat is transferred through the mat using a hot air flow. The temperature of the air can be set between 60°C and 200°C and the flow can be controlled. The fiber mat is

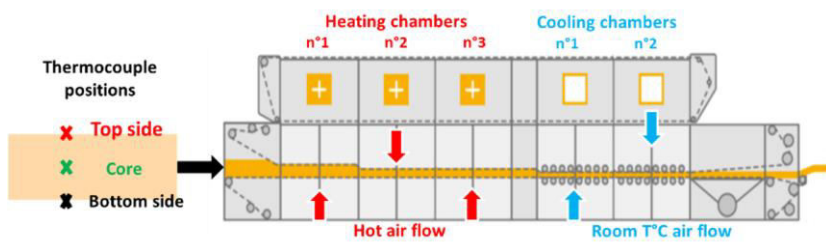


Fig. 63: Cavac Biomatériaux industrial oven

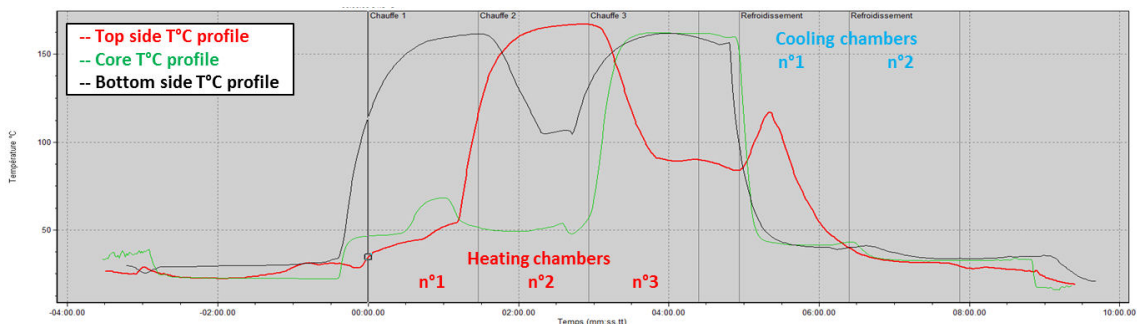


Fig. 64: Temperature profiling of Cavac industrial oven

transported at a defined speed in the oven using two conveying belts on both side of the mat. For each chambers the spacing between the superior and inferior belts can be adjusted which allows to calibrate the final material thickness and density. The thermal treatment in the heating chambers melts the thermoplastic fibers which bond the vegetable fibers after solidification. The solidification is ensured in the cooling chambers using air at room temperature.

During thermal treatment, the temperature profile has been recorded using thermocouples placed on the top, the core and the bottom of the fiber mat before it passes through the oven. K type thermocouples and a Datapaq® (Fluke) recorder were used to record the temperature profile. Fig. 64 illustrates the temperature profile of a 200mm thick flexible insulation panel manufacturing process. The oven temperatures were set at 165,170 and 165°C in the heating chamber 1, 2 and 3 respectively and the conveyor belts speed at 1.5 m/min. The melting temperature at the core is only obtained in the third heating chamber. Such results show the difficulty to manufacture thick or dense panels.

2.3 Panel manufacturing using Cavac industrial oven

Mixing:

Aggregates are mixed with the liquid binder or with water using a laboratory blender (Sama). Aggregates are first introduced in the blender and then the liquid is progressively added. The mixing is maintained during 2 min. Tested formulations are listed in Tab. 30.

Tab. 30: Tested composite formulation

Aggregates		Binder		Water
Type	w/w%	Type	w/w%	L/S
Wheat straw	100	None	0	1.5
Hemp shiv	87	Acrodur	13	0.25;0.5; 1

Mat forming:

The mat forming step is conducted on the conveyor belt at the oven entrance. The aggregates/binder mix is disposed in a flexible mould of internal dimension 400x300 mm² (or 600x600 mm²) and manually dispersed in order to obtain a homogeneous mat. Two air permeable protective layers are placed on the top and the bottom of the mat surfaces to prevent adhesion to the conveyor belt. Targeted final thickness is 50 mm.

Pre-pressing step (optional)

Some mats are cold pre-pressed using hydraulic press before placing on the conveying belt. This step is conducted in a steel mould of internal dimension 400x300 mm. A pressure of 400 kPa is applied on the mat (Fig. 66). The pre-pressed mat is carefully demolded and disposed on the conveying belt in the flexible mould.

Thermal treatment using the industrial oven:

The conveyor belt is used to transport the hemp shiv/binder mat through the oven. The heating conditions are controlled by adjusting oven parameters (air temperatures and flow, thicknesses and conveying belt speed). Using the heating chambers dimension, the heating time is adjusted by controlling the conveyor

belt speed (min 1.5 m/min). If additional curing time is required, the conveying belt is stopped when the mat is located in a heating chamber.



Fig. 65: Mat forming on the conveyor belt



Fig. 66: Cold pre-pressing step device

2.4 Panel manufacturing using the thermopressing process

Mixing:

The mixing hemp of shiv with the bio-based thermosetting binder is ensured using an Imal Pal wood laboratory gluing blender containing spraying nozzles illustrated Fig. 67. The exact weight of each constituent is easily controlled as the hemp shiv are introduced directly into the drum blender and the totality of binder to be sprayed is placed in the liquid resin container. The mixing time is set to 4 min. After the mixing is started, the binder is sprayed in the drum blender through the spraying nozzles using the Venturi effect. Tested formulation with thermopressing process are illustrated in Tab. 31. The five bio-based thermosetting binder presented in Tab. 29 are used.



Fig. 67: Imal Pal laboratory blender with spraying nozzles

Three levels of crosslinker proportion in the composite are defined according to the classification presented Tab. 32.

Mat forming

The mat is formed in two wood frames of internal dimension 600x600 mm. A 1mm thick aluminum plate

is placed under the wood frames. The bottom wood frame having a height of 50 mm has the function of mould. The top wood frame allows to the mat formation in the mould. The hemp shiv/binder mix is introduced in the two frames and manually dispersed to form a homogeneous mat. The mat is manually pre-pressed using a wood plate. Finally, the top wood frame is removed and a second aluminum plate is placed over the mat.

Tab. 31: Thermopressed panel formulation

Panel n°	Targeted density (kg/m ³)	Binder		Panel composition (w/w%)		
		Ref	Content (%)	Hemp shiv	Matrix	Cross-linker
1	170	C10%	8%	92%	7.2%	0.8%
2	170	C10%	20%	80%	18.0%	2.0%
3	170	C30%	8%	92%	5.6%	2.4%
4	170	C30%	20%	80%	14.0%	6.0%
5	230	C10%	8%	92%	7.2%	0.8%
6	230	C10%	20%	80%	18.0%	2.0%
7	230	C30%	8%	92%	5.6%	2.4%
8	230	C30%	20%	80%	14.0%	6.0%
9	200	C20%	14%	86%	11.2%	2.8%
10	200	C20%	14%	86%	11.2%	2.8%
11	200	C20%	23%	77%	18.4%	4.6%
12	200	C20%	5%	95%	4.5%	0.5%
13	200	C35%	14%	86%	9.1%	4.9%
14	200	C05%	14%	86%	13.3%	0.7%
15	245	C20%	14%	86%	11.2%	2.8%
16	155	C20%	14%	86%	11.2%	2.8%
18	200	C20%	14%	86%	11.2%	2.8%
19	200	C20%	14%	86%	11.2%	2.8%

Panel thermopressing

This step is conducted using an ENERPAC hydraulic hot press having a capacity of 200 tons. It is composed of two heating plates. The temperature of each plate can be set up to 250°C. The mat situated in the wood mould and maintained by the aluminum layers, is placed between the two plates of the press. The press is closed until both plates are in contact with the wood mould. The pressing time is set to 15min at 180°C. The density of the panel is controlled by adjusting the weight of material inserted in the mould (dimensions 600x600x50 mm³). After pressing, the panel is removed from the press, cooled at air for 10 min and demoulded.

Tab. 32: Classification of the crosslinker proportion in the composite

Crosslinker proportion in the composite	Criteria
Low	$W\%_{crosslinker} \leq 2\%$
Moderate	$2\% < W\%_{crosslinker} \leq 4\%$
High	$4\% < W\%_{crosslinker}$



Fig. 68: Mat forming, pressing and cutting of thermopressed panels

2.5 Characterization

Sample preparation

Each panel is cut to produce five samples (dimensions 50x50x50 mm³) for the compressive tests and 3 samples (dimensions 260x120x50 mm³) for the three points bending test. Samples are stabilized during at least two weeks in a climatic chamber at 23°C and 50%RH before testing. The composite density of each sample is measured before testing. The hemp shiv content is calculated using the panel composition according the following formula:

$$HS = \rho_{Comp} \times W\%_{hemp\ shiv} \tag{1}$$

With:

HS: hemp shiv content in the composite (kg/m³)

ρ_{Comp} : composite density (kg/m³)

$W\%_{hemp\ shiv}$: hemp shiv proportion (w/w%)

Compressive test

The compressive tests are performed using an Instron loading machine at a constant displacement velocity of 5 mm/min and ended at a displacement of 15mm (≈30% strain). Standard EN 826 specifies to measure a compressive strength at 10% strain ($\sigma_{c,10}$) [AFNOR 2013a]. However the strength ($\sigma_{c,max}$) reached when the elastic deformation changes to elastoplastic deformation is used as compressive strength which corresponds to the change of slope as illustrated Fig. 69. Compressive elastic modulus is evaluated by fitting the linear part of the recording stress versus strain.

Flexural testing

The flexural strength and flexural elastic modulus are evaluated according to the three points bending test (standard EN 12089) using an Instron loading machine [AFNOR 2013b]. The distance between the two supporting pins is set to 225 mm (4.5 x t). The constant displacement velocity of the loading pin is set to 10 mm/min.

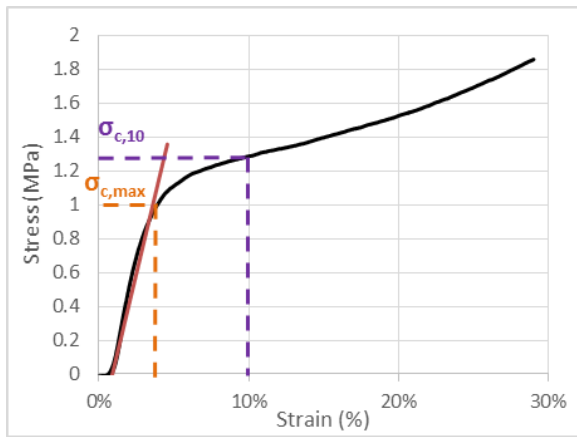


Fig. 69: Illustration of compressive test analysis



Fig. 70: Compressive test



Fig. 71: Flexural test

3 RESULTS AND DISCUSSION

3.1 Manufacturing process using Cavac industrial oven

Self-bonded wheat straw panel:

A partial cohesion is obtained with the self-bonded wheat straw panels (around 200 kg/m³) as illustrated Fig. 72 which shows the binding properties of lignins and hemicelluloses after thermal treatment in wet condition. However, to develop sufficient cohesion between aggregates, a combination of heat, moisture and pressure is required. One can suppose that the pressure applied in the oven during heating is not sufficient to provide lignins and hemicelluloses flowing. Indeed, Stramit process leads to high density panel production (350-450 kg/m³) requiring high pressure level. With the tested oven, such pressure level cannot be reached and air flow induces vapor release reducing the hygrothermal activation. A pulping step involving high liquid/water ratio such as those used in wood fiber insulation board may be required to produce material in the expected range of density/pressure.



Fig. 72: Wheat straw panel after thermal treatment

Hemp shiv/Acrodur® panel

All hemp shiv panels produced (around 200 kg/m³) without the pre-pressing step are weak and friable (Fig. 73). Using the pre-pressing step, rigid panels with good cohesion are obtained. This information provides that the pressure applied by the belt which controls the

mat thickness in the oven cannot supply enough pressure to reach targeted density of rigid hemp shiv panels. The heating time required to cure the panel is strongly linked to the water/solid ratio increasing from 17 to 40 min for a water/solid ratio of 0.25 and 1 respectively (Tab. 33: *Heating time vs liquid/solid ratio*). It can be explained by the difficulties to heat and thus evaporate moisture at panel core as observed for flexible panels during the thermal profiling Fig. 64. This proves that the wet process, involving high water/solid ratio, is not suitable for the production of thick panel industrially.



Fig. 73: Hemp shiv/Acrodur® composite manufactured using Cavac oven. (On the left, without the pre-pressing step. On the right, with the pre-pressing step)

Tab. 33: Heating time vs liquid/solid ratio

Formulation	Liquid/Solid ratio	Total heating time required (min)
Hemp shiv/Acrodur 950L®	0.25	17
	0.5	22
	1	40

3.2 Manufacturing using thermopressing process.



Fig. 74: Bio based insulation panel manufactured using the thermopressing process

Fig. 74 illustrates a 60x60x5 cm³ panel manufactured using the thermopressing process. This process allows to cure the panels in 15 min for all the formulation presented Tab. 31.

Effect of formulation on dimension compliance:

Tab. 34 shows thicknesses for several thermopressed panels at a thickness of 50mm.

Tab. 34: Panel thicknesses after thermopressing for different binders.

Panel N°	Binder		Panel Thickness (mm)	
	Ref	w/w%		
5	C10%	8	57.51	± 0.234
6	C10%	20	56.53	± 1.022
7	C30%	8	50.45	± 0.137
8	C30%	20	48.42	± 0.469

The targeted density of these panels is 230 kg/m³ which is closed to the upper limit of our range of densities. Panels 7 and 8 manufactured using the binder containing 30% of crosslinker (C30%) have final thicknesses very close to the 50mm target. However, panel 5 and 6 manufactured with the binder containing 10% of crosslinker (C10%) have respectively 6.53 mm and 7.51 mm over the targeted value. This means that the binder with 10% of crosslinker (C10%) does not provide enough cohesion between aggregates so that the panel thickness can be maintained at the press opening. This is furthermore confirmed by the fact that increasing the binder proportion does not improve the panel cohesion because panel 5 and 6 have respectively 8 and 20%w/w of binder. Thus, these results show that the crosslinker is crucial to respect the targeted panel dimension.

Effect of water content:

Panels 2,4,6,8 and 11 containing high proportion of binder (20 – 23%) were weaker just after pressing and not fully dried whether for panels bound with binder containing 10% of crosslinker (C10%) or 30% (C30%). High binder content induces high water content is the mix. It increases the amount of heat required to evaporates the water and reach the crosslinking temperature at panel core. Thus, the lower the water content before pressing, the faster the panel can be manufactured. This also justify the importance of looking for high solid content binder solution so that the added water can be reduced.

Compressive testing:

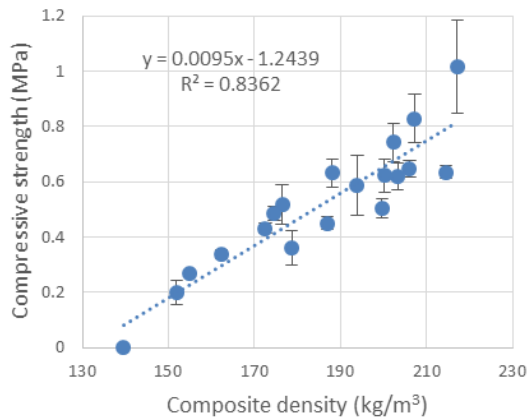


Fig. 75: Compressive strength ($\sigma_{c,max}$) vs composite density

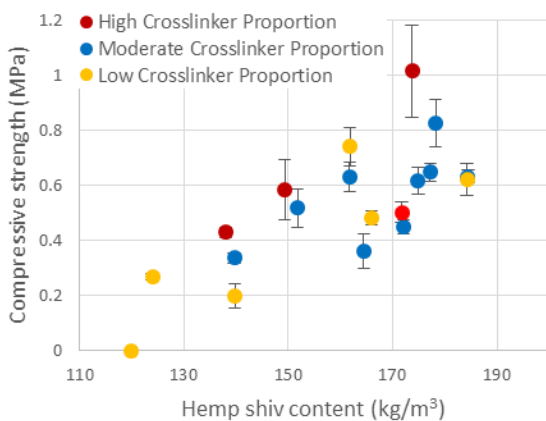


Fig. 76: Effect of crosslinker proportion and hemp shiv content on the compressive strength ($\sigma_{c,max}$)

Fig. 75 illustrates the compressive strength ($\sigma_{c,max}$) versus the composite density. One can see that the compressive strength is strongly correlated to the composite density. The compressive strength evolves almost linearly between 0 MPa and 1.01 MPa for samples having densities of 139 kg/m³ and 217 kg/m³ respectively. For the sample having a density of 139 kg/m³, $\sigma_{c,max}$ cannot be determined during mechanical tests because as soon as the compression starts, a plastic deformation is observed. It is important to note that all the composites do not have the same formulation (binder and crosslinker proportion) which means that the crosslinker proportion may not have a significant effect on the compressive strength. These observations are confirmed by Fig. 76 which shows the development of the compressive strength versus the hemp shiv content according to the three levels of crosslinker proportion.

One can see that the compressive strength increases with the hemp shiv content. However, for the same the range of density (i.e. hemp shiv content of 160 kg/m³), composites having a low crosslinker proportion can have higher compressive strength than composites having a moderate crosslinker proportion. This means that the compressive strength is mostly correlated to the composite density rather than to the crosslinker proportion. The highlighted red point corresponds to panel 13 having a high crosslinker proportion. A discussion about this panel will be developed later.

Fig. 77 illustrates compressive elastic modulus versus the hemp shiv content for the three crosslinker proportions. One can see that the crosslinker proportion has an important effect on the compressive elastic modulus. Furthermore, a synergic effect can be observed between the crosslinker proportion and the hemp shiv content. At a hemp shiv content of approximately 140 kg/m³, the compressive elastic modulus for a low, moderate and high crosslinker proportions are 7, 13 and 19 MPa respectively but at a hemp shiv content of approximately 170 kg/m³, the values increase to 9, 20 and 37 MPa respectively.

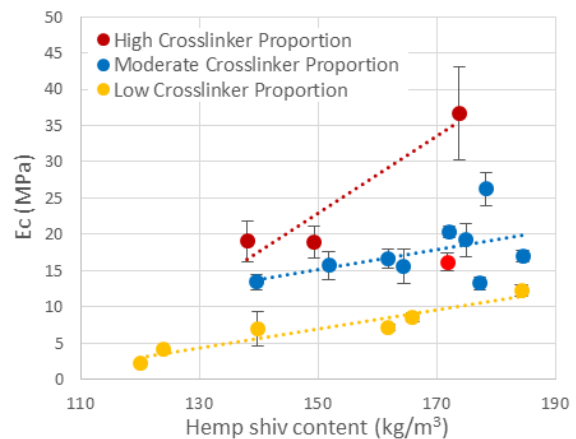


Fig. 77: Compressive elastic modulus vs hemp shiv content. The highlighted red point corresponds to panel 13 having a high crosslinker proportion.

Flexural testing:

Fig. 78 and Fig. 79. present respectively the flexural strength and elastic modulus developments versus the hemp shiv content for the three crosslinker proportions. One can see that the flexural strength as well as the flexural elastic modulus are directly correlated to the crosslinker proportion and hemp shiv content. A low synergy between hemp shiv content and crosslinker

proportion is observed for the flexural strength but not as important as for the elastic modulus. For a hemp shiv content of approximately 140 kg/m^3 , the flexural strength for a low, moderate and high crosslinker proportions are 0.29, 0.58 and 82 MPa respectively and the elastic modulus are 25, 52 and 82 MPa respectively. At a hemp shiv content of approximately 170 kg/m^3 , the flexural strength increases respectively to 0.83, 1.24 and 1.77 (low, moderate and high) and the elastic modulus to 45, 85 and 150 MPa.

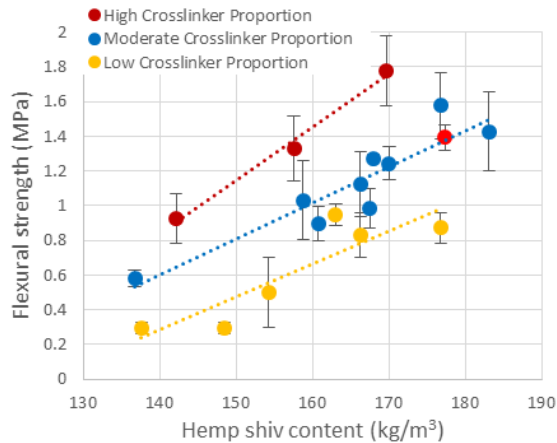


Fig. 78: Flexural strength vs hemp shiv content. The highlighted red point corresponds to panel 13 having a high crosslinker proportion.

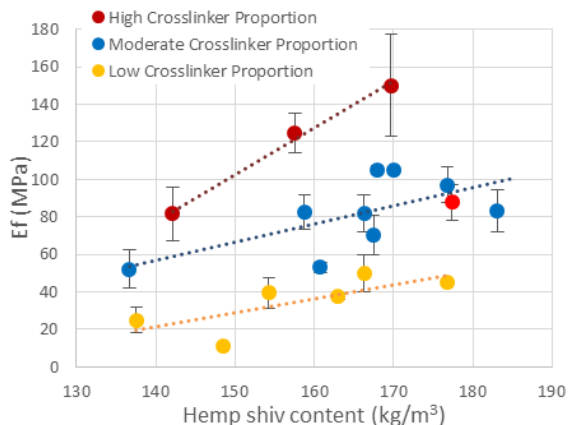


Fig. 79: Flexural elastic modulus vs hemp shiv content. The highlighted red point corresponds to panel 13 having a high crosslinker proportion.

The highlighted red point in Fig. 76, Fig. 77, Fig. 78 and Fig. 79 corresponds to the panel 13. This panel is the only one bond with the binder C35% having a very high crosslinker concentration (65% of matrix and 35% of crosslinker). With a crosslinker proportion of 4.9%, this panel is normally classified as a high crosslinker proportion panel according to the classification presented Tab. 32. However, the results obtained with this binder do not fit with the corresponding expected mechanical properties. Indeed, as one can see on Fig. 76, Fig. 77, Fig. 78 and Fig. 79, the obtained results are similar to a moderate crosslinker content. This could be explained by the fact that beyond a crosslinker concentration of 30% in the binder, the unlinked excess of crosslinker leads to a decrease of global binding properties.

4 CONCLUSION

In this paper, the manufacturing constraints associated with the industrial production of biobased insulation panel were studied.

The study on Cavac industrial oven showed that the heating time is strongly linked to the water content of the mat. The production of self-bonded wheat straw panel requires adjustment of the water/solid ratio and of the pressure during the thermal treatment. These conditions were not reached using Cavac industrial oven for the targeted panel density. This study shows that, using the wet process, it is complex to control panel density and extensive thermal treatment are required to dry material core. Such process use is then limited to the production of low thicknesses material for a narrow range of density. The use of a synthetic thermosetting binder allowed to reduce the water content and thus the heating time to reach panel cohesion. Using Cavac industrial oven the pressure applied by the conveyor belt was not sufficient to reach the targeted density for hemp shiv composite. The addition of a pre-pressing step was necessary to obtain good cohesion. This shows that the oven is not adapted to produce rigid insulation panel.

The hot thermopressing process was the most suitable to produce low density hemp shiv insulation panel bound with a bio-based thermosetting binder. Indeed, using this process, the density can be easily controlled and reduced as low as 150 kg/m^3 . The heating time is considerably reduced due to the low moisture content of the mat before thermal treatment. The mechanical properties obtained for the targeted density range are higher than other wood fiber insulation product having a compressive stress at 10% strain generally lower than 0.2 MPa. Hemp shiv insulation panel can reach a compressive strength as high as 1 MPa. The study of the effect of binder formulation on the mechanical properties showed that the crosslinker proportion is a crucial parameter to provide dimension compliance and to maintain good mechanical properties at very low density.

Using this study, the production of low density hemp shiv panel can be easily transposed at the industrial scale because the panel formulation and manufacturing process can be adapted to comply with already existing industrial hot thermopressing process. Furthermore, this study allows to optimize the processing time (reduced embodied energy), the material cost and the thermal performance by adjusting the panel density and formulation (the binder content and the crosslinker proportion) to the desired mechanical performances.

5 ACKNOWLEDGMENTS

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