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Composite Panels from the Combination of Rice Husk and Wood Chips with a Natural Resin Based on Tannins Reinforced with Sugar Cane Molasses Intended for Building Insulation: Physico-Mechanical and Thermal Properties

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Abstract

The objective of this work is to develop new biosourced insulating composites from rice husks and wood chips that can be used in the building sector. It appears from the properties of the precursors that rice chips and husks are materials which can have good thermal conductivity and therefore the combination of these precursors could make it possible to obtain panels with good insulating properties. With regard to environmental and climatic constraints, the composite panels formulated at various rates were tested and the physico-mechanical and thermal properties showed that it was essential to add a crosslinker in order to increase certain solicitation. an incorporation rate of 12% to 30% made it possible to obtain panels with low thermal conductivity, a low surface water absorption capacity and which gives the composite good thermal insulation and will find many applications in the construction and real estate sector. Finally, new solutions to improve the fire reaction of the insulation panels are tested which allows to identify suitable solutions for the developed composites. In view of the flame tests, the panels

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obtained are good and can effectively combat fire safety in public buildings.

Keywords

Composite Panels, Tannins Reinforced, Sugar Cane Molasses, Building Insulation, Mechanical and Thermal Properties

1. Introduction

Scientific advances in the field of the valorization of natural substances from plants make it possible to envisage in the long term that green chemistry products, substitutable and competitive, will replace those derived from fossil materials, in the fields of energy, materials and fine chemicals. As these fossil materials are in the process of being exhausted, researchers are turning to easily renewable sources such as wood. Note that 52% of current medicines are made up of or derived from natural products [1]. Waste from the first processing of wood constitutes a considerable source of raw material that must be recovered. Cameroon has a significant forest heritage, representing nearly 22 million hectares or approximately 42% of the national surface area. This heritage is made up of nearly 300 species, of which only around 80 are subject to regular exploitation. It is the second-largest forest in Africa after that of the Democratic Republic of Congo. Wood is the second largest export product after oil. In 2019, the activity generated 453 million m^3 of wood residue. Thus, for a factory input of 2,500,000 m^3, we only have 748,000 m^3 of wood produced, i.e. a material yield of 30% (70% of waste produced). We note that the wood industry in Cameroon generates large quantities of waste which are most often used as fuel or left abandoned in nature [2]. In many industrialized countries, its waste is used in the manufacture of particle boards, composite materials, energy production and represents real economic potential and various jobs. The use of these residues for the development of construction materials represents an interesting solution for reducing the embodied energy associated with buildings. Numerous studies have already shown the high hygrothermal performances of these materials making their use relevant in particular for the insulation of buildings [3] [4]. Such resources could partly replace certain conventional materials and thus contribute to slowing down the depletion of natural resources such as aggregates, soils, rocks, hydrocarbons, etc. On the other hand, even if the performance of certain materials from Agro-resources, such as hemp concrete or fibrous insulation has been the subject of numerous studies, their use still remains marginal in the field of construction. Despite the significant development potential of this type of material, the only biosourced insulating products today democratized and significantly present on the market are insulation based on wood fibers, cellulose wadding, and some insulation based on long fibers plants (hemp, linen, recycled cotton, etc.). Furthermore, very few studies have focused on the industrialization of construction materials derived from agricultural co-products. Polymers derived from petro-

leum chemistry have been part of our daily lives since their development around the middle of the 20th century. The demand for oil for energy and chemical applications continues to grow year after year, especially with demand from emerging countries. This growing demand around the world is fueling experts' fears that oil resources will be exhausted in the years to come [5] although this subject has been the subject of debate among scientists for years. Indeed, new oil resources have been discovered, again in recent years, particularly in the form of shale gas. However, the majority of global economic systems are based on what is commonly called the "price of the barrel", the effects are felt in all areas: economic, of course, but also political and social. At the same time, recent decades have seen the rise around the world of a growing desire for ecological developments with the protection and preservation of the environment. The development of renewable energies then increased and numerous research into the integration of natural products into industrial processes emerged. This was particularly the case in the wood panel industry with a specific objective of considerably reducing, or even completely replacing, the resins based on petroleum derivatives currently used by products of natural and renewable origin. Wood is a very rich resource from this point of view on the one hand because it is composed of polymeric materials such as cellulose, hemicelluloses and lignins but also because it contains polymerizable components such as tannins which are notably become essential in the wood glue and bonding industry [6] [7] [8]. Sustainable development cannot be carried out without the integration into the design of a product of an ecological dimension from its birth to its end of life and even beyond with the notions of recycling which can no longer be ignored. Concerning particle boards, glues represent approximately 10% by weight of the formulation. On the other hand, studies have already shown that compounds emitted by this type of materials and in particular the glue they contain are harmful to humans and the environment. The idea of replacing all or part of the resins currently included in the composition of the panels seems to be an interesting and promising alternative for economic and ecological reasons. Among these, tannins can be recovered from bark which is found in large quantities in primary wood processing industries at the end of the debarking phase. Despite research which is developing day by day around this theme of "green glues", industrial applications remain poorly developed compared to the costs which must defy the tough competition with those generated by the use of synthetic glues and the appearance technique to obtain performances at least equal to those currently used [9]. The objective of this work is on the one hand to produce an adhesive resin based on biosourced tannins and aldehydes whose objective is to partially and completely replace the formaldehyde used as a hardener for these resins and which constitutes a health problem. major (because carcinogenic), both in its handling and in the emissions of Volatile Organic Compounds (VOC) from products manufactured with such adhesives and on the other hand, the development of foam formulations based on tannins with the aim of obtaining materials that are relatively homogeneous in density and cell morphology, not very

dense and relatively efficient from a mechanical point of view, with the aim of making panels that can be used for thermal comfort in homes.

2. Material and Methods

The first step consists of preparing the resin. To do this, extracts were purchased on the market and the following protocol gives the formulation of the organic resin.

2.1. Resin Formulation

Tannin Resins

The tannins extract used for the preparation of the resin were the mixture of two commercial mimosa tannin extracts (sulfited and non-sulfited) and a non-sulfited quebracho tannins extract. A tannin solution in water was first prepared at a concventration of 55% and mechanically stirred; it could be heated to a maximum temperature of 40˚C to reduce the amount of lumps. The pH was adjusted to 10.4 with the addition of a 33%NaOH water solution. After a few minutes of stirring 6% hexamethylenetetramine was added as 30% water solution. The hexamethylenetetramine /tannin ratio was 0.06:1 (on dry mater basis) and the final tannin concentration was, in theory, 45%. In practice, it was slightly less than this, depending on the amount of NaOH added and the amount of lumps that formed [10].

2.2. Characterization of Formulation Precursors

2.2.1. Measurement of the Apparent Density of Residues

This measurement is carried out according to the protocol defined by the RILEM 236-BBM technical committee [3]. The materials are dried at 60˚C until the mass variation is less than 0.1% over three consecutive days. A transparent cylinder with an internal diameter of 94 mm and a height of 204 mm was used to determine the volume occupied by the aggregates. A known mass of material is placed in the cylinder. The quantity of material is adjusted so that the height of the material in the cylinder is between 100 and 150 mm. The cylinder is turned over 10 times then shaken so as to form a flat surface on the surface of the material. A piece of cardboard is placed on top of the material and the level is marked. The cylinder is then emptied of the material and filled with water to the mark. The mass of water is determined by weighing, which then makes it possible to determine the volume occupied by the material.

2.2.2. Thermal Conductivity (CT) Measurement

The measurement by the hot wire method is carried out using a thermal conductivity meter and a wire probe. A 50 mm probe was used. The principle of this transient measurement is to create local heating within the material using the hot wire whose power and heating time are defined. The temperature change at the center of the material is recorded using the thermocouple of the wire probe. The temperature rise is correlated to the thermal conductivity by the following

relationship [11]: $\Delta T = q/4\pi\lambda\ ((ln(t) + cste))$.

With ΔT: *The temperature difference between the temperature at time t and the initial temperature* [*K*].

q: the linear flux imposed by the wire probe [*W/m*].

(*CT*)*: the thermal conductivity of the material analyzed* [*W*/(*m*/*K*)].

t: the time elapsed after the start of the warm-up [*s*].

cste: Constant which depends on the diffusivity of the material and the contact resistance.

2.3. Test Specimen Manufacturing Protocol and Preparation of Organic Binders

Mixing resin, shavings and rice husk and molding the composites. The chips and rice husks were taken in equal proportion and the rate of incorporation of the resin into the composite was varied. The binder or previously reconstituted resin is introduced and mixed with the chips in a mixer. The mixture is maintained for 3 - 4 min to ensure that the dispersion of the binder or resin on the plant particles is homogeneous.

The samples measuring 100 mm × 100 mm × 50 mm are manufactured using a metal mold. The mass of glued particles introduced into each mold is controlled by weighing the mold during the filling stage. The material in the mold is then cold compressed to the desired final thickness using an INSTRON model 5588S universal press with a capacity of 400 kN. After compression, the material is kept compressed using a metal grid placed on the upper face of the material. Once the materials are compressed, the mold is placed in an oven to apply heat treatment to the composite. The heating condition retained after various calibration tests of 1 hour at 90˚C. The molds are left to cool at room temperature for 45 min before unmolding the samples. Figure 1 illustrates the composite samples made with this mold manufacturing method.

2.4. Characterization of the Physical Properties of Composites

2.4.1. Volumic Mass

The density of composite products is identified on the molded samples or on

Figure 1. Steps in manufacturing composites by molding.

pieces of plates cut with a band saw. The density is calculated from the measurement of the mass of the sample (Sartorius balance, precision 0.01 g) and the estimation of the volume by measuring the dimensions of the sample. Each dimension corresponds to the average of 4 measurements taken with a caliper (Dexter, precision 0.01 mm).

2.4.2. Measurement of Thermal Conductivity Using the Hot Wire Method

The measurement by the hot wire method is carried out using the CT meter and the 50 mm wire probe presented previously. The measurements are carried out on the cased surfaces of the samples. The measurement on the composites is carried out, for each formulation, on three samples measuring 100 mm × 100 mm × 100 mm or six samples 100 mm × 100 mm × 50 mm in the case where the thickness is less than 100 mm . A mass is placed on top of the samples to ensure good contact between the probe and the samples. For each formulation, nine measurements are taken to determine the average thermal conductivity. The measurement is carried out in a closed cabinet, in order to avoid any disturbances linked to a draft and the tests are carried out in a temperature-stabilized room. For the dry point measurement, the samples are dried in an oven at 60˚C until the mass variation is less than 0.1% over three consecutive days. For these dry point measurements, silica gel is placed in the cabinet to maintain a dry environment.

2.4.3. Characterization of Mechanical Properties

1) Compression test

The compression tests are carried out using an INSTRON universal machine (model 5588S with capacity 400 kN, sensor precision: ±1 µm and ±1 N) at a constant displacement speed of 5 mm/min and the test is stopped when the displacement reaches approximately 30% strain. Data acquisition is done using Bluehill software. The EN 826 standard specifies measuring the compressive resistance of insulating materials at 10% deformation (σc, 10) [12]. However, in our case, we have also determined the stress $\sigma c,_{max}$ identified as being the point of change in the slope beyond which the essentially elastic reversible deformation becomes an elasto-plastic deformation. The stresses $\sigma\ c,_{max}$ and σc,10 will be identified as the compressive strength of the composite and the compressive strength at 10% deformation. The elastic modulus in compression is evaluated by regression of the stress-strain curve in the elastic zone. The samples were stabilized for at least two weeks in a climatic chamber at 23˚C and 50% RH before being characterized. The tests are carried out on samples measuring 100 mm × 100 mm × 10 mm.

2) Three-point bending test

The bending strength and the bending modulus of elasticity are evaluated according to the standardized three-point bending test (NF EN 12089) using an INSTRON universal machine (model 5588S with capacity 400 kN, Sensor accuracy: ±1 µm and ±1 N) (AFNOR, 2013b). The distance between the two lower

support points is set at 225 mm, which corresponds to 4.5 times the thickness of the composite. The loading point movement speed is set at 10 mm/min.

2.4.4. Characterization of Water Absorption of Composites

The water absorption of composites is evaluated according to the NF EN 1609 standard for insulating material: Short-term water absorption by partial immersion [13]. The test consists of measuring the quantity of water absorbed by an insulating product when it is immersed in 10 mm of water for 24 hours. The dimensions of the analyzed samples are 200 mm × 90 mm × 5 mm. For each formulation, the measurement is repeated on three samples whose apparent density and mass before immersion are determined before the test. This method uses a steel tray including a continuous water supply device. The water level is kept constant thanks to a water supply throughout the test and an outlet placed 10 mm from the bottom of the tank. A weight is placed on top of the samples to prevent them from floating. The mass of the samples after 24 hours of immersion is recorded. The short-term water absorption value, denoted WS and expressed in kg/m^2, is determined by the following equation:

$WS = (Wf - Wi)/A$ With WS the water absorption value at short term (kg/m^2) Wi and Wf, the initial and final mass of the sample respectively (kg) A, the area of the exposed face of the sample (m^2).

2.4.5. Characterization of the Reaction to Fire of Composites

1) Small flame test

The small flame test is carried out according to standard NF EN 11925-2 [12] [13] [14] [15] [16] making it possible to certify the conformity of a product with Euroclass E. The samples measuring 250 mm × 90 mm × 5 mm are arranged in a ventilated test chamber so that the flame impacts 50 mm from the bottom of the sample. The flame calibrated at 20 mm is inclined at 45˚. To obtain Euroclass E, the standard specifies that the spread of the flame must not exceed 150 mm during the 20 s following the start of exposure. For this test, the flame is applied to the sample for 15 s. To have access to Euroclass D, the flame is applied for 30 s to the sample and it must not exceed 150 mm during the 60 s following the start of exposure. These two tests are carried out on both sides of each sample although this is not specified in the standard. All tests are filmed. After each exposure, the char length is measured. The standard does not require this last measure; however, it gives a good appreciation of the effectiveness of different flame-retardant treatments and allows them to be compared.

3. Results and Discussion

3.1. Thermal Conductivity and Density

Table 1 gives the values of the thermal conductivity and the density of the samples.

Corn cobs and pineapple fibers do not seem to be ideal resources for the development of insulating rigid composite panels because their bulk density

Table 1. Thermal conductivity and density.

Residues	Thermal conductivity (W/m·K)	Density (kg/m³)
Crushed corn cob	392.49 ± 2.37	0.0926 ± 0.0017
Crushed pineapple fiber	94.67 ± 2.18	0.0496 ± 0.0006
Crushed rice husk	47.24 ± 1.91	0.0441 ± 0.0009
Wood chips	28.20 ± 1.60	0.0361 ± 0.0003

and thermal conductivity are too high. On the other hand, rice husks and wood chips have a low apparent density. However, these low values seem to come mainly from the significant inter-granular porosity. Their transformation into rigid composite could result in a significant increase in the density of the composite. For each formulation, six samples measuring 100 mm × 100 mm × 100 mm are manufactured by molding according to the method described in the methodology section and characterized by compression testing.

3.2. Density, Compression Test and Water Absorption of the Composite

The density, compression test and water resistance of the composites were visually evaluated after complete immersion of the composites made with the different binder formulations. The observations are summarized in Table 2.

The analysis in Table 2 shows that the compressive strength strongly depends on the density of the composite. It is important to note that this high value in density, water absorption and compressive strength is obtained for a resin content of 15% with an equal amount of filler in chips and rice husks in the composite. Composite panels have resistance values that can exceed 1 MPa. This significant difference clearly illustrates the excellent compressive behavior of the hemp composite. A water absorption of 8.5 kg/m^2 over 10 mm submerged corresponds to 85% of the porosity. However, based on the actual density of the constituents, the porosity calculated from the formulation is close to 88%. The hypothesis of saturation of the submerged zone is very acceptable [4] [17].

In order to improve water resistance, the addition of a crosslinker has proven essential to improve the reactivity of the binder so that it is compatible with the manufacturing processes and to improve the water resistance of the composite. In view of this, we did a screening and decided to see the parameter behavior at incorporation rates from 12% to 30%.

Table 3 gives the mechanical and thermal properties of the composite panels associated with a sugar cane molasses content.

Thermopressedorganic binder composites, the study of the influence of the formulation showed that the mechanical properties of the composite depend mainly on the apparent density and the proportion of the incorporated resin. The mechanical properties of the rigid insulating panel have far exceeded the mechanical requirements of this type of product. For comparison, rigid wooden

Table 2. Density, compression test and water absorption of the composite.

No.	%Resin and %precursors	Density (kg/m³)	Compression test (MPa)	Water absorption (kg/m²)
1	5% + 95%	196 ± 1.54	0.198 ± 0.044	18 ± 1.07
2	10% + 90%	209 ± 2.75	0.244 ± 0.021	9 ± 0.54
3	15% + 85%	257.34 ± 7.18	0.356 ± 0.035	8.6 ± 1.32
4	20% + 80%	204 ± 2.51	0.702 ± 0.058	8.7 ± 1.11
5	25% + 75%	193 ± 3.8	0.699 ± 0.011	8.5 ± 0.66
6	30% + 70%	189 ± 1.21	0.518 ± 0.044	8.4 ± 0.53

Table 3. Mechanical and thermal properties of composite panels.

% sugarcane molasses	Volumic mass (kg/m³)	Flexural strength (MPa)	Flexural modulus of elasticity (MPa)	Compressive strength (MPa)	Modulus of elasticity in compression (MPa)	Thermal conductivity (W/mK)
12	169 ± 12	0.4899 ± 0.201	39.6 ± 8.1	0.198 ± 0.044	7 ± 2	0.0709 ± 0.0017
14	171 ± 9	0.292 ± 0.032	25.1 ± 6.8	0.269 ± 0.010	4 ± 0	0.0690 ± 0.0017
16	176 ± 14	0.896 ± 0.032	53.2 ± 2.7	0.360 ± 0.063	16 ± 2	0.0688 ± 0.0025
18	179 ± 10	0.925 ± 0.101	81.7 ± 1.43	0.430 ± 0.019	19 ± 3	0.0721 ± 0.0023
20	193 ± 11	0.871 ± 0.1088	44.9 ± 1.1	0.062 ± 0.060	12 ± 1	0.0755 ± 0.0030
22	203 ± 10	0.947 ± 0.063	37.7 ± 1.9	0.741 ± 0.069	7 ± 0	0.0754 ± 0.0021
24	199 ± 16	1.428 ± 0.228	83.5 ± 1.12	0.450 ± 0.024	20 ± 1	0.0846 ± 0.0033
26	214 ± 12	1.774 ± 0.203	150.01 ± 2.5	1.016 ± 0.168	37 ± 6	0.0818 ± 0.0034
28	196 ± 12	1.273 ± 0.025	105 ± 25	0.631 ± 0.052	17 ± 1	0.0785 ± 0.0030
30	186 ± 11	1.030 ± 0.230	82.8 ± 9.2	0.518 ± 0.070	16 ± 2	0.0793 ± 0.0022

insulating panels generally have a compressive strength of 0.2 MPa while those measured on the panels can exceed 1 MPa for an equivalent density. The characterization of the thermal conductivity of the panels made it possible to show that this type of composite presents interesting performances for the insulation of buildings with values between 0.050 and 0.060 W/(mK). The thermal conductivities of the panels were compared with the values measured on bulk agro resources and those obtained with composite panels. The results show an increase in thermal conductivity which could be linked both to a reduction in inter-granular porosity but also to a reduction in intra-granular porosity by deformation of the aggregate during the thermocompression stage. Composites retain their excellent ambient air regulation properties. However, it was identified that, in the same way as for thermal conductivity.

3.3. Evaluation of the Flammability of Composites Using the Small Flame Test

Some composites were chosen on the basis of their properties. Table 4 presents the Results of the small flame test conducted on the panels.

Table 4. Small flame tests of some panels.

% sugar cane molasses	Volumic mass (kg/m³)	Flame propagation after 15 seconds of exposure	Length charred average after 15s exposure (mm)	Spread of the flame after 30 seconds of exposure	Length charred average after 30 seconds of exposure (mm)
Commercial witness	186 ± 3	No	85 ± 3	No	112 ± 30
12	211 ± 5	No	76 ± 0	No	114 ± 21
16	242 ± 210	No	69 ± 3	No	108 ± 11
20	214 ± 4	No	74 ± 6	No	116 ± 7
24	209 ± 8	No	63 ± 4	No	147 ± 34
28	211 ± 7	No	73 ± 2	The flame spread over two of the three sample	98 ± 3

All panels successfully passed the test of exposure for 15 s and 30 s to a small flame with the exception of the panel treated with functionalized silica particles. For the latter, one of the samples did not meet the criteria following exposure for 30 s because the flame propagated over a length greater than 150 mm. This result could be explained by the fact that the functionalized silica particles are dispersed in an IMS solution ("Industrial Methylated Spirit", 99% ethanol), highly flammable solvent which can increase the flammability of samples if added in excess. The solution of functionalized silica particles used for these tests is concentrated at 7% (solid content). Therefore, the significant amount of IMS incorporated in the composite could be responsible for the increased flammability of the samples. An additional study could be carried out to identify whether reducing the solvent content in the solution makes it possible to resolve this problem and improve the flame-retardant effect of the treatment [16].

4. Conclusion

The main objective being to identify alternative solutions making it possible to totally or partially replace synthetic resins, in particular by exploiting biosourced compounds. The results showed that it is possible to replace synthetic resins with organic resins and obtain very interesting physico-mechanical and thermal properties. The composites made from these binders have achieved mechanical performances compatible with the targeted applications. However, the addition of a crosslinker proved essential to improve the reactivity of the binder so that it was compatible with the manufacturing processes and to improve the water resistance of the composite. The association of cane molasses with the resin made it possible to produce composites presenting ranges of values of use characteristics (mechanical resistance, thermal conductivity) meeting the specifications. The properties of thermosetting biosourced panels have made it possible to identify numerous key parameters, in particular the mechanical properties in compression and flexion, which mainly depend on the apparent density of the

composite panel and the volume content of precursors. Thermal conductivity mainly depends on the apparent density of the composite. It would appear that the thermocompression step causes deformation of the aggregate, reducing intra-granular porosity, which further penalizes the thermal conductivity of the composite. Formulation work has shown that the use of a thermosetting bio-sourced binder makes it possible to produce composites with apparent densities of between 150 and 250 kg/m^3. In view of the flame tests, the panels obtained are good and can effectively combat fire safety in public buildings.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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