

NEW PARTICLEBOARDS BASED ON AGRICULTURAL BYPRODUCTS: PHYSICO-CHEMICAL PROPERTIES WITH DIFFERENT BINDERS

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Abstract

Because the timber market is more and more competitive, the particleboard manufacturers are looked for new sources of vegetal raw material supply. In the same time, the use of healthier, safer and more environmentally friendly materials become a priority in the building sector. In this context, some agricultural byproducts as annual plant stems can be an interesting alternative. In fact these resources are abundant, renewable and safe raw material. Moreover their porous structure gives them interesting properties for building materials such as lightness and thermal insulation capacity. In order to diversify raw material supply sources of a particleboard manufacturer, two agricultural byproducts have been studied: flax shives and sunflower bark. The particleboards are made at a laboratory scale by thermocompression of vegetal raw particles at a target density of 500 kg.m⁻³. Two particle sizes have been compared in the panels for each agrosresource: 0.5-2 mm and 2-5mm. The vegetal particles are bonded by different methods:

- without addition of any binder. In that case water is sprayed on the vegetal particles before the forming process at 80% w/w. The lignocellulosic compounds contained in the agrosresources can act as binders.
- with a biosourced binder based on casein, incorporated at different rates with the vegetal particles.

The observed mechanical behavior (by bending test and internal bond) for the particleboards can be very different in function of the agrosresource, the particle size and the binder used. The different materials are also compared by their thermal properties and their behavior with water. By using a biobased binder the mechanical properties of the particleboards are very better. For all studied properties, panels made with flax shives present better properties than these made with sunflower bark so flax shives seem more suitable for particleboard manufacturing. But with optimization of the formulation and the process, both studied agrosresources could be used in particleboards for applications as furniture or door panel and efficient 100% biobased panels can be obtained.

Keywords:

Particleboards, agricultural byproducts, biobased binder, internal bond

1 INTRODUCTION

The exponentially growing consumption of raw materials in the building sector has a very heavy impact on the objectives of sustainable development, both environmental and socio-economic. More than in any other industry, the use of biomass feedstocks can provide a set of relevant responses to these needs. Renewable by nature, available in large quantities, sustainably stocking carbon, generally requiring little production energy and local development vectors, agricultural raw materials are adapted to the manufacture of insulating materials for the building sector [Vo 2016]. In addition, the problem of end-of-life management of building materials containing synthetic resins due to the resistance of polyolefins to environmental degradation make the development of

biobased materials coming from renewable resources a priority axis of research [Laycock 2017].

Agricultural productions generate many co-products that are potential sources of renewable raw materials for the production of environmentally friendly materials. The search for optimization of resources tends towards the valorization of the whole plant: seeds for food; long fibers for the textile industry or technical composite materials; straw, bark, marrow, oilcake and other co-products as new sources of supply for the manufacture of materials that do not compete with food production.

In this context the development of particleboards based on agricultural by-products, such as annual plants stems, is an attractive alternative to wood. Many studies use agricultural residues as raw material for particleboards such as wheat straw and corn

pith [Wang 2002], rice straw [Wei 2015] and coir fibers [Zhang 2014], sunflower stalks [Mati-Baouche 2014, Klimek 2016], flax shives [Papadopoulos 2003].

In the search for alternatives to petroleum-based wood adhesives, efforts have been dedicated to develop adhesives by using phenolic substitutes based on lignin [Ghaffar 2014], tannin [Pizzi 2016], gum Arabic [Abuarra 2014] or lignosulfonate [Privas 2013]. In many studies proteins are used as biobased adhesives, for example wheat gluten [Khosravi 2015] and soy proteins [Mo 2013]. Casein, the protein from bovine milk obtained by precipitating in the milk after acid treatment, is available in large quantities but few studies propose to use it as adhesive for vegetal particles. El Hajj et al. [El Hajj 2012] have used casein as agrobinder with flax shives. In this study the casein-based agrobinder is used in high proportions in the composite material (50% to 70%). The results are interesting in terms of mechanical, thermal and acoustical properties but the resistance to humidity must be improved. Varzi et al. use casein as adhesive for electrochemical double layer electrodes [Varzi 2016]. In different types of materials, it has been demonstrated that casein or caseinate can bring other interesting properties as flame retardant for cotton fabrics [Alongi 2014] and antimicrobial action in edible films with carvacrol or lysozyme [Arrieta 2014, Colak 2015]. These properties can be interesting in building material to which specifications in terms of resistance to fire and durability against microorganisms are required.

The study presented in this paper consists in formulating a binder based on casein in aqueous solution and to optimize it according to the mechanical properties obtained thanks to this binder in particle boards made of agricultural by-products. The optimized biobased binder is then used to bind flax shives and sunflower bark of two different particle sizes in order to obtain particleboards of 500 kg.m⁻³. To evaluate the effects of the binder addition, these particleboards are compared with other ones made with the same vegetal particles but without any binder. All the particleboards are characterized by their mechanical, thermal and hydric properties, which allow to understand the structure-property relationships of the vegetal particles with the binder and to conclude on the capacity of these biosourced and made of by-products materials to be used as building materials according to the EN 15197 standard.

2 MATERIALS AND METHODS

2.1 Raw materials

Agroresources

The flax shives used in this study are by-products of the production of flax fiber and come from the Northwest of France. Flax shives represent the woody part of plant stalks. They are used as animal litter and for particleboard manufacturing (only with synthetic resin as binder). This agricultural byproduct is very abundant in the North of France and new valorization way can be considered.

The sunflower bark is the external part of sunflower stalks. It can be obtained after separation of the sunflower pith which forms the internal part of the stalks. The sunflower bark used in this study came from the center-west of France. A small proportion of pith residues, which cannot be eliminated during the separation process, is present with the bark. Currently

the sunflower bark is not at all valorized. It is left in the field after head harvesting. All the harvesting and industrial valorization chain must be carried out but this agricultural byproduct presents a very high potential of valorization for agromaterials.

The agro-resources used are heterogeneous materials consisting essentially of structural compounds (cellulose, hemicelluloses, lignin) and, to a lesser extent, of non-structural compounds (other polysaccharides, proteins, water-soluble organic compounds and inorganic compounds). The chemical composition of an agro-resource depends on its botanical origin, of course, but also on the location of the agroresource in the plant and on its growth and harvest conditions. These parameters also influence the physical structure of the agroresource: its porosity, its apparent density, its mechanical strength.

Biobased binder

In the case of the particleboards prepared without addition of any binder, the lignocellulosic compounds contained in the agroresources act as binders. They are extracted thanks to evaporation of the added water during the thermocompression process.

The others particleboards are bound thanks to addition of a biobased binder based on casein. The casein is the protein extracted from bovine milk. It is dissolved in a basic solution containing potassium hydroxide (KOH), which allows to form a water soluble caseinate. The used casein and KOH came from Sigma Aldrich.

Different rates of KOH compared with the mass of casein have been tested on panels formed with small flax shives. Similarly, different rates of casein compared with the panel mass have been tested (with fixed KOH rate). Thanks to these results an optimized formulation of the casein-KOH binder has been defined and used to compare the panels with various types of agroresources and with binderless panels.

2.2 Particleboards manufacturing

The raw particles of flax and sunflower were crushed and then sieved to obtain two batches of particles of different sizes. The small particles were sieved between 0.5 and 2 mm while the big particles were sieved between 2 and 5 mm. Particleboards are made with big or small particles of flax shives or sunflower bark and with the optimized biobased binder or without any binder addition, which leads to 8 different panels. The quantity of matter used was calculated in order to obtain a panel density of 500 kg.m⁻³.

The vegetal particles blended with the biobased binder (12% mass/panel mass) or with water (80% mass/vegetal particles mass) were thermocompressed during 20 minutes for the panels containing casein based binder and 40 minutes for the panel without binder addition at 190°C. Chocks are utilized to stop the closure of the mold in order to obtain a fixed panel volume and so the target density.

2.3 Particleboards characterization

Mechanical behavior by bending tests

Three-point bending tests were carried out on each particleboard with a universal mechanical properties testing machine (TA.XTplus texture analyser). Samples of 15 cm long are put on two supports spaced out by 10 cm. A force with a constant speed of 6 mm.min⁻¹ is applied in the middle of the samples. The required force to deform them was measured using a 500 N load cell. The sample dimensions were 150x30x15 mm³. They were conditioned at 50%

relative humidity before testing. The tests were repeated five times, at ambient temperature. The three-point bending test allows to determine the modulus of rupture (MOR) and the modulus of elasticity (MOE) according to EN standards methods (EN 310).

Internal bond

Internal bending strength (IB in $N.mm^{-2}$) corresponds to the energy required to break the sample by a z-directional tensile test. It gives an estimation of the fiber-to-fiber bond. The IB was measured according to the standard EN 319, on five squared samples ($50 \times 50 \times 15 mm^3$) of each particleboard. Prior to testing the samples were conditioned at 50% relative humidity then glued onto stainless steel supports with a hot melt glue. These supports were positioned in the holders of the TA.XTplus texture analyser. A loading rate of $3 mm.min^{-1}$ was applied until failure of the sample. The occurrence of the failure inside the sample and not at the interface between the sample and the glue is checked for each test.

Thermal conductivity

The thermal conductivity of the particleboards was measured with a Heat Flow Meter HFM 436 Lamda from Netzch. The sample size was $150 \times 150 \times 15 mm^3$. The measurements were performed in a steady state at $20^\circ C$ with a temperature gradient between the hot and the cold plate of $20^\circ C$ and repeated at least 3 times for each material.

Behavior with water

Thickness swelling (TS) and water absorbed (WA) were measured on samples of $50 \times 50 \times 15 mm^3$, after fully immersion in $20^\circ C$ distilled water during 24 hours. After removal from the water bath, excess of water was removed with absorbent paper tissues. The three dimensions of the samples (width, length and thickness) were measured by a caliper in the mid of each sample before and after the water immersion. The evolution of the sample thicknesses and volumes is calculated. The water absorption was determined by weighting the samples before and after the immersion, on a scale with 0.1mg precision. The measurements are repeated on five samples for each particleboard.

3 RESULTS AND DISCUSSION

3.1 Biosourced binder formulation

First tests have been carried out to formulate the binder based on casein. Casein can be dissolved in water only in a basic solution, where casein forms a caseinate. That is why we have added potassium hydroxide in the solution. In order to fix the proportion of KOH which is required to obtain the best binder with casein, different KOH rates compared with casein mass have been tested. The efficiency of the obtained binders has been evaluated by comparison of the panel mechanical properties. For the test of binder formulation, only small flax shives have been used in the panels. Fig. 1 presents the maximal bending strength of the panels containing 12% (mass / panel mass) of casein and various KOH rates (the rate of KOH is given by comparison with casein quantity) dissolved in 80 ml of water. The pH of the casein-KOH solution is neutral for 4% KOH but the maximal bending strength of these panels increase significantly with the increasing of the KOH rate beyond the neutrality zone.

According to these results, the KOH rate given the best mechanical result (8.5MPa) is selected for the last steps of the study, that is to say 20% of KOH compared with the casein mass.

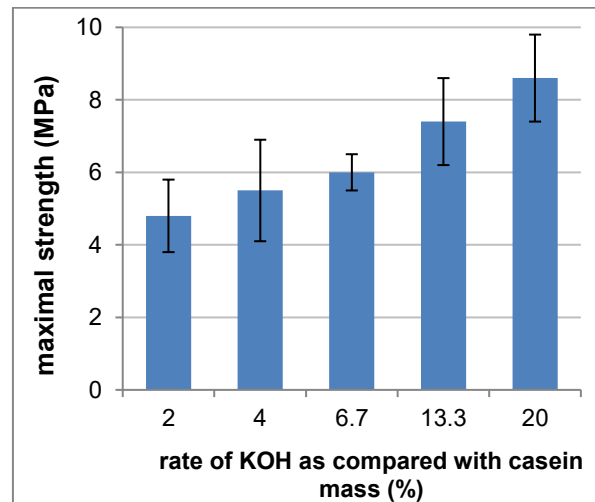


Fig. 1: Maximal strength by 3 point bending test of small flax shive panels containing 12% (m/panel m) of casein and various KOH rates.

The second step of the binder formulation has been to fix the casein proportion in the panel. We wanted to limit the total proportion of binder in the panel (casein + KOH) at a maximum of 18% because it is the mass proportion of synthetic binder in the classical wood based particleboards. Fig. 2 presents the maximal bending strength of the panels containing various mass proportion of casein (always with 20% mass /casein mass of KOH). We can see that the maximal strength increases with the casein rate until 9% casein and then the panel bending resistance is approximately constant between 9 and 15% of casein with 8.5 to 8.8 MPa.

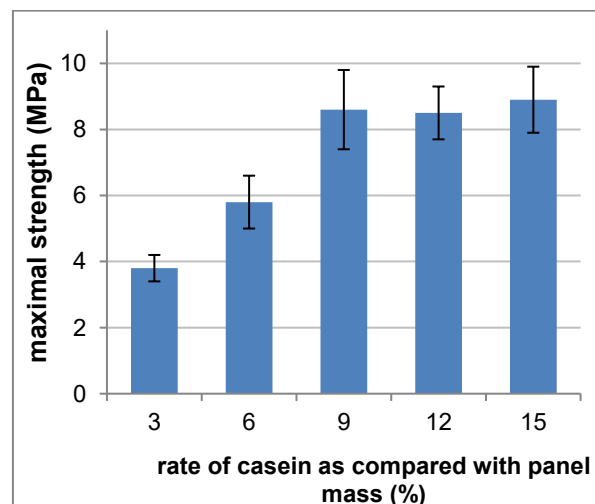


Fig. 2: Maximal strength by 3 point bending test of small flax shive panels containing 20% (m/casein m) of KOH and various casein rates.

In order to make a choice between 9%, 12% or 15% casein in the flax shives panels, we have compared their internal bond, which represents the internal cohesion between the particles. Fig. 3 presents the internal bond of these three panels. The result obtained with 9% casein is significantly lower than those obtained for both other panels. With 12% and 15% casein in the panels, the internal bond is approximately $0.35 N.mm^{-2}$. This is a good result

according to the European standard NF EN 15197 which defines the specifications of flaxboards for various applications. IB of the same range (0.1 to 0.5 N.mm²) have been obtained on particleboards made of wood particles with wheat gluten-based binder used at 12% in boards which present densities between 630 to 700 kg.m⁻³ [Khosravi 2011]. With the formulations of the biobased binder presented here the flaxpanels present as good mechanical properties as panels manufactured with synthetic binder. The IB values are equivalent in panels with 12% and 15% casein. This means that we can minimize the binder proportion up to 12% casein without decrease panel mechanical resistance. That is the reason why we choose 12% casein for the binder formulation in further panels.

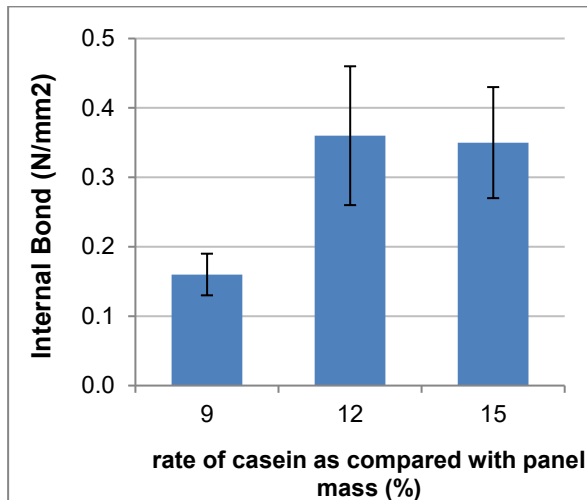


Fig.3: Internal bond of small flax shive panels containing 20% (m/casein m) of KOH and various casein rates.

20% of KOH compared with the casein mass has been selected. It corresponds to 2,4% of KOH compared with the panel mass containing 12% of casein. It represents the better compromise between bending resistance and internal cohesion. This binder formulation is used in the panels studied further in which botanic origin of the particles and particle size vary and the obtained panels are compared with binderless panels.

3.2 Comparison of panel properties with or without binder

Mechanical behavior by bending tests

Table 1 presents the modulus of rupture and the modulus of elasticity obtained by bending test of the different studied panels. A large difference is observed between the panels bound with the biobased binder or without. It seems interesting to realize a particleboard without binder because this corresponds to valorization only of the agroressource and its internal composition. In fact it has been demonstrated that interesting compounds must be extracted from the vegetal matter thanks to vapor and then activated with heating to act as binder and then obtain cohesive particleboards. Water extractable compounds are responsible for the self-adhesion capacity of agro-resources during the thermomechanical steam treatment of binderless particleboards [Pintiaux 2015]. The vapor can be obtained by vaporization in the mold of the water previously blending with the vegetal particles [Halvarsson 2009] or by direct vapor injection in the mold during pressing [Xu 2003].

In the study presented here the binderless panels manually seems to have interesting cohesion but the bending test shows that their MOR and MOE are low with 0.8 to 3.3 MPa for MOR and 127 to 274 MPa for MOE. Nevertheless we can notice that mechanical resistance is slightly better for the binderless panel made of big flax shives than for the three others. Comparable results have been obtained in a previous study [Mahieu, 2015] in which various agricultural by-products have been used in three-layer binderless particleboards. Flax shives and sunflower bark binderless panels showed MOR less than 1 MPa but it seemed a proper value because a panel based on wood bound with UF resin presented a MOR of 0.7 MPa by the same laboratory scale thermopressing process. But in the present study the same process can lead to panels with very much better MOR when biosourced binder is added.

Bending tests carried on panels containing the casein based binder show that panels made of big vegetal particles present better mechanical resistance than these made of small particles in all cases. Reducing the vegetal particle size induces a loss of the inherent mechanical resistance of the vegetal matter. That is the reason why the commercial particleboards are made of three layers with small particles on both external layers for esthetic aspect and big particles in the middle to provide the mechanical resistance.

The results of bending tests also show a difference according to the botanical origin of the vegetal particles. In fact at same particle size and same binding process, the panels made of flax shives always present higher MOR and MOE than the panels made of sunflower bark. The morphology of these agroressources can explain this result. The flax shives are fine and flexible particles (thickness between 0.1 and 0.4 mm) whereas sunflower bark is thicker (between 0.3 to 3.5 mm) and these particles are hard and brittle. With a maximal bending strength of 7.5 MPa, the panel made of big sunflower bark with casein based binder could be used to replace classical wood based particleboards in applications for non-working panels (first class of requirements described in the EN 15197 standard). With a maximal bending strength of 9.2 MPa, the panel made of big flax shives with casein based binder could be used to replace classical particleboards in applications corresponding to the first and the second classes of requirements described in the EN 15197 standard.

Insulation properties

Although all studied panels have the same target density, the measured thermal conductivities vary. A significant difference is observed between panels made with the KOH-casein binder or without binder. As shown in Table 1, the thermal conductivities of the binderless panels are comprised between 72 and 78 mW.m⁻¹.K⁻¹ whereas these of panels which contain the biobased binder are between approximately 80 and 94 mW.m⁻¹.K⁻¹. This can be due to the replacement of a part of the vegetal particles by the binder. The vegetal particles present a honeycomb structure with fine cavities full of air, the best insulator, whereas the binder is denser. This also explains the results presented in Table 2: the thermal conductivity increases with the increase of binder rate in the panel. El Hajj et al. [El Hajj 2012] come to the same conclusion on agromaterials made of casein based binder with flax shives (50% to 70% of binder).

Tab. 1: Modulus of rupture, modulus of elasticity and thermal conductivity of the different panels

binder	agro-resource	particle size	MOR (Mpa)		MOE (Mpa)		λ (mW.m ⁻¹ .K ⁻¹)	
			average	standard deviation	average	standard deviation	average	standard deviation
casein (12%)	flaxes shives	small	8,5	0,8	768	80	84,1	0,7
		big	9,2	0,7	962	60	79,7	0,6
KOH (2,4%)	sunflower bark	small	5,7	0,3	288	23	94,3	1,2
		big	7,5	0,6	790	84	84,5	0,6
binderless	flaxes shives	small	1,2	0,2	165	14	76,7	2,2
		big	3,3	0,4	274	22	72,0	2,1
	sunflower bark	small	0,8	0,1	127	18	77,9	2,1
		big	1,0	0,0	139	12	72,9	1,1

Tab. 2: Thermal conductivity of panels made of small flax shives with different casein rates (and 20% (w/casein w) of KOH)

casein rate in the panel (%)	λ (mW.m ⁻¹ .K ⁻¹)	
	average	standard deviation
9	81,8	0,8
12	84,1	0,7
15	88,2	0,7

The particleboards include two types of porosity: the internal porosity of the vegetal particleboards and the porosity induced by the arrangement of the particles in the panel, which is linked to the panel density. At the same panel density, the internal porosity of the vegetal particles has a more important role in the material insulation capacity than the external porosity. This observation is confirmed by the difference observed between thermal conductivities of comparable panels made of flax shives or sunflower bark. Indeed the bulk density of the flax shives is 100 kg.m⁻³ whereas that of sunflower bark is 180 kg.m⁻³. The sunflower bark includes much less internal porosity than the flax shives. Consequently, the panels made of sunflower bark present higher thermal conductivity than these made of flax shives at the same panel density with

KOH-casein binder for small and for big particles. The difference of thermal conductivity between sunflower and flax particles is less significant for binderless panels. It is evident that the biobased binder has a higher influence on sunflower bark than on flax shives. The particle size also has an influence on the panel thermal conductivity at constant panel density. In all case λ is lower with big particles than with small ones. Due to the crushing, a part of the initial internal porosity is destroyed in the small particles. The most insulating panel is the one made of big flax shives without binder with 72 mW.m⁻¹.K⁻¹. But all the studied panels present interesting thermal conductivities in comparison with classical wood based particleboards which present λ around 100 mW.m⁻¹.K⁻¹.

Properties to water

Table 3 presents the mass percentage of water absorbed after 24h of immersion in water and the corresponding percentage of thickness swelling. The vegetal particles present naturally a high affinity with water and are very absorbent due to their high porosity. In binderless panel, the cohesion of the particles is due to internal components extracted during the thermocompression process thanks to the water sprayed on the agroresources. So the components that act as binder are water soluble.

Tab. 3: Water Absorbed (WA) and Thickness Swelling (SW) of the different panels after 24h immersion in water.

binder	agro-resource	particle size	Water Absorbed (%)		Thickness Swelling (%)	
			average	standard deviation	average	standard deviation
casein (12%)	flaxes shives	small	103	12	18	1
		big	149	8	23	1
KOH (2,4%)	sunflower bark	small	140	9	26	3
		big	141	14	30	3
binderless	flaxes shives	small	262	5	53	1
		big	245	11	74	1
	sunflower bark	small	274	18	84	4
		big	221	7	101	8

The binderless panels absorb high quantities of water, 220 to 270% of the panel initial mass and swell a lot, from 50 to 100% of the initial thickness. Consequently the binderless panels present very bad cohesion after the water immersion. On the contrary, the panels containing the KOH-casein binder present very better cohesion after the immersion. Thanks to the biobased binder the WA and the TS are well reduced. Nevertheless the values obtained for the panels with the binder remain high with WA from 100 to 150% and TS from 18 to 30%.

A significant difference of thickness swelling is observed between panels based on small or big particles. In all cases the TS is higher for big particles than for small ones. It can be explained by the higher size of the pores in the big vegetal particles.

Another solution will have to be found to improve the resistance to water of these particleboards. The panels based on flax shives and sunflower bark with biobased binder may still be used for application in dry environment according to the standard NF EN 15197

4 CONCLUSION

In order to understand the structure-property relationships of particleboards from agricultural by-products, two agro-resources from different botanical origin at two different particle size and two types of particle agglomeration have been tested.

The mechanical strength of the particleboards depends on the interactions involved between the surface of the particles and the binder used. Vapor extractable compounds are responsible for the self-adhesion capacity of the agro-resources during the thermocompression of the binderless particleboards. By using a biobased binder the mechanical resistance is improved thanks to increasing of the adhesion between the vegetal particles. The addition of a biobased binder can also allow to adapt the formulation to the required performances of the final material like improvement of the water and the fire resistance [Alongi 2014] or durability with antimicrobial compounds [Arrieta 2014, Colak 2015], while keeping good thermal properties.

A formulation of a biobased binder based on casein has been optimized according to the mechanical properties of particleboards made of flax shives. It appears that an excess of KOH in comparison with the casein mass leads to better mechanical properties of the panels. A proportion of 12% (w/w) of casein in the particleboard is sufficient to obtain interesting mechanical resistance. The comparison of the different studied panels shows that:

- Big particles provide better mechanical resistance than small ones.
- In all cases flax panels are more resistant than sunflower based panels.
- Addition of the biobased binder based on casein leads to particleboards with very better mechanical properties than the binderless ones.

The casein-based binder has also a good effect on the panel resistance to water. The thickness swelling is reduced by approximately 3 for all panels containing the biobased binder as compared with the corresponding binderless panels.

Nevertheless the binderless panels have one advantage over the panels containing binder: their thermal conductivity is lower thanks to the porous structure of the vegetal matter.

The microstructure of the plant material directly influences the performance of the biobased materials. From a mechanical point of view, the porosity makes it possible to obtain materials with low densities, so with interesting thermal insulation capacity and generally having a lower compressive strength but capable of deforming with little or no degradation. The open porosity facilitates the absorption of sound waves and contributes to acoustic insulation [Glé 2011]. In further work acoustic properties of our particleboards can be evaluated, as well as their resistance to fire.

Big flax shives seem more suitable for particleboard manufacturing but with optimization of the formulation and the process, both studied agro-resources could be used in particleboards for non-working applications in a dry environment. It could allow to choose the raw vegetal particle to manufacture particleboards according to the locally available agro-resources.

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